# CONDENSED AND DRIED MILK





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#### Module 1

#### History, Status and Scope of Condensed Milks in India and Abroad

#### Lesson-1

#### 1.1. INTRODUCTION

Milk, skim milk, whey, and other milk products can be concentrated, i.e., part of the water can be removed. Its main purpose is to diminish the volume and to enhance the shelf life quality. Water can be removed from milk by evaporation and in addition to water, volatile substances, especially dissolved gases are also removed. Evaporation is usually done under reduced pressure — hence, decreased temperature — to prevent damage caused by heating. Water can also be removed by a membrane process called as reverse osmosis, i.e., high pressure is applied to a solution to pass its water through a suitable membrane. Water as well as part of some low-molar-mass substances passes through the membrane. A different method of concentrating is by freezing. Thus, condensed milk is cows' or buffaloes' milk from which a considerable portion of water has been removed.

#### 1.2. HISTORY

The evaporation of milk has been known for many years, even as early as in the year of 1200 when Marco Polo described the production of a paste like milk concentrate in Mongolia which was used by mixing with water.

Then 600 years passed before we again find concentrated milk in the literature, but thereafter the development progressed rapidly.

The advent of condensed milk belongs to 19<sup>th</sup> century. The condensed milk industry was introduced at about the same time as the factory system of butter and cheese making. Its rapid development stands in sharp contrast to the production of butter and cheese.

Nicolas Appert condensed milk in France in 1820 and was developed afterwards in the United States in 1856 by Gail Borden, Jr. Before this development, milk could only be kept fresh for a short while and so was only available in the immediate vicinity of its production. After several attempts, Borden was able to produce a usable milk derivative that was long-lasting and needed no refrigeration. By 1858 Borden's milk, sold as Eagle Brand, had gained a reputation for purity, durability and economy.

In 1864, Gail Borden's New York Condensed Milk Company constructed the New York Milk Condensery in Brewster, New York. This condensery was the largest and most advanced milk factory and was Borden's first commercially successful plant.

The basic principle of the process of sterilization by heat was introduced by Mr. John B. Meyenbarg - a native of Switzerland in the year 1884-'87. He conceived the idea of making condensed milk without the addition of cane sugar or other preservatives and without the necessity of keeping it cold. Mr. Meyenbarg experimented with the sterilization of condensed

milk by steam under pressure and as the result of these experiments; he decided that it was possible to preserve milk without the aid of sugar by the use of revolving sterilizer which he designed.

Originally the unsweetened sterilized condensed milk was sold under the trade name of Evaporated Cream. The Federal Food & Drug act of 1906 changed the name "Evaporated Cream" to "Evaporated Milk".

The vacuum pan that was developed in 1883 has undergone marked improvement as a result of scientific knowledge and at present modern types of evaporators are available in different designs and shapes, which have much more advantages than the original one in terms of evaporating capacity, fuel efficiency and product quality.

In 1911, Nestlé constructed the world's largest condensed milk plant in Dennington, Victoria, Australia.

In 1914, Professor Otto F. Hunziker, head of Purdue University's dairy department, published a book titled *Condensed milk and milk powder*. This text, along with additional work of Professor Hunziker and others involved with the American Dairy Science Association, standardized and improved condensery operations in the U.S. and other countries.

#### 1.3. DEVELOPMENT OF CONDENSING PROCESS

The simplest evaporator is an ordinary open pan heated with steam or direct gas. The evaporation takes place from the surface while the liquid to be evaporated is heated up to the boiling point corresponding to the ambient pressure, which at sea level will be 100°C.

As the evaporation has to take place from the surface, which is limited in relation to the content of the pan, the evaporation will naturally take long time. The milk will be exposed to the high temperature with deterioration of the proteins and chemical reactions such as the Maillard reaction or even coagulation results.

As the development went on, the concentration was carried out in forced recirculation evaporators and multi stage evaporators. For the separation of liquid and vapours, centrifugal separators are preferred.

There are principally two kinds of condensed milk namely Sweetened Condensed and Unsweetened Condensed milks. Both reach the market in hermetically sealed tin cans. Sweetened Condensed Milk as the name suggests is sweetened with addition of sugar which acts to enhance its shelf life.

Unsweetened condensed milk is known by trade name "Evaporated Milk". It is made by removal or evaporation of water from milk without the addition of any preserving material. The canned product is heat sterilized to extend shelf life. Fortification with vitamins of either or both A or  $D_3$  is common. Because of its concentrated form, evaporated milk is a multipurpose, convenient dairy product ready for every milk use.

Evaporated skim milk is obtained by a simple concentration of skim milk by vacuum evaporation or reverse osmosis. Evaporated filled milk is a prepared blend of skim milk, vegetable oil, stabilizers and vitamins.

The improvements in modern equipment are:

- a. Change of metal from copper to stainless steel.
- b. Double and triple effect or combination of single effect evaporator with thermotemperature control.
- c. Plate type evaporators were developed in 1958.

#### 1.4.4. USES OF CONDENSED MILKS

The common uses of concentrated / condensed milks in different parts of the world are shown in Table 1.1 as under:

Table- 1.1: Different uses of condensed milks

Uses	 	 VE:11

- Bakeries
- Confectionaries
- Ice Cream manufacture
- Making prepared food products such as creaming coffee or tea and as flavouring
- Topping on chocolate or similar cream flavor snowball.
- Pouring on cereals and fruits
- Used in meat patties and loaves for consistency
- Coatings for baked or fried meats
- In place of milk in the manufacture of candies, frostings and pies.
- Brazilian candy Brigadeiro
- Use on toast.
- Mixed with sugar and some butter and baked to form a popular sweet candy called Tablet (confectionery) or Swiss-Milk-Tablet.
- Mixed with some evaporated milk and eggs, to make a stiffer and more filling version of crème brulée known as Leche Flan.
- Boiling a can of condensed milk in water for about 2 hours, resulting into product called Kaymak- sweet semi-liquid substance which can be used as a cake icing or put between dry wafers.

## 1.5. STATUS OF CONDENSED AND DRIED MILK INDUSTRY IN INDIA AND ABROAD

#### 1.5.1. Status in India

Milk production in India is 116.4 million tones in 2010-11. Around 135 Dairy Plants both in private and public sector in India are producing or dealing with concentrated and dried milks of various kinds. The milk utilization pattern and its related statistics is given in Table-1.2 below.

Table- 1.2: Milk Utilization pattern in India

No.		Utilization	Ye	ar
			2001	2005
1	Mill	k Production (million tones)	84.6	94.5
2	Mill	k Utilization (%)	100	100
3	A.	Liquid Milk	46.0	50.0
	В.	Traditional Products	50.0	45.0
		(a) Ghee / Makkhan	33.0	-
		(b) Dahi (Curds / Chakka)	7.0	-
		(c) Khoa	7.0	-
		(d) Chhana & Paneer (Cottage Cheese)	3.0	12
	C.	Western Products: Milk Powder, etc.	4.0	5.0

(Source: Dairy India 2007-6<sup>th</sup> edition, p-33)

Table- 1.3: Share of organized and traditional (unorganized) market

(Year 2003-04, Rs. billion)

No.	Product	Organized	Non-organized
1	Packaged Liquid Milk	98.0	-
2	Ethnic Sweets	62.5	455
			(Khoa-based) 375
			(Chhana-based) 80
3	Curd & Curd products	6.3	160
1	Cheese	2.0	(Paneer) 20
5	Ice creams	8.0	-
5	Butter	5.2	60
7	Ghee	35.0	210
3	Milk Powder	38.0	-
	Total	255.0	905

(Source: Dairy India Year book 2007-6th edition, p-34)

Table -1.4: Raw material cost of dairy products as % of sale price

No.	Products	Raw material cost
		as % of sale price
1	Market Milk (bulk vended)	90
2	Market Milk (packaged)	80
3	Milk Powder / Butter	70
4	Paneer	58
5	Sandesh	42
6	Peda/Burfi/Kalakand	52
7	Gulabjamun	33
8	Rasogolla	33
9	Shrikhand	43
10	Dahi	60
11	Mishti Doi	40

(Source: Dairy India 2007-6th edition, p-33)

Table -1.5: Milk flow for powder manufacture

		2005			2011		
	Organized	Organized		Total Organized			
	Coop/Govt	Private		Coop/Govt		Private	
Milk (Million tones)	0.12	0.40	0.52	0.20	0.50	0.70	
Price (Rs/Kg)	90	90		130	130		
Value (Rs million)	10800	36000	46800	26000	65000	91000	

#### 1.5.2. Status in World

Table -1.6: Global trade in dairy products

Million tonnes

Product	Intra-EU Trade	World Trade	World Production	World Trade as % of Production
Condensed Milk, retail pack	0.2	0.5-0.6	4	10-15
Whole Milk Powder	0.25	~1.3	2.5	50
Skim Milk Powder	0.5	~ .	3.5	30

(Source: CFCE / IDF, Rabobank International, Dairy India-2007, P-47)

 $\textbf{Fonterra}, \ a \ \text{New Zealand based multinational company, is the world's largest} \\ \text{producer of milk powder controlling 40 percent of the global whole milk powder.} \\$ 

Table -1.7: Market size of condensed & dried milks

'000 tonnes

Year	Butter	Ghee	Milk Powders	Malted milk food	Condensed milk	Cheese
1991	130.0	942.6	150	41	8.1	2.5
2001	500.0	1830.1	235	68	12.0	8.0

(Source: FAOSTAT for butter & ghee (FAO 2005); Ministry of Food Processing Industries (2002). Dairy India-2007, P-63)

#### 1.5.1. Condensed milk

The market for condensed milk is small and has been growing at a slow pace. The production of condensed milk has increased with an average annual compound growth rate of  $\sim 4 \%$  during 1991 & 2001 period. This growth is marked by large fluctuations in production on a year to year basis.

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#### Module 2

#### Definition and legal standards for evaporated and condensed milks

#### Lesson-2

#### **Legal Standards for Evaporated and Condensed Milks**

#### 2.1. INTRODUCTION

The condensed and dried milks are manufactured by the commercial plants as per the legal standards prescribed by FSSA and BIS in our country.

## 2.2 FOOD SAFETY AND STANDARDS (FOOD PRODUCTS STANDARDS AND FOOD ADDITIVES) REGULATIONS

1. **EVAPORATED MILK** - means the product obtained by partial removal of water from milk of cow and/ or buffalo by heat or any other process which leads to a product of the same composition and characteristics. The fat and protein content of the milk may be adjusted by addition and/ or withdrawal of milk constituents in such a way as not to alter the whey protein to casein ratio of the milk being adjusted. It shall have pleasant taste and flavour free from off flavour and rancidity. It shall be free from any substance foreign to milk. It may contain food additives permitted in these regulation including Appendix A. It shall conform to the microbiological requirements prescribed in Appendix B. It shall conform to the following requirements:

No.	Product	Milk Fat	Milk Solids	Milk Protein in milk solids not fat
	(1)	(2)	(3)	(4)
(i)	Evaporated milk	Not less than 8.0 percent m/m	Not less than 26.0 percent m/m	Not less than 34.0 percent m/m
(ii)	Evaporated partly skimmed milk	Not less than 1.0 percent and not more than 8.0 percent m/m	Not less than 20.0 percent m/m	Not less than 34.0 percent m/m
(iii)	Evaporated skimmed milk	Not more than 1.0 percent m/m	Not less than 20.0 percent m/m	Not less than 34.0 percent m/m
(iv)	Evaporated high fat milk	Not less than 15.0 percent m/m	Not less than 27.0 percent m/m	Not less than 34.0 percent m/m

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2. **SWEETENED CONDENSED MILK** - means the product obtained by partial removal of water from milk of Cow and/or Buffalo with the addition of sugar or a combination of sucrose with other sugars or by any other process which leads to a product of the same composition and characteristics. The fat and/ or protein content of the milk may be adjusted by addition and / or withdrawal of milk constituents in such a way as not to alter the whey protein to casein ratio of the milk being adjusted. It shall have pleasant taste and flavour free from off flavour and rancidity. It shall be free from any substance foreign to milk. It may contain food additives permitted in these regulations including Appendix A. It shall conform to the microbiological requirements prescribed in Appendix B. It shall conform to the following requirements:

No.	Product	roduct Milk Fat		Milk Protein in milk solids not fat
	(1)	(2)	(3)	(4)
(i)	Sweetened condensed milk	Not less than 9.0 percent m/m	Not less than 31.0 percent m/m	Not less than 34.0 percent m/m
(ii)	Sweetened condensed skimmed milk	Not less than 1.0 percent m/m	Not less than 26.0 percent m/m	Not less than 34.0 percent m/m
(iii)	Sweetened condensed partly skimmed milk	Not less than 3.0 percent m/m and Not more than 9.0 percent m/m	Not less than 28.0 percent m/m	Not less than 34.0 percent m/m
(iv)	Sweetened condensed high fat milk	Not less than 16.0 percent m/m	Not less than 30.0 percent m/m	Not less than 34.0 percent m/m

The legal standards as per The Food Safety and Standards Regulations can be referred on web site http://www.fssai.gov.in

#### 2.2. BIS REQUIREMENTS FOR CONDENSED MILKS

#### **2.2.1. BIS Specification for Condensed Milk:** (IS: 1166-1986 (1&3) (First Revision))

Sr. Full Cream Full Cream Characteristic Skim Skim No. Sweetened Unsweetened Sweetened Unsweetened Total Milk Solids, % 31.0 31.0 26.0 20.0 by Wt., Min. Fat, % by Wt., **NLT 9.0 NLT 9.0** NMT 0.5 NMT 0.5 Sucrose, % by Wt., 40.0 40.0 Min. Titratable Acidity (as 0.35 0.30 0.35 0.30 lactic acid), % by Wt. Max. Colour Whitish to Whitish to light Whitish to Whitish to light light brown light brown cream cream Bacterial Count, per 500 500 g, Max. Test for Coliform Negative Negative Organisms 8 Mould 10 10 Yeast Count per g., Max.

Table 2.3: BIS Specification for Condensed Milk

NLT- Not Less Than, NMT- Not More Than

Accelerated Storage Test: Incubate the sample at temperature of 38°C for 14 days. The sample shall pass the test:

- i. Does not show any bulge due to positive pressure within and
- ii. The product inside the can has not curdled or turned and is free from any objectionable taste, odour and sliminess.

## **2.2.2.** BIS Specifications related to the Sweetened Ultra High Temperature (UHT) Treated Condensed Milks:

Table- 2.4: BIS specifications related to the sweetened ultra high temperature

Sr.	Characteristics	Requirements for	
No.		Full Cream	Skim Milk
1	Total milk solids, % by mass, Min	26.0	20.0
2	Fat, % by mass	NLT 8.0	NMT 0.5
3	Sucrose, % by mass, Min.	18	18
4	Titratable acidity (as % LA) Max.	0.35	0.35
5	Titratable acidity (as Acetic acid)	0.37	0.37
	After incubation of packages for 7 days at 3	37 °C by mass	, Max.
6	Bacterial spores (plate count) per $0.1~\mathrm{g},~\mathrm{Max}.$	10	10
7	Coliform test	Negative	Negative
8	Coagulase positive Staphylococcus aureus	Negative	Negative

NLT- Not less than

Permitted limits for additives and microbiological counts are given as Appendix –A and B respectively.

#### INTERNATIONAL STANDARDS

The international standards prescribed by American Dairy products Institute (ADPI) and Codex Alimentarius can be referred on the web sites:

- 1. http://www.adpi.org
- 2. http://www.codexalimentarius.org

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#### Module 3

## Grading and quality of raw milk &pretreatment for concentrated & dried milks

#### Lesson-3

#### 3.1. INTRODUCTION

Milk and milk products are a rich and convenient source of nutrients for people in many countries and international trade of milk-based commodities is significant. All foods have the potential to cause food borne illness, and milk and milk products are no exception. Dairy animals may carry human pathogens. Such pathogens present in milk may increase the risk of causing food borne illness. Moreover, the milking procedure, subsequent cooling and the storage of milk carry the risks of further contamination from man or the environment or growth of inherent pathogens. Further, the composition of many milk products makes them good media for the outgrowth of pathogenic micro-organisms. Potential also exists for the contamination of milk with residues of veterinary drugs, pesticides and other chemical contaminants. Therefore, implementing the proper hygienic control of milk and milk products throughout the production, processing, and handling operations is essential to ensure the safety and suitability of these foods for their intended use as milk and milk products form a large portion of the diet of consumers especially infants, children, and pregnant and lactating women.

#### 3.2. GOOD QUALITY MILK AND MILK PRODUCTS

For better understanding of the quality of milk and milk products and their grading, it is necessary to know the real meaning of fresh, good quality milk and milk products. The following few terms help to understand it better:

- 1. **Milk** is the normal mammary secretion of milking animals obtained from one or more milkings without either addition to it or extraction from it, intended for consumption as liquid milk or for further processing.
- 2. **Milk product** is a product obtained by any processing of milk, which may contain food additives, and other ingredients functionally necessary for the processing.
- 3. Composite milk product is a product of which the milk, milk products or milk constituents are an essential part in terms of quantity in the final product, as consumed provided that the constituents not derived from milk are not intended to take the place in part or in whole of any milk constituent.
- 4. **A reconstituted milk product** is a product resulting from the addition of water to the dried or concentrated form of the product in the amount necessary to re-establish the appropriate water to solids ratio.

5. **A recombined milk product** is a product resulting from the combining of milk fat and milk-solids-non-fat in their preserved forms with or without the addition of water to achieve the appropriate milk product composition.

Several chemical and physical properties of milk follow from its composition and structure. These properties can affect the processing of milk and the quality of milk products.

Conservation of milk solids in form of dried milks has shown an increasing trend in the recent decades owing to the extended shelf life and cost benefits in packaging, transportation and storage of such products. Availability of such products has also facilitated to overcome seasonal and regional deficiency of fluid milk.

In course of manufacture of condensed and dried milks, the quality of raw milk, the pretreatments employed and the drying variables used determine, to a great extent, the physical, chemical, microbiological and organoleptic characteristics as well as the shelf life of the finished product. The importance of raw milk quality in relation to manufacture of concentrated and dried milks is innumerable.

The quality of raw milk to be used should be judged with respect to the following criteria:

- 1. Relationship of milk quality with finished product quality.
- 2. The influence of milk quality on product yield and the process efficiency.
- 3. The quality criteria appropriate for today's technology.
- 4. Requirement of separate quality criteria for each end use, if any.
- 5. The terms specified by the manufacturer of concentrated and dried milks for milk quality.

Raw milk is extremely variable in its composition and characteristics. The quality of raw milk arriving at the dairy premises is of vital concern more so today than ever before, largely because milk, the raw material, now constitutes something like 60-70 % of the final product cost. It is therefore vitally important that

- § The quality is high, so that wastages are minimized.
- § Processing speeds optimized and
- § Product quality raised.

The poor quality milk is not readily accepted by manufacturers for conversion to products. Milk which is unsuitable for the liquid market is certainly unsuitable for manufacture of condensed milks and milk powders as quality specifications are becoming more stringent and manufacturer of the products is required to produce "tailor made" products for specific end uses.

Quality of raw milk used for manufacture of condensed milks and milk powders has the following significance:

- 1. It decides the ease with which it can be handled during processing and manufacture.
- 2. It influences the processing parameters and their effects on powder characteristics.

- 3. It influences quality specifications of the finished product.
- 4. It affects the functional and physico-chemical properties of the finished product.

#### 3.3. HIGH QUALITY MILK

The quality of the raw milk is the single most important criterion that determines the quality of the end product. The quality of the raw incoming milk in turn is dependent on the sanitary procedures followed during the milk production and transportation.

Healthy cows, milk produced under clean environment, clean and hygienic utensils, freedom from colostrum, prompt cooling of milk soon after milking and transport under refrigerated state are the factors that determine the number of microorganisms.

The received milk is thoroughly checked for organoleptic qualities like colour, odour, taste (raw milk is seldom tasted at the entry point) etc. along with other platform tests like clot on boiling (COB), alcohol / ethanol test to determine the suitability of milk for heat processing.

When producing condensed milks / powder for commercial use it must comply with certain bacteriological, chemical, and physical standards. Obviously the quality of the final product is a function of the raw milk quality. It is therefore vital for a profitable production to have milk of first class.

The following criteria are used to define high quality raw milk:

- 1. It has a normal taste and appearance.
- 2. It has a normal chemical and physical composition.
- 3. It does not contain harmful chemical contaminants or residues.
- 4. It has a low total count of bacteria and somatic cells.

The ideal milk for the dairy industry is one which is rich in constituents, which is drawn from the udder of a healthy animal, to which no non-milk constituents have been added and in which no changes have taken place.

The compiled quality criteria, their suggested and/or desirable limits and their relevance to product quality are depicted in Table 3.1.

#### 3.3.1. Raw Skim Milk / Raw Whole Milk

The milk should be fresh and stored at maximum 5°C for maximum 48 hours after milking, and should not have had any pretreatment other than:

- Separation of cream / desludging by means of a centrifuge.
- One heat treatment at max. 72°C and 15 s.
- Standardization of fat content by separation of cream and or addition of skim milk.

The milk should be of a very good quality and be free from any additives (apart from fresh cream for standardization) and suspended solids (filtered  $100~\mu$ ).

Table-3.1: Quality of raw milk in relation to manufacture of condensed milks and milk powders

	powders		
No.	Parameter	Suggested / Desirable Level	Relevance to Milk Powder
1	Appearance, Smell		Influence appearance, flavour of
_	and Taste	flakes, clots, blood	finished product
2 3	Sediment Milk Fat	0.5 – 3.0 mg/Litre Normal – As per local and/orlegal requirements	Scorched particles level may increase Influence on yield, composition
4	MSNF	-do-	-do-
5	Acidity	≤ 0.15 % LA	<ul> <li>Problems with protein stability during processing – early fouling of evaporator</li> <li>Influence on basic standards of products made - e.g. S.I., Total acidity</li> <li>Poor shelf life – colour, flavour,</li> </ul>
			more free fat
6 7	pH Heat Stability	6.6 – 6.8 10 min at 140° C, Alcohol test – Negative	-do- Heat processing of milk becomes problematic - Early fouling of evaporators - Poor solubility and other related problems
8	Copper	0.10 mg/kg	Catalyze oxidation of fat – poor shelf life
9 10	Iron Heavy metals, Pesticides, Insecticides,	1.0 mg/kg Absent	-do- Hazardous to human health
	Mycotoxins		
11	Antibiotics	0.02 IU/ml	Public health view point undesirable, product will be unsuitable for fermented milk products
12	Somatic cell count	5,00,000 /ml	<ul> <li>Poor stability – early fouling of evaporator</li> <li>High Solubility Index</li> <li>Poor shelf life – tallowy flavour</li> </ul>
13	Free Fatty Acids	1 m mol/100 g fat	Appearance, flowability, reconstitutability and shelf life of powder
14	Non-condensable gases	0.01 to 0.02 % by $wt_{\omega}$ max.	Can affect the working of evaporator
15	Total Bacterial Count	1,00,000–5,00,000 CFU/ml	- Bacteriological quality of finished product
16	Thermoresistant Bacteria	5,000 CFU/ml, Max. 25,000 CFU/ml	- Basic standards – S.I., Total Acidity Bacteriological quality of finished product especially LHP
17	Psychrotrophic Bacteria	$2x10^6$ CFU/ml	End use of powder in recombined sterile products.
18		Disk reading 3 ½ - 6	Bacteriological quality of finished product
19	MBR Time	SCM: > 3.5 h. > 2.5 h - Bulk	F
20	Direct	condensed milk. Every 2 weeks to 2-months EM: Not less than 2.5 h	-do-
20	Microscopic Count	Not more than 10,00,000/ml No Morphological types	Mastitic milk lowers heat stability, have higher serum albumin & higher pH plus powder oxidizes during storage further

(Compiled from various sources)

The following Table 3.2 shows reasons for rejection of skim milk powder for not meeting the quality specifications. It is evident from the tabulated values that the total acidity of raw milk and its bacteriological quality can affect the powder

Table- 3.2: Analysis of reasons for rejection of Skim Milk Powder

No.	Cause for Rejection	% Rejection	Origin of Problem
1	Fat	4.5	Process
2	Water	21.0	Process
3	Total acidity	28.0	Milk
4	Neutralization agents	Nil	Milk
5	Phosphatase	Nil	Process
6	Purity (Scorched particles)	9.2	Process
7	Solubility	22.0	Process
8	Germ content	13.0	Milk and/or Process
9	Colon Bacillus	Nil	Process
10	Taste & smell	Nil	Milk or Process
11	Appearance	2.3	Milk or Process

Compiled from various sources

#### **3.1.1.** Sweet Whey

The whey originates from the production of cheese made from fresh milk, free from any additives. The whey should have undergone one single pretreatment, i.e. separation of fat and insoluble particles. Table 3.3 shows desired specifications for the sweet whey.

Table-3.3: Desirable specifications for sweet whey to be used for condensing and drying

No.	Constituents	Suggested Limits			
1	Total solids	Approx. 6%			
2	Curd fines test	Max. 200 mg/kg			
3	pН	Min. 6.3			
4	Titratable acidity	Max. 0.12% as lactic acid			
5	True lactic acid	Max. 20 mg/100 g			
6	Fat content	Max. 0.05%			
7	Lactose content	Min. 70%, max. 74% on SNF			
8	Protein content	Min. 12% on SNF			
9	Calcium content	Max. 300 ppm			
10	Magnesium content	Max. 100 ppm			
11	Chloride content	Max. 1200 mg/L			
Non condensable gases		Max. 0.02% by weight			

(Compiled from various sources)

When the whey is stored between the cheese factory and the evaporation process at a temperature exceeding 10°C, then the storage time should not exceed one hour. If the storage temperature is below 10°C, then the storage time should not exceed ten hours.

\*\*\*\*\*\*

#### Lesson-4

#### **Pretreatments for Concentrated Milks**

#### 4.1. INTRODUCTION

In course of manufacture of concentrated and dried milks, raw milk is subjected to various pretreatments, which in turn determine to a great extent, the physical, chemical, microbiological and organoleptic characteristics as well as the shelf life of the finished product. The technological aspects of various pretreatments and their significance in the manufacture of dried milks are discussed here.

The following pretreatments are given to milk during the processing schedule:

- 1. Filtration and/or centrifugation
- 2. Standardization
- 3. Forewarming /Preheating
- 4. Concentration / Concentrate heating
- 5. Homogenization

## 4.2. FILTRATION AND/OR CENTRIFUGATION (SEPARATION, CLARIFICATION AND BACTOFUGATION)

#### **Filtration**

The basis for high quality and fine flavor of the product is "clean milk" in preference to "cleaned milk". However, for sanitary, ethical as well as for technological reasons the milk that is received at the dairy is filtered and/or centrifuged.

Weigh can strainers, strainers and filters in the milk line or a duplicate filter unit with by-pass connection are general filtration techniques. Bactocatch method developed by Alfa-Laval Co., Sweden, involves filtration of the milk through ceramic filters prior to pasteurization. This microfiltration technique removes 99.6% of the bacteria in the milk so that the following normal low pasteurization results in 50% life extension when milk is kept at 8°C. The advantages of this process are:

- § Economical
- § Bacillus cereus level is reduced appreciably
- § Absence of disagreeable flavour that sometimes is present in overheated milk

- § Excellent quality low heat powder for cheesemaking, least adverse effect on rennetability, curd characteristics etc.
- § Low heat powder having extended shelf life.

#### Centrifugation

There are three types of centrifugal processes to which milk may be subjected during manufacture of dried milks. These are:

- § Separation proper i.e. skimming
- § Clarification
- § Bactofugation

**Centrifugal clarification** is done in hot or cold condition in a centrifugal triprocess separator, which serves the purpose of clarification. A clarifier removes leucocytes, cellular debris and particles from earth or fodder gaining entry into milk which might act as a catalyst for chemical reaction during storage of powders.

The **Bactofugation** process, originated by Professor Simonart is designed to remove bacteria from milk by means of centrifugation. Basically the bactofuge is a clarifier with one inlet and one outlet for the treated liquid. Additionally, there are two nozzles of about 0.4m diameter fitted into the bowl wall for the discharge of skim milk or what is called 'bactofugate' rich in protein and bacteria. The machine is characterized by a much greater efficiency of removal of bacteria than conventional clarifiers. Bactofugation is a selective method of removing bacteria, the size and density of the bacteria being the criteria of their removal. Bactofugation of milk is always applied in combination with heat treatment, a temperature of 55-65 ° C offering the optimum bactofugation effect. A combination of heat treatment and bactofugation results in a greater reduction of bacteria than is possible by heat treatment alone. Likewise, bactofugation of milk by single pass process is less effective (90% removal of bacteria) than two pass process where two bactofuges are arranged in series (bacterial reduction may be up to 99.9%). To improve the quality of raw milk for dried milk, bactofugation has been suggested by some workers. Though clarification has also some beneficial effects, cream separation has no effect on ultimate quality of powder. However, some losses of constituents, though negligible, do occur while bactofuging the milk. Heating (130 - 140 ° C) the bactofugate and remixing it with treated milk can eliminate the losses due to bactofugation.

Influences of centrifugation (i.e. clarification and bactofugation) of milk on quality of milk powders can be summarized as follows:

- 1. Centrifugation prior to concentration improves casein dispersion and heat stability.
- 2. Improved shelf life of powder due to reduced tendency of oxidation and lipolysis due to removal of slime, foreign materials (which may act as chemical reaction catalyst), leucocytes, lipolytic bacteria and enzymes etc.
- 3. Improved solubility to powder (1.8-2.0%) when clarification is applied at 35-  $40^{\circ}$ C.

- 4. Decrease in sediment formation
- 5. Better organoleptic quality of resultant powder.
- 6. Improved bacteriological quality of finished product.

#### 4.3. Standardization

- § Milk intended for dry milk manufacture is standardized to obtain a product of expected quality, to meet the legal standards and to optimize yield and profit.
- § In manufacture of dried milks, the milk fat and SNF are so standardized that the finished product will meet the standards of not less than 26% fat (BIS, IDF and ADPI standards for whole milk powder).
- § The fat to SNF ratio adjustment is done by addition of calculated amount of cream or skim milk to the milk. This purpose is accomplished in the present day industry by use of tripurpose separator which has the standardizing device too. Other computerized systems are also used commercially.

#### 4.4. PREHEATING / FOREWARMING OF MILK

In manufacture of milk-powders milk is subjected to heat treatment in two stages-

- (A). Prior to concentration
- (B). After concentration, before atomization.

#### 4.4.1. Heating prior to concentration has the following objectives:

Preheating of milk to at least the boiling temperature of the first effect evaporation is essential, so that evaporation can start immediately the milk enters the heating tubes. The most common boiling temperatures of the first effect in the food industry are between 40-100°C. Heating of milk to a temperature exceeding the boiling temperature of the first effect are required for the purpose of:

- (a) Enzyme inactivation, e.g. lipoprotein lipase.
- (b) Pasteurization effect improved microbiological quality of resultant product due to microbial inactivation. Heat treatment employed in manufacture of low and high heat powders had profound effect on bacterial profile. Both pasteurization and high heat treatment (90 ° C, 10 min) almost completely eliminate psychrotrophic bacteria. Pasteurization had little effect on either thermoduric or spore counts whereas high heat treatment brought about significant reduction in bacterial numbers.
- (c) To lengthen the running time of evaporators (as most of the whey proteins are denatured when milk is preheated before evaporation).

- (d) To generate specific properties in the resultant milk powders, e.g. heat stability, viscosity, etc. by altering the characteristics of some of the components of milk such as casein, whey protein, mineral balance etc.
- (e) To improve oxidative stability of whole milk powder during storage. This is due to production of -SH groups and other reducing substances in milk on account of heating.

To produce skim milk powder of various heat classification (Table 4.1 and 4.2). The actual treatment required depends on many factors, including the composition of milk as influenced by the breed, season, climate etc. and the equipment including type and construction of heater. The influence of spray drying on WPNI is negligible.

Table- 4.1: Classification of Non-Fat Dry Milk on the basis of Heat Class

No.	Heat Class	Heat / Casein	WPNI (ADMI)	Cysteine
		Number, %	mg/g	Number
1	Extra Low Heat	-	-	26-33
2	Low Heat	< 80.0	> 6.0	34-41
3	Medium Heat	80.1-83.0	4.5-5.99	2
4	Medium High Heat	83.1-88.0	1.5-4.49	-
5	High Heat	> 88.1	< 1.5	-

Table- 4.2: Typical time-temperature combinations for different types of powders

Type	Temperature	Time
Low Heat	74°C	15 Sec
Medium Heat	79°C	3 min
High Heat	82°C	30 min

## 4.4.2. CONCENTRATION/ HEATING AFTER CONCENTRATION / CONCENTRATE HEATING:

The viscosity of the concentrate has bearing on drying efficiency as well as certain properties of the dried milks. In general, high concentrate viscosity can be the source of many practical problems by increasing the likelihood of blockage of feed lines and in the calandria of evaporators.

Therefore, it is often desirable to reduce the viscosity of concentrate to as low a viscosity as possible. The viscosity of concentrated milk is significantly affected by temperature. To achieve desired low viscosity of the feed, the concentrate has to be heated to 70-75°C prior to atomization. This in turn has the following advantages:

- (a) Saving of energy as it is more efficient to heat the concentrate by means of steam or hot water than to use heat from the air supplied to the dryer. The evaporation capacity of the dryer is increased by  $\sim 5\%$  when the concentrate is supplied to the atomizer at  $70^{\circ}$ C instead of  $50^{\circ}$ C.
- (b) The average size of the droplets into which the concentrate is atomized is reduced due to the lower viscosity. This facilitates the drying making it possible to reduce the air outlet temperature and still maintain the specified moisture content of the powder. The lower outlet temperature results in increased capacity and gentler drying which results in improved powder quality especially solubility.

(c) As the temperature conditions in the last stages of the evaporator and in the feed tank for the dryer can be favorable for growth of micro organisms, heat treatment of the concentrate immediately before drying will contribute to safeguard the bacteriological quality of the powder. Heating of the concentrate must be done in such a way that no holding takes place, i.e. the concentrate heater must be placed immediately before the atomizer. This is necessary in order to avoid the so called "age-thickening" which occurs very rapidly at temperatures above 60 ° C. The time dependent thickening of hot concentrate is irreversible and results in very high viscosity. Table 4.3 shows changes in viscosity of concentrated skim milk (39%TS) held for varying period at 75°C. Table 4.4 shows the effect of storing concentrated skim milk (39%TS) at 50°C and 60°C on powder solubility. Since concentrate is a complex system its heating has to be done carefully to minimize adverse effects of heating on chemical and physical characteristics of the concentrate. Otherwise, such change will reflect on the quality of resultant milk powders. Tables 4.5 and 4.6 show influence of concentrate heating on the various characteristics of whole milk powder.

Heating of milk after concentration helps to retain higher percentage of antioxidants.

Table 4.3: Changes in viscosity of concentrated skim milk (39 % TS) held for different periods at 75°C

No.	Holding time (min)	Viscosity (cP)
1	0	14
2	15	41
3	30	90
4	45	168
5	60	360
6	75	460

Table 4.4: The effect on powder solubility of storing concentrated skim milk (39 % TS) at 50 and 60°C

No.	Storage time	Solubility Index of powder (ml)							
	(h)	Storage temp. 50°C	Storage temp. 60°C						
1	0	0.02	0.02						
2	2	0.02	0.02						
3	4	0.02	0.02						
4	6	0.02	0.11						

Table 4.5: Influence of the heating on the characteristics of whole milk powder concentrate (Non-homogenized, Dry matter content: 49.5%)

Temperature of concentrate, <sup>0</sup> C	Moisture, %	Viscosity cP	ADPI Solubility Index (ml)	Degree of homogenization, %	Free-fat content,	Bulk Density, g/cm³	N <sub>2</sub> penetration cm <sup>3</sup> /100 g	Mean particle density	Vacuole volume cm <sup>3</sup> /100 g
50	3.18	3610	1.20	50	13.8	0.58	2.95	1.05	17.4
60	2.87	2945	0.60	52	14.5	0.57	3.18	1.04	18.7
70	3.04	1510	0.30	55	15.9	0.55	4.00	1.01	21.2
80	3.02	1150	0.20	58	21.1	0.54	5.08	1.00	22.5

Table 4.6: Relationship between the heating and the dry matter content of non-homogenized concentrates concerning the free fat content of whole milk powder

Temperature of	Dry matter content of concentrate (%)								
concentrate, <sup>0</sup> C	43.9	44.9	46.9	47.3	48.1	48.3	49.3	49.5	
50	22.8	21.9	22.8	20.3	14.7	14.3	19.3	13.8	
60	22.7	22.2	21.3	20.9	16.7	14.7	19.6	14.5	
70	22.5	25.6	22.6	22.4	17.8	18.1	22.0	15.9	
80	22.3	23.8	24.9	23.8	22.8	19.6	25.9	21.1	

#### 4.4.3. HOMOGENIZATION

- § Homogenization is used as one of the pretreatments in manufacture of whole milk powder as well as some specific products.
- § The purpose of homogenizing milk/concentrate prior to spray drying is to reduce the size of the fat globules and consequently to change the physical structure in such a way that the dried product obtained has a low free fat content.
- § The type of homogenizer, the stage at which product is homogenized, the temperatures and pressures of homogenization employed have profound effect on the various properties of the concentrate and this in turn have influence on the quality of resultant milk powders.

Table 4.7, 4.8 show influence of homogenization on the various characteristics o[ whole milk powder.

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Table 4.7: Influence of the concentrate homogenization on various characteristics of whole milk powder

Concentrate	Dry Matter, %	Moisture Content, %	Viscosity, cP	ADPI Solubility Index (ml)	Degree of homogenization, %	Free-fat content, %	Bulk Density, g/cm <sup>3</sup>	N <sub>2</sub> penetration cm <sup>3</sup> /100 g	Mean particle density	Vacuole volume cm <sup>3</sup> /100 g	Y 10 Permeability factor
Non-	43.2	2.73	1380	0.05	36	23.7	0.49	5.48	0.91	32.9	16.6
homogenized	49.1	3.18	1635	0.40	54	12.0	0.54	3.09	1.01	21.2	14.5
	54.7	3.52	3040	1.20	79	5.2	0.62	1.98	1.06	17.2	11.4
Single –stage	43.2	2.57	800	0.10	77	7.3	0.50	3.71	0.90	34.3	10.8
homogenized	49.1	3.07	1630	1.80	80	4.8	0.55	2.01	1.05	24.3	8.2
100.0	54.7	3.41	5960	2.20	88	4.6	0.61	1.17	1.10	18.7	6.2
Two-stage	43.2	2.58	700	0.05	79	7.1	0.49	4.25	0.93	30.4	13.9
homogenized	49.1	3.09	1000	0.30	77	6.3	0.53	2.66	1.03	29.1	9.1
	54.7	3.46	3260	0.70	89	5.1	0.62	2.28	1.10	23.9	9.5

Table 4.8: Influence of the homogenization pressure on various characteristics of the concentrate and whole milk powder

		Concentrate			Powder			
Concentrate	Homogenization Pressure, MPa	Viscosity, cP	Degree of homogenization, %	Viscosity, cP	ADPI Solubility Index (ml)	Degree of homogenization, %	Free-fat content, %	N <sub>2</sub> penetration cm <sup>3</sup> /100 g
Non-	-	50	20	1250	0.10	50	16.5	4.69
homogenized								
Single -stage	14	58	58	1075	0.10	65	7.2	3.20
homogenized	16	80	68	1100	0.10	66	5.8	2.42
	18.5	110	85	1090	0.10	77	3.9	2.62
	20	135	85	1130	0.10	81	3.7	2.07
Two-stage	14	50	50	975	0.05	55	12.1	4.35
homogenized	16	60	65	1010	0.05	68	7.1	4.42
	18.5	75	75	1000	0.05	77	4.9	4.04
	20	96	80	985	0.05	84	4.2	4.10

From the above write up it can be seen that the various pretreatments employed during manufacture of dried milks have profound effect on successful manufacture of these products. Therefore, a better understanding of the effects of relevant pretreatments is essential to avoid unexpected difficulties in the course of manufacture and to meet ever increasing stringent product specifications .

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#### Module 4

#### **Manufacturing techniques**

#### Lesson-5

#### **Basics of Evaporators**

#### **5.1. INTRODUCTION**

The vacuum pan or vacuum evaporator is the heart of milk condensery. It is the retort in which the condensing proper is accomplished. It is used in the manufacture of every type of concentrated milks.

#### 5.2. DEVELOPMENTS IN EVAPORATORS

While unchanged in principle from that of originally used by Gail Borden, the vacuum pan unit has undergone marked improvement. The vacuum pan of today has appreciably increased evaporative capacity due to type and arrangement of heating surface and of condenser; it is capable of greater fuel economy, more efficient use of condenser water, better control of entrainment losses and more effective protection of the milk against heat damage. Systems are also designed to achieve the maximum plant efficiency, with the minimum downtime, and the desired balance between steam and electricity use.

The simplest evaporator is an ordinary open pan heated with steam or direct gas. The evaporation takes place from the surface while the liquid to be evaporated is heated up to the boiling point corresponding to the ambient pressure, which at sea level will be 100°C.

As the evaporation has to take place from the surface, which is limited in relation to the content of the pan, the evaporation will naturally take long time. The milk will be exposed to the high temperature with a deterioration of the proteins, chemical reactions such as the Maillard reaction, or even coagulation as a result.

As the development went on, the concentration is carried out in forced recirculation evaporators. The heating surface is provided either by a steam jacket or a series of steam coils or by milk tubes enclosed in a steam chest or calandria. Evaporating capacity of the plant depends upon:

- (1) Type and arrangement of heating surface in condenser.
- (2) More efficient use of water for condensing plant.
- (3) More effective control of milk against heat damage.

The area, design, and arrangement of the heating surface play an important part in determining the evaporating capacity of the vacuum pan. The latter is directly proportional to

- (1) Total area of heating surface,
- (2) Temperature difference between steam and milk, and
- (3) Overall heat transfer co-efficient which is influenced by
- a. Velocity of milk and steam flow
- b. Viscosity
- c. Density
- d. Sp. Gravity
- e. Sp. heat
- f. Thermal conductivity of milk, metal, milk film, steam condensate film and incrustation.

Some systems used to regenerate heat are:

#### **5.2.1. Falling Film Evaporators**

Over the years the falling film evaporator has practically replaced the forced recirculation evaporator. This type of evaporator is desirable from a product point of view, as it offers a short holding time. Further, the amount of product in the evaporator is reduced and the surface from which the evaporation takes place is increased. Figure 5.1 shows a diagram of a falling film evaporator.

#### Fig.5.1: Falling film evaporator

The liquid to be evaporated is evenly distributed on the inner surface of a tube. The liquid will flow downwards forming a thin film, from which the boiling/evaporation will take place because of the heat applied by the steam. See Figure 5.2. The steam will condense and flow downwards on the outer surface of the tube. A number of tubes are built together side by side. At each end the tubes are fixed to tube plates, and finally the tube bundle is enclosed by a jacket, see Figure 5.3. The steam is introduced through the jacket. The space between the tubes is thus forming the heating section. The inner side of the tubes is called the boiling section. Together they form the so-called calandria. The concentrated liquid and the vapour leave the calandria at the bottom part, from where the main proportion of the concentrated liquid is discharged. The remaining part enters the subsequent separator tangentially together with the vapour. The separated concentrate is discharged (usually by means of the same pump as for the major part of the concentrate from the calandria), and the vapour leaves the separator from the top. The heating steam, which condenses on the outer surface of the tubes, is collected as condensate at the bottom part of the heating section, from where it is discharged by means of a pump.

#### Fig-5.2: Evaporation in a falling film evaporator tube

#### Fig. 5.3: Evaporator calandria

Since milk, due to the protein content, is a heat-sensitive product, evaporation (i.e. boiling) at 100°C will result in denaturation of these proteins to such an extent that the final product is considered unfit for consumption. The boiling section is therefore operated under vacuum, which means that the boiling/evaporation takes place at a lower temperature than that corresponding to the normal atmospheric pressure. The vacuum is created by a vacuum pump prior to start-up of the evaporator and is maintained by condensing the vapour by means of cooling water. A vacuum pump or similar is used to evacuate incondensable gases from the milk.

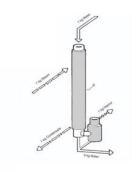


Fig. 5.4: One stage evaporator, definition of various specific quantities and the corresponding condensate

As vapour, see Figure 5.4, from the evaporated milk contains almost all the applied energy, it is obvious to utilize this to evaporate more water by condensing the vapour. This is done by adding another calandria to the evaporator. This new calandria - the second effect - where the boiling temperature is lower, now works as condenser for the vapours from the first effect, and the energy in the vapour is thus utilized as it condenses.

Multiple effects: In the first evaporation unit, steam is introduced and part of the water is evaporated from the milk. The steam condenses, and vapour is separated from the concentrated milk. The vapor is used to evaporate water from the concentrated milk in the second effect, etc. The flows of milk and vapour are cocurrent. The number of effects varies from 3 to 7. The vapour coming from the last effect is condensed in a special condenser; the temperature of the water in the condenser determines the boiling temperature in the last effect. The boiling temperatures in the other effects are determined by the pressure drop of the vapour when being transported to the next effect. The temperature difference  $\Delta T$  between the condensing vapour, and the boiling liquid will be smaller for a larger number of effects N. A higher N implies greater saving of steam but needs a larger heating surface; hence, a bigger and more expensive plant, more loss of heat by radiation, and higher cleaning costs. Especially in the last effect, the heat-transfer rate may become very small: here, the temperature is lowest, and the concentrate viscosity is very high.

From Figure 5.5 we can see that 1 kg of steam can evaporate 2 kg of water and by applying a third effect 3 kg of water is evaporated using only 1 kg of steam.

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Fig- 5.5: Principle of two-stage evaporation of water

#### 5.3. SEPARATORS

**Separators with Tangential Vapour Inlet:** As the vapours generated from the evaporation are used as heating media in the "next" calandria, any product must be separated, since it would otherwise contaminate the condensate and further represent a loss. The majority of the concentrate is discharged from the bottom of the calandria below the tube bundle. Due to the high vapour velocity some of the concentrate will be carried along with the vapour as small droplets. The separation is done in a separator with tangential vapour inlet, see Figure 5.6 connected to the calandria below the tube.

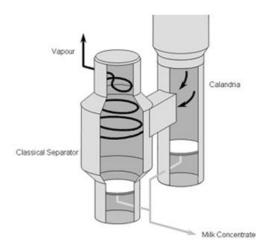


Fig. 5.6: Separator with tangential inlet

#### 5.3.1. Wrap-Around Separator

To reduce space requirements a new development has taken place with the design of the Wrap-Around Separator. It is integrated into the base of the calandria. It has the same high efficiency as the classical separator with a low pressure drop. It is typically used on big calandrias with MVR compressors connected to the wrap-around separator with a very short vapour duct minimizing the pressure drop.

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#### Lesson-6

#### **Condensed Milk**

#### 6.1. INTRODUCTION

Condensed milk is a milk product obtained by evaporating part of water of whole milk, or fully or partly skimmed milk, with or without the addition of sugar. They are intended for use as such or for pre-condensing the fluid milk or fluid milk by–product preparatory to the manufacture of dried milk products.

The term 'condensed milk' is commonly used when referring to full cream sweetened condensed milk whereas the term evaporated milk is generally used while referring to full cream unsweetened condensed skim milk. Skimmed milk products are known as sweetened condensed skim and unsweetened condensed skim milk respectively.

#### 6.2. SWEETENED CONDENSED MILKS

Sweetened condensed milks are milk products which can be obtained by the partial removal of water from milk with the addition of sugar, or by any other process which leads to a product of the same composition and characteristics. The fat and/or protein content of the milk may have been adjusted, only to comply with the compositional requirements by the addition and/or withdrawal of milk constituents in such a way as not to alter the whey protein to casein ratio of the milk being adjusted.

It may sometimes contain added refined lactose, calcium chloride, citric acid and sodium citrate, sodium salts of orthophosphoric acid and polyphosphoric acid not exceeding 0.3 per cent by weight of the finished product.

Such kind of addition need not be declared on the label. Sweetened condensed milk should contain not less than 9.0 percent milk fat, and not less than 31 per cent milk solids and 40.0 per cent cane sugar.

#### They may be

- 1. Sweetened condensed milk
- 2. Sweetened condensed skimmed milk
- 3. Sweetened condensed partly skimmed milk
- 4. Sweetened condensed high-fat milk

#### 6.3. EVAPORATED MILKS

Evaporated milks are milk products which can be obtained by the partial removal of water from milk by heat, or by any other process which leads to a product of the same composition and characteristics. The fat and/or protein content of the milk may have been adjusted, only to comply with the compositional requirements by the addition and/or withdrawal of milk constituents in such a way as not to alter the whey protein to casein ratio of the milk being adjusted.

It may contain added calcium chloride, citric acid and sodium citrate, sodium salts of orthophosphoric acid and polyphosphoric acid not exceeding 0.3 per cent by weight of the finished product. Such addition need not be declared on the label. Unsweetened condensed milk should contain not less than 8.0 percent milk fat, and not less than 26 per cent milk solids.

They may be:

- 1. Evaporated milk
- 2. Evaporated skim milk
- 3. Evaporated partly skimmed milk
- 4. Evaporated high-fat milk
- 5. Evaporated Filled Milk: Evaporated filled milk is a prepared blend of skim milk, vegetable oil, stabilizers and vitamins.

Because of its concentrated form, evaporated milk is a multipurpose, convenient dairy product ready for every milk use. Pouring directly from the can, evaporated milk can be used in countless applications: creaming coffee or tea, poured on cereals and fruits, providing consistency in meat patties and loaves, coatings for baked or fried meats, or in place of milk in the manufacture of candies, frostings and pies.

#### 6.4. ADVANTAGE OF CONDENSING MILK IN VACUUM

The chief advantages of condensing milk in vacuum over evaporation under atmospheric pressure are three fold. They are

#### 1. Economy of Operation

The total heat units expended when condensing in vacuum pan at the usual temperature of about 55 to 60 °C are slightly greater than the total heat units required for evaporation under atmospheric pressure. There is no saving in total heat units due to evaporation under reduced pressure. However evaporation in vacuum has the distinct advantage of making possible the economical utilization of steam at low temperature and of exhaust steam. Hence where exhaust steam is available there is opportunity for a considerable saving in fuel by its use.

The fact that under reduced pressure milk boils at relatively low temp (55 to 60°C) makes possible the use of low pressure steam such as exhaust steam, without sacrificing the rapidity of evaporation. This assists in saving fuel and reduces operating cost. In short, it helps reutilization of steam used in condensing and adoption of double and triple system of condensing. Other advantages are:

• About 90% of the water contained in the milk is removed in the evaporator and only 10% in the spray dryer. However, the energy required per Kg water evaporated in the spray dryer is 16-20 times the energy required per Kg water removed in the evaporator.

- In absolute terms, the energy consumption of the dryer is approximately twice that of the evaporator if a six effect evaporator is used. If a 4 effect evaporator is used the energy consumption of the evaporator and the dryer will be approximately the same. The above illustrates that although only 10 % of the water is removed in the dryer, this should not lead to the assumption that the efficiency of the dryer is of minor importance.
- In an effort to improve the overall efficiency of the process, the tendency has been to concentrate the milk as much as possible prior to drying. The importance of such tendency is illustrated by the fact that if for a given milk throughput, the evaporator concentration falls from 50 to 48%, the dryer evaporative load is increased by 8.7%, while if the evaporator concentrations increases to 52% the dryer load is reduced by 7.5%. Since, the energy supplied to the dryer is about 60% of the total required for drying process; these variations have a marked effect on the overall economy.
- In modern plants, skim and whole milks are concentrated to 48-50 % solids and whey to 55-60 %.
- In view of the above, it has been suggested that evaporators and dryers should be developed that can handle milk of higher total solids. However, the use of higher concentrations is not an engineering problem related to the design of evaporators and dryers, it is a technological problem.

#### 2. Rapidity of Evaporation

The fundamental factor that determines the rapidity of evaporation is the rate of heat transmission or the amount of heat transmitted by the steam to milk per unit area of heating surface per hour. The rapidity of heat transmission in turn depends largely upon the temperature difference between steam and milk. The greater the difference, the more heat is transmitted. The rapidity of evaporation also depends upon surface area available and viscosity of the product. The rapidity of evaporation is necessary:

- (1) To check the growth of microorganisms that may have survived higher heat treatment.
- (2) To have economy of evaporation.
- (3) To protect the milk against heat damage.

#### 3. Protection of Milk against Heat Damage during Operation

Condensing in vacuum pan makes possible evaporation at a relatively low temperature (55 to 60°C). With present evaporating equipment, milk temperature of 52 to 57°C is usually maintained. Exposure to high evaporating temperatures hampers the quality. The low boiling point saves the milk from heat damage.

In addition, it widens the gap between temperature of steam and milk, so steam of low pressure and hence of low temperature, protects the milk from a shock from heating surface which would have been the case in atmospheric pressure.

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#### Lesson-7

#### **Manufacture of Sweetened Condensed Milk**

#### 7.1. INTRODUCTION

The basic principle underlying the production of condensed milk is that milk of high quality is filtered or clarified, standardized, forewarmed, and condensed to the desired level as shown in Fig. 7.1. The preservation of sweetened condensed milk is by the addition of sugar whereas the evaporated milk is preserved by heat sterilization.

#### 7.2. MANUFACTURE OF SWEETENED CONDENSED MILK

Sweetened condensed milk is milk that is concentrated by evaporation, to which sucrose is added to form an almost saturated sugar solution, after which it is packed. The high sugar concentration is primarily responsible for the keeping quality of the product and for its fairly long shelf life, even after the can has been opened, although it then will eventually become moldy.

#### Fig. 7.1: Manufacture of Sweetened Condensed Milk

#### 7.1. Selection of Milk

The milk for the concentration purpose should have the quality as discussed in Lesson 3. As far as possible, milk should be fresh, sweet and should be free from off-flavors. Milk having off – flavours should be rejected because it is not possible in this case to remove all off-flavours. It should not have sediments. Milk after weighing should be clarified and filtered to remove visible foreign matter, which is not only unsightly but also cause consumer complaints. It is a common practice to preheat the milk before clarification in the warm clarifiers in order to increase the efficiency of the operation. It should be cooled to 4.4°C if it is not be used immediately.

#### 7.2. Standardization of Milk

This is done to conform to the legal standards in the end product. The standardization of milk meant for condensed milk preparation is done with a view:

- 1. To bring the Fat: SNF ratio to the desired level.
- 2. To establish the desired ratio between the added sugar and total milk solids.
- 3. To adjusts the concentration of the finished product to the desired percentage of total solids.

#### 7.3. Correcting the Fat Shortage of the Batch

The fat shortage of the batch is corrected by the addition of cream.

**Example:** We want 8.55 % Fat and 28.00% TS in the finished product. We have got 100,000 kgs of milk having 3.6% Fat and 12.11 % TS. How much cream of 40% Fat must be added to provide the desired ratio of fat to SNF?

#### **Solution**

The finished product must contain 28.0% - TS, 8.55% fat and 19.45% SNF.

Now ratio of Fat to SNF desired is: 8.55/19.45 = 0.4395:1

So for each kg of SNF there must be 0.4395 kgs of fat.

We know that milk on hand posses 12.11% TS and 3.6% fat, so

Therefore, milk on hand contains 12.11% TS, 3.60% fat and 8.51% SNF.

Hence, the fat content should be:  $8.51 \times 0.4395 = 3.74 \%$ 

If 1 kg of SNF contains 0.4395 kg of fat, therefore 8.51 kgs of SNF contains 3.74 kgs fat.

Therefore, fat deficiency is equal to: 3.74 - 3.60 = 0.014% fat

Now, 100 kgs of milk = 0.14 kgs of fat deficit.

Therefore, 100000 kgs of milk = (100000 x 0.014) / 100 = 140 kgs of fat shortage.

This fat shortage can be made up by addition of the cream of 40% Fat. However, cream also contains SNF. The Skim milk portion of 3.6% fat milk contains 8.82% SNF.

Therefore, 40% fat cream contains:  $\{(100-40)/100\} \times 8.82 = 5.3 \% \text{ SNF}$ 

Hence, in order to balance or obtain the desired ratio of Fat to SNF, it is necessary to reserve

 $5.3 \times 0.4395 = 2.33 \%$  Fat to balance the SNF added with the cream.

This then leaves the fat available in the cream to (40 - 2.33) = 37.67% Fat to recover the fat shortage of 140 kgs and hence requires the addition of  $(140 \times 100) / 37.67 = 372$  kgs of cream.

So to have desired ratio of Fat to SNF, it is necessary to add 372 kgs of cream of 40% Fat in the 100000 kgs of 3.6% Fat milk.

#### **Proof of Example**

No.		Fat kgs	SNF kgs
1	Original Milk solids in kgs from 100,000 kgs milk with	3600.0	8510.0
	3.6 % Fat, 3.51% SNF		
2	Milk solids from added 372 kgs cream with	148.8	19.7
	40% Fat, 5.3% SNF		
3	Total solids in kgs	3748.8	8529.7
4	Ratio of Fat:SNF in Standardized milk	3748.8 / 8529.	7=0.4395:1

#### Use of following formula can also be made for calculation

$$C = \frac{(SNF \times R1) - F}{F1 - (SNF1 \times R1)} \times M$$

Where, C = Kgs of cream to add

M = Kgs of milk on hand

F = % Fat in milk on hand.

F1 = % Fat in Cream.

SNF = % SNF in milk on hand.

SNF1 = % SNF in cream from this milk

SNF2 = % SNF in skim milk from this milk

R1 = Fat / SNF desired

#### 7.1. Correcting the Fat Surplus Batch

The fat surplus is corrected by the addition of skim milk.

**Example**: We want 8.55% Fat, 28.00% TS in the finished product. Raw materials available are:

- (1) 100,000 kgs of milk having 4.2% Fat and 12.95% TS.
- (2) Skim milk testing 0.05% Fat.

How much skim milk must be added to provide the desired ratio of Fat to SNF?

#### **Solution**

The ratio of Fat to SNF desired in the finished product is

$$8.55 / (28.00 - 8.55) = 8.55 / 19.45 = 0.4395 : 1$$

The SNF content of the fresh milk = 12.95 TS - 4.2% Fat = 8.75% SNF with a fat content of 4.2% the SNF in the milk must be (4.2) / (0.5395) = 9.556%

Hence the deficiency of SNF = 9.556 - 8.75 = 0.806 %

The deficiency of SNF can be recovered by the addition of skim milk.

Skim milk is separated from 4.2% milk, hence it contains  $(100 \times 8.75) / 95.8 = 9.13 \%$  SNF

This skim milk contains 0.05% Fat. So we are adding both fat as well as SNF.

We must therefore reserve (0.05 / 0.4395) = 0.1138% SNF to balance the 0.05% fat contained in the added Skim milk.

This then leaves (9.13 - 0.1138) = 9.016 SNF available to cover the SNF shortage of 0.806 kgs per 100 kg of milk.

Therefore, for 100,000 kgs the requirement will be 806 kgs SNF.

The amount of skim milk needed therefore is  $(806 \times 100) / 9.016 = 8940 \text{ kgs}$  of skim milk.

So to have decided ratio of Fat to SNF in the finished product, 8940 kgs of skim milk must be added to 100,000 kgs of 4.2% Fat milk.

#### **Proof For Example**

No.		Fat kgs	SNF kgs
1	Original Milk solids in kgs from 100,000 kgs milk with	4200.0	8750.0
	4.2 % Fat, 8.75% SNF		
2	Milk solids from added 8930 kgs skim milk with	4.5	815.3
	0.05% Fat, 9.13 % SNF		
3	Total solids in kgs	4204.5	9565.3
4	Ratio of Fat:SNF in Standardized milk	4204.5 / 9565.3 = 0.4395:1	

#### Use of Formula

$$S = \frac{\frac{F}{R1} - SNF}{SNF2 - \frac{F2}{R1}} \times M$$

Where, S = Kgs of skim milk

M = Kgs of milk on hand

F = % of fat in milk

F2 = % of fat in skim milk

R1 = Ratio of Fat to SNF desired

SNF = % SNF of milk on hand

SNF2 = % SNF of skim milk from above milk

# 7.1.1. Forewarming

The standardized milk is Forewarmed / Pasteurized prior to condensing for purposes as discussed in working of the evaporator's part. Pathogens and potential spoilage organisms must be killed. Among the enzymes, milk lipase should primarily be inactivated; bacterial lipases are not inactivated and, if present, can cause severe rancidity. Deterioration caused by proteinases has not been reported. The heating intensity considerably affects viscosity and also age thickening and gelation of the product, so the actual heat treatment must be adjusted to these properties. UHT heating at about 130 to 140 °C is commonly applied.

# **7.1.2.** Homogenization:

Creaming is often not a major problem, and therefore homogenization is not always done. Currently, however, sweetened condensed milk is made less viscous (and exhibits less thickening) than previously. The density difference between fat globules and continuous phase is large, so homogenization is often done at low pressure, i.e., 2 to 6 MPa.

## 7.1.3. Addition of sugar

Sugar is added to the milk for the purpose of preserving the condensed milk without resorting to sterilization by heat. This can simply be added to the original milk. The amount added can be adjusted readily, and the sugar is pasteurized along with the milk. However, this procedure causes fairly extensive Maillard reactions during heating and evaporation, and above all, a faster age thickening. Alternatively, a concentrated sugar solution, which should be sufficiently heat-treated to kill any osmophilic yeasts, is added at the end of the evaporation step. The sugar should be refined and be devoid of invert sugar to prevent excessive Maillard reactions.

# 7.1.3.1. The Quality of Sugar desired

- (1) It must have known preservative property.
- (2) It must not be readily fermentable
- (3) It must have the power of inhibiting the activity of the germ life.
- (4) Its solubility at required concentration should be such that it has sufficient concentration to avoid super-saturation and crystallization within the range of temperature to which it is supposed to be exposed. Most commonly used sugar is sucrose.
- (5) The sugar used must convey to the milk flavour that is pleasing to the palate of the consuming public. Sucrose is used, which is properly refined, ferments with difficulty in concentrated solution. It inhibits the growth of bacteria and other ferments present in the milk. Its flavour has universal approval of the human palate.

## (6) **Dextrose** has **disadvantages** of

- a. Age thickening and
- b. Discolorations. So, for exporting or long period storage of sweetened condensed milk, it is not recommended.
- (7) The chief advantage of dextrose lies in its cheapness.

# 7.1.3.2. Quality and Care of the Sugar

- 1. Low grade sucrose is an unsafe product for use because it contains sufficient quantities of acid of invert sugar which damage the quality.
- 2. Sugar stock should be kept sealed in barrels and should be in a place that is kept clean and dry as sucrose has hygroscopic property. In the absence of above precaution there is danger of gassy fermentation.

# 7.1.3.3. Amount of Sugar / Sugar Ratio

- (1) Amount of sugar in sweetened condensed milk should be such that concentration will avoid activity of micro organisms.
- (2) It will be referred as "Sugar Ratio".
- (3) The sugar ratio is expressed in percent.
- (4) A 62.5% sugar ratio means that the water present in the sweetened condensed milk contains 62.5% of added sugar. This ratio of 62.5% is adequate to protect the product from age deterioration due to bacterial causes provided
- i. The milk is of good quality and
- ii. Is efficiently forewarmed in manufacture and there is
- iii. No post forewarming contamination.
- (5) This ratio will not prevent development of defects in condensed milk heavily contaminated with sucrose fermenting yeasts and molds.
- (6) Age thickening or thinning etc. will also not be avoided by this.

# 7.1.3.4. Method of Adding Sugar

The temperature and time of adding the sugar to the milk in the batch have a definite effect on keeping quality and age thickening of the finished product.

(1) The presence of added sugar in the fresh milk at the time of forewarming increases the heat resistance of the microorganisms and enzymes. So higher temperature for longer time has to be taken.

- (2) Furthermore, the presence of added sugar in milk at forewarming temperature decreases the physical stability and increases the tendency to age thickening of the finished product.
- (3) It is therefore recommended to dissolve sugar in water at 88<sup>o</sup>C or above making about 65% sugar solution and drawing this solution in to the pan separately during the condensing period. This eliminates stability weakening effect of sugar in the milk at high temperature.
- (4) In order to ensure freedom from foreign material, the syrup must be passed through pressure filter or a centrifugal clarifier.

# 7.1.3.5. Precaution when using Dextrose

- (1) The tendency of progressive thickening & discolouration of sweetened condensed milk containing dextrose can be largely avoided by dissolving dextrose.
- (2) When dextrose is used in place of part of the sucrose, there is definite tendency to
- a. Early discolouration and
- b. Age thickening of the finished product, unless special care is taken in adding dextrose.
- (3) These defects are due to a peculiar action of dextrose on the casein, forming a protein dextrose complex in hot water making 60 65% dextrose in water solution.
- (4) Forewarming the milk and the dextrose syrup separately and drawing the latter into the vacuum pan separately near the end of the condensing period helps in avoiding this defect.
- (5) The preparation and forewarming of the dextrose syrup should be done in the equipment that has been washed and rinsed completely free of milk remnants and of alkaline washing solution. It is also important that the water used to make the syrup, be on the acid side. In the case of hard water, it is advisable to add a small quantity of acid, such as acetic or hydrochloric acid, reducing the pH to  $\sim$  6.8 in the Bromo Thymol Blue test.

# 7.1.3.6. Amount of sugar to be added

Suppose we want 8.5 % Fat and 30 % of milk solids in finished product with 60 % sugar ratio, then the above combination of milk solids and sugar ratio yields a sugar content in the finished product of:

$$\{(100-30) / 100\} \times 60 = 42 \%$$

## Example 1

Condensed milk contains 30% TS and 43.75% added sugar. Find out the sugar ratio?

Answer:

In (100 - 30) = 70, i.e. 70 parts of water, sugar is 43.75 parts.

Therefore, Sugar Ratio =  $(100 \times 43.75) / 70 = 62.5$ .

# Example 2

Condensed milk contains 30% TS and the sugar ratio desired is 62.5%. What must be the % of sugar in condensed milk?

Answer:

In 100 parts of water 62.5 parts of sugar is desired.

Therefore, in (100 - 30) = 70 parts water sugar required will be  $(70 \times 62.5) / 100 = 43.75 \%$ .

# Example 3

Condensed milk containing 8.5% Fat, 21.5% SNF and 43.75% sugar is required. The fresh milk standardized to the above ratio of Fat to SNF contains 3.47% Fat and 8.78% SNF. How much sugar must be added to the fresh milk to give the condensed milk a sugar content of 43.75%?

Answer:

= 17.86 kgs / 100 kgs of milk

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# Lesson-8

# **Manufacture of Sweetened Condensed Milk- Operations**

## 8.1. INTRODUCTION

Concentration for manufacture of sweetened condensed milk is usually done by evaporation. A falling-film evaporator is generally used to remove the bulk of the water and a circulation evaporator to remove the remainder. Relatively high temperatures (up to 80 °C) are often applied, which implies a lower viscosity in the evaporator but a higher initial viscosity of the final cooled product. The low water content of the sweetened condensed milk implies a high viscosity and boiling point. Evaporation in continuously operating equipment with many effects is, therefore, not easy. Fouling, thus, readily occurs. It is difficult to accurately adjust the desired water content, which is mostly monitored by means of refractive index.

# 8.2. Striking the Batch

The term "Striking" refers to the drawing of a sample from the pan and testing it for density. When the boiling milk approaches the desired concentration, there are various indications that suggest to the experienced pan operator that the milk is nearly 'done':

- § The general behaviour and appearance of the milk is characteristically different.
- § The milk has "settled down" to a quite boil.
- § Its surface assumes a glossy glistening luster and
- § There is heavy roll from periphery towards centre of the pan, forming a small 'puddle' of foam in the centre.

# 8.3. Method of Sampling

Numerous sampling devices have been developed that make possible the rapid drawing of samples representative of the boiling milk in the pan without interrupting or disturbing pan operations.

# 8.4. Temperature of Sample for Density Tests

The result of the density test is influenced by the temperature of the sample at the time of the test. For accurate and comparable results, therefore, the adoption of a standard temperature is indispensable. Towards the end of the condensing period, the pan temperature usually drops to near 49°C. Therefore, a standard testing temperature adopted is 49°C and the sample used is to be taken accurately. For accurate density determinations, the temperature of the sample should therefore be taken at the time of the density test. If it varies several °C from 49°C, it should be adjusted to the standard temperature or if this is not practicable, the density readings should be corrected by applying the proper temperature correction factor.

## 8.5. Density Tests

The density tests that have been found most practical for use at the pan are

- (1) Judging Density by appearance to eye
- (2) The Picnometer test and
- (3) The Hydrometer test.
- (4) Refractometer test
- (5) Viscometer test.

# **8.5.1.** Temperature Correction Factor

If the temp of the sample at the time of the density test varies from the adopted standard temperature, the Baume's reading may be corrected to what it would be at the standard temperature by multiplying the temperature deviation. If the temperature of the sample is above the standard temperature, the product is added to the observed Baume's reading. If the temp of the sample is below the standard temperature (120°F or 48.9°C), the product is deducted from the observed Baume's reading.

# 8.5.2. Formula for Calculating the Correct Baume's Reading

The Sp. Gr. of sweetened condensed milk of any composition may be calculated by dividing the figure 100 by the sum of the quotients that result from dividing the % of each group of ingredients by its respective Sp. Gr. of sweetened condensed milk at 60  $^{0}F(15.6^{0}C)$ . It is thus represented by following formula:

Specific Gravity of Sweetened
Condensed Milk at 60 
$$^{\circ}$$
F (15.6 $^{\circ}$ C)
$$= \frac{100}{\% \text{ Fat}} + \frac{\% \text{ SNF}}{\% \text{ SNF}} + \frac{\% \text{ Sugar}}{\% \text{ Sp.Gr.}} + \frac{\% \text{ Water}}{\% \text{ Sp.Gr.}}$$

## 8.2.1. Conversion of Specific Gravity to Baume's Degrees

The composite Sp. Gr. values of the milk thus obtained by the use of the above formula are then converted to Baume's Degrees (<sup>0</sup>Be) by means of the following formula:

$$^{0}$$
Baum'e at 60  $^{0}$ F (15.6 $^{0}$ C) = 145 -  $\frac{145}{\text{Sp. Gr. at 60 }^{0}$ F (15.6 $^{0}$ C)

This is then corrected to the standard temperature used for the hydrometer test of the pan sample by the "Temperature correction factor".

# 8.2.1. Specific Gravity Values for Sp. Gr. Formula

The constituents or groups of constituents, the Sp. Gr. of which determine the composite Sp. Gr. value of sweetened condensed milk consist of the Fat, SNF, added sugar or sucrose and the water. Their respective sp. Gr. at  $60^{\circ}$ F (15.6°C) are:

Fat	0.930
MSNF	1.608
Sucrose	1.589
Water	1.000

# 8.2. Finishing the Batch

When the desired density has been reached, the condensing process is stopped in the following order:

- § All steam to the pan is shut off,
- § The valve in the water line to the condenser is closed,
- § The vacuum pump is stopped and
- § The vacuum relief valve is opened.

The above operations should be carried out in the order named to prevent milk from burning on to the heating surface and condenser water from flooding the pan. When the vacuum has been dissipated, the condensed milk is then drawn from the pan. This should be done promptly so as to avoid super heating due to the static status of the hot milk in the pan, which has a tendency to accelerate age thickening especially in the case of milk of naturally unstable viscosity.

## 8.3. Final Standardization

The fat, SNF, and the added sugar are already present in their desired ratio to each other. The third and final standardization, therefore, has to do only with the adjustment of the % of total solids, if such adjustment is necessary or desired.

# 8.4. Cooling of Sweetened Condensed Milk

It is one of the important steps in manufacture of sweetened condensed milk. It is absolutely essential to cool the product promptly to desired temperature because i n these steps, formation of large lactose crystals must be avoided. Consequently, seed lactose is added. Before that, the condensed milk must be cooled to a temperature at which lactose is supersaturated so that the seed lactose does not dissolve. However, the temperature must not be so low that spontaneous nucleation can occur before the seed crystals are mixed in. After seeding, cooling should be continued to crystallize the lactose.

If we do not cool down quickly

- (1) There is tendency of discoloring and
- (2) Tendency of age thickening during storage.

Cooling will also help in

- (1) Formation of smoother texture in finished product and
- (2) Prevent objectionable sugar deposit.

## 8.4.1. CRYSTALLIZATION

The relative smoothness of product will largely depend upon number and size of crystals present. Our main aim is not to prevent the formation of crystals but our main aim is to form more crystals having smaller size. In this process major constituent responsible is lactose because it has low solubility than sucrose. Also crystals formed are hard and thick. In case of sweetened condensed milk, the rate of lactose crystallization is further impeded (retarded) by

- (1) The milk colloids and
- (2) The higher viscosity which reduces the rate of diffusion.

Lactose is a disaccharide and reducing sugar. It is not very soluble and not so much sweet as sucrose. It is fermented by lactic and acidophilus organisms etc. The concentration of this sugar varies from 48-50 gm/liter. In solution, lactose exists in two forms i.e.  $\alpha \& \beta$  forms. In crystalline form, three forms are available:

- 1.  $\alpha$  lactose hydrate.
- 2.  $\alpha$  lactose anhydrate

# 3. $\beta$ - lactose anhydrate

Solubility of lactose is low at room temperature in pure water. Its solubility is 18 parts/100 parts.  $\alpha$  &  $\beta$  lactose in sweetened condensed milk are in definite proportion and when these proportions are changed, they change their forms and again form lactose equilibrium. This change is very slow. So lactose present in sweetened condenses milk takes more time for crystallization. The solubility of these two forms is also varying and  $\alpha$  -lactose has lower solubility as compared to  $\beta$  -lactose. So  $\alpha$  -lactose will crystallize first. So equilibrium will be changed and  $\beta$  -lactose will turn to  $\alpha$  -lactose which will take time and crystallization process will be slow.

# 8.4.2. Forced- Crystallization – Importance of Mass Crystallization in Cooling

The problem of ensuring permanently smooth texture in the finished product is not a problem of preventing the formation of lactose crystals during the cooling process. It is a problem of preventing the crystals that are present at the conclusion of cooling process from subsequently growing larger. This is accomplished by providing condition at the conclusion of the condensing period, i.e. in the cooling process that produce mass crystallization. A multitude of crystal nuclei i.e. small crystals, great enough to reduce the state of supersaturating to a state of saturation must be formed while the crystals are still exceedingly

small. This eliminates sugar in solution in excess of maturation and thereby the possibility of objectionable further crystal growth after completion of the cooling and agitation period.

# 8.4.3. Forced crystallization period

The purpose of this period is to provide for mass crystallization. It is a period in cooling process when condensed milk has reached to the temperature which will provide super saturation of lactose with minimum viscosity and such temperature at which the condensed milk is held at the optimum temperature of seeding. The optimum temperature for forced crystallization at which the super saturation takes place varies from 40 - 85.6  $^{0}$ C. In our country, it is varying between 32.2 - 85.6  $^{0}$ C, depending upon original lactose content. The milk should be held at this temperature for about 60 minutes, under vigorous agitation after which it is cooled rapidly to the final temperature. For best results, at the end of the condensing process, the hot condensed milk should be cooled from pan temperature to seeding temperature as rapidly as possible, to prevent slow initial crystallization such as would yield a relatively small number of crystals, designed to grow to relatively larger size, giving the finished product a coarse sandy texture. For milks of fairly normal concentration, the optimum temperature for forced crystallization ranges from  $30-40\,^{0}$ C.

## 8.4.4. Purpose of Seeding

The purpose of seeding is to give the lactose present in super saturation form an additional incentive for crystallization. When condensed milk at optimum temperature is seeded with properly prepared lactose in presence of vigorous agitation, numerous crystals of lactose of uniform size will be formed. This operation should be continued for about one hour. The number of crystals centers present at any time, that are capable of developing successively to crystal nuclei and to crystal is a function of degree of super saturation and that the existing crystal centers will increase with the degree of super saturation. In the absence of crystal nuclei, or because of difficulty of migration of particles to existing nuclei, lactose even in highly super-saturated solution may refuse to crystallize or will crystallize only very slowly.

In addition, seeding at optimum temperature for mass crystallization with properly prepared seed lactose, and in the presence of vigorous agitation, yields crystals of uniform size. Literally born at or near the same time, they get their start under identical conditions of time and degree of super saturation with respect to lactose and of viscosity of milk and they are checked in their growth at the same time that is when super saturation has been dissipated and scattered. Crystals of larger size will be few in numbers, being limited largely only to crystals that were already present in the hot milk in the vacuum pan and at any other time before the seeding period.

## 8.4.5. Seed Material

The seed material commonly used is powder of lactose hydrated. Lactose should be of smallest possible size preferably 200 mesh (  $<10~\mu$ ) having sharp crystal edges. To avoid contamination of product, the lactose used for seeding should be sterilized under vacuum at 93 $^{\circ}$ C which will convert lactose hydrate to anhydrate. Then it is ground to fine particles in a perfect pulverized mill, filling in the can and sealing it. Then sealed can is sterilized at 130 $^{\circ}$ C for 1-2 hours.

The smaller size and sharper particle edge of the seed material have advantages such as:

- § Greater incentive to the formation of crystals centers, and crystal nuclei
- § It brings about mass crystallization
- § More numerous and smaller permanent crystals and
- § Smoother texture of the finished product.

# 8.4.6. Amount of Lactose to be used

It is always desirable not to use more seed material than necessary for optimum mass crystallization. When using ground lactose of 200 fine mesh, than 37 - 50 gm of lactose per 100 kgs of original fluid milk will be sufficient.

# 8.4.7. Adding the Seeding Material

The seed material is not added in dry form to batch. Precaution should be taken that it should not form lump into large aggregate which will not disaggregate. In that case the seed material is incapable to initiate mass crystallization. To obtain the desired result, it is usually dissolved in sweetened condensed milk in a suitable sterilized container. Stir the product till all the lumps disappear and form homogeneous mixture. This mixture is added slowly to the whole batch while stirring vigorously and keeping temperature constant Agitation is continued for 50 minutes. Agitation plays on important role because it promotes and increases the rate of crystallization. Such agitation facilitates active migration of the particles to the crystal nuclei in this product. In addition, vigorous agitation is necessary from beginning to the end of the cooling process in order to cool all parts of the batch as much as possible to the same temperature. The usual seeding temperature is 27 - 39  $^{0}$ C for cow's milk and for buffalo milk it ranges from 30 - 36  $^{0}$ C.

After crystallization is over, lower down the temperature to room temperature. This cooling is done by several methods. After cooling, it is again stirred for 20 minutes because, due to lower temperature, solubility decreases and there is possibility of crystallization.

# 8.4.8. Packaging Sweetened Condensed Milk

Packaging in cans is common. The cans are then covered with a lid and the seams are sealed. Cans and lids are first sterilized. The packaging room is supplied with air purified through bacterial filters. Sweetened condensed milk reaches the market in the form of bulk good and also in different size retail packs. It is important to fill the cans full in order to exclude as much of the air from the container as possible. For sanitary reasons the filling machines should be emptied and washed after each day's use. It is important to close the tins immediately after filling. If they are left open their contents are exposed to air and light and prolonged exposure may cause the surface to crust over and to develop a tallowy flavor. It may also invite contamination with insets. The seal must be air tight and sufficiently rigid to withstand a rough handling.

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## Lesson-9

# **Manufacture of Evaporated Milk**

## 9.1. INTRODUCTION

Evaporated milk is sterilized, concentrated, homogenized milk. The product can be kept without refrigeration and has a long shelf life; it is completely safe for the user. After dilution, flavor and nutritive value of the product are not greatly different from that of fresh milk. A major problem with sterilization is the heat stability; the higher the concentration of the milk, the lower its stability. That is why concentrating cannot be by more than about 2.6 times, which corresponds to a level of about 22% solids-not-fat in the evaporated milk.

Currently, bottled evaporated milk is often used in coffee in certain countries. It can be added while cold because a fairly small amount is involved as compared to non-evaporated milk. After the bottle has been opened, the milk can be kept in a refrigerator for up to 10 days because it initially contains no bacteria at all and because contaminating bacteria grow somewhat more slowly owing to the reduced water activity, which is about 0.98.

# Fig. 9.1: Manufacture of Evaporated Milk

Milk received at the reception dock is inspected, weighed and sampled and then is pumped through filter and cooler into holding tanks where it is standardized to the desired ratio of Fat to SNF. From the holding tank, it is pumped through fore-warmer into hot well. From there the hot milk is drawn into vacuum pan and the condensed milk is discharged from the bottom of the pan through sampling valve into drop tank as shown in Fig.9.1.

The condensed milk may pass to homogenizer without the use of a drop tank but the availability of a drop tank may prove helpful in case of accidental interruption in the flow of the milk between pan, homogenizer and storage tanks and especially when using the batch system of condensing.

From the drop tank, the condensed milk passes through homogenizer over cooler into storage tank where it is standardized for total solids and held cold until ready to be tinned. The standardized milk made homogeneous through agitation in the storage tanks is then forced from these tanks by pump through evaporated milk warmer into a supply tank that feeds the filling machines by gravity. From the filled and sealed tins reconvened through a leak detector to the sterilizer where they are subjected to steam under pressure destroying the bacteria contained in the milk and giving the evaporated milk necessary keeping quality and desired consistency.

# 9.1. Inspection and Treatment of milk before Manufacture

Besides inspection by the senses of smell, taste and sight, the use of rapid chemical test adopted to the routine of the receiving platform, such as the acidity test, the direct microscopic count and M.B.R. test, the alcohol test and the phosphate test have proved helpful. Variation of the quality of the milk has a direct effect on heat stability of evaporated milk and thus indirectly also influences the viscosity of the finished product.

#### 9.2. Standardization of Fat to SNF desired

The milk that has passed inspection is then dumped into the weigh tanks and after sampling and recording weights; it is dropped into supply tank from which it is pumped over or through a cooler into holding tanks. There, it is tested for fat and TS and standardized to the ratio of Fat to SNF desired in the evaporated milk. The addition of fat in the form of cream or melted butter to the cooled homogenized evaporated milk in the storage tank (if standardization is done after manufacture) would require re-homogenization after such standardization in order to prevent objectionable fat separation after sterilization. Also see Sweetened Condensed Milk.

## 9.3. Addition of Casein Stabilizers:

In factories where casein stabilizers such as disodium phosphate or Na-citrate are used for control of heat coagulation in the sterilizer, it is considered good practice to add at least a portion of the required quantity anticipated to the fluid milk before manufacture.

## 9.4. Fore-warming of Milk for Evaporated Milk

**Preheating** serves to enhance the heat stability of the evaporated milk, inactivate enzymes, and kill microorganisms, including a significant proportion of the bacterial spores present. Here the destruction of biological agencies injurious to health or damaging to keeping quality, that might be present in the raw milk, is the function of the process of sterilization by steam under pressure. However, aside from this, the fore-warming is indispensable for satisfactory vacuum pan operation also because

- (1) The fore-warming process is one of the most important steps in the manufacture, upon which the heat stability of evaporated milk depends.
- (2) Through their effect on heat stability, variation in fore-warming procedures has a definite effect on the viscosity of the evaporated milk.
- (3) Increasing the temperature of fore-warming and limited increase of the period of exposure to the fore-warming temperature diminishes viscosity and increases the stability of the milk towards sterilizing heat.

# 9.4.1. Temperature of Fore-warming

The heating temperature—time relationship is usually selected on the basis of heat stability. Formerly, a long heat treatment (e.g., 20 min) at a temperature below 100°C was often applied. Currently, UHT treatment is generally preferred. It reduces the number of spores in the milk considerably, and therefore a less intensive sterilization suffices.

The fore-warming temperature commonly used in USA ranges from ~ 93°C to boiling with an approximate exposure of 10-25 minutes. However, an increase in milk solids lowers the stability of evaporated milk. In addition, the higher concentration definitely increases the danger of objectionable cooked flavour and darkening of colour. It also tends to cause deposits of crystals of Ca-citrate and of other mineral salts in the container.

Low heat stability can no longer be considered a factor which might limit evaporated milk to 26% solids content. From a heat stability point view point, it should be commercially possible to manufacture evaporated milk of ~ 32% TS content. A good grade of milk should be quickly fore warmed to about 120°C and held 2-4 minutes, then drawn into the vacuum pan and handled in the usual way. For the usual type of normal milk supply, fore-warming temperature below the boiling temperature (within the approximate range of 87-99 °C for 10-15 minutes) are considered adequate to produce desired heat stability in evaporated milk and sterilized by the conventional process of 115°C for 18-20 minutes.

It should be clearly recognised that each milk supply has its own peculiarity as to optimum forewarming temperature or treatment for accomplishing the viscosity and desired body.

# 9.4.2. Seasonal Adjustment of Forewarming Temperature:

The milk coming in flush season has highest heat stability and is lower during early stage and later stage of milkings due to change in salt content and albumin content (higher). Therefore, it becomes necessary to change forewarming temperature. A higher forewarming temperature (120°C, coming up time to seconds and holding time 3 to 4 minutes) will make up the lowering of heat stability due to late and early lactation milk.

# 9.4.3. Equipment for Forewarming Evaporated milk:

The equipments used in condensed milk can be safely used for evaporated milk. However, while for the manufacture of highly viscous sweetened condensed milk, the batch system has been found most suitable, Evaporated milk with its much fluid consistency tends it self advantageously to continuous

Table-9.1: Equipments used for forewarming

1	Hot Well	a. By injection of live steam.
		b. With steam jacket supplemented by the steam.
2 Continuous flow Heaters		a. With flash pasteurizer supplemented by live steam.
		b. Plate heat exchanger.
		c. Single Pass Tube Heater.
		d. Recirculation tube heater.
		e. Multi Velocity tube heater.
3 Forewarming Above 100°C	a. Closed Pressure Tank	
		b. Tubular pressure heater.
		c. Live Steam Injector.
4	Two Stage Forewarming	<ul> <li>Here warming is done at 87.8°C and milk is held for 15 minutes in first stage heater after which it is pumped through the second stage heater which raises the temperature the 112.8°C.</li> <li>Uses of Ste-Vac heater report practically to build-up of milk solids in second stage high temperature milk tube.</li> </ul>
5	B.D.I. (Bureau of Dairy Industry) High Temperature continuous Heater	■ Even temperature can be raised to 140.5 – 150°C in 1.5 – 3.0 sec.

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# 9.1. Condensing of Evaporated Milk:

The forewarmed milk is condensed to desired concentration in the vacuum pan or evaporator in a similar manner as done in the case of SCM. Evaporated milk boils at some what more vigorously and needs somewhat closer watching because of absence of the stabilizing influence of sugar. Its rate of evaporation especially toward the end of condensing period is slightly faster than that of more sluggish sweetened product. Preparation, starting and operation of the pan is quite similar as in the case of SCM.

## 9.2. Ratio of Concentration:

On the bases of fluid milk with an average total solids content of 12.25%, the ratio of concentration of evaporated milk is applied as follows:

PFA Standard E.M. (26% TS) = 
$$\frac{26.00}{12.25}$$
 = 2.1224:1

# 9.1. Striking the Batch:

The striking or sampling and testing for density of evaporated milk is more easily accomplished than that of viscous syrup SCM. When evaporated milk approaches the desired concentration, its consistency resembles that of rich milk or thin cream, its fluidity, therefore, ensures ready equilibrium of the hydrometer.

The Baume' hydrometer, with a scale of range from 0 to  $15^0$ Be subdivided into 1/10 degrees is commonly used for testing the pan sample. Establishment of standard striking temperature helps to avoid misleading results. A convenient temperature is the pan temperature which near the finish of the batch usually ranges from approximately  $48.9 - 54.4^0$ C.

# 9.2. Temperature Correction Factor:

If the temperature of the sample used for the density test varies from the adopted standard temperature, correction of Baume' reading to standard temperature may be made by multiplying the temperature deviation by the factor 0.031 for each degree of deviation. If the temperature of the sample is **above** the standard temperature the product is **added** to the observed Baume reading. If the sample is **below** the standard temperature the product is **deducted** from the observed Baume reading.

**Example:** The temperature at which the Baume reading was taken was 130° F and the reading was 6.7° Be. What is the Be reading at 120° F?

**Answer**: 
$$6.7 + (0.031 * (130-120) = 7.01^{0}$$
 Be at  $120^{0}$  F.

# Formula for Calculation of correct Hydrometer Reading for Desired Concentration:

The specific gravity of evaporated milk of any desired % composition may be calculated by the following formula:

Sp. gr. of Evaporated milk at 
$$60^{\circ}$$
 F = 
$$\frac{100}{\% \text{ Fat}} + \frac{\% \text{ SNF}}{\text{Sp. Gr.}} + \frac{\% \text{ Water}}{\text{Sp. Gr.}}$$

Sp. gr. values that were adopted for the milk constituents of SCM. also apply to evaporated milk. They are: Fat = 0.93 & SNF = 1.608

**Example**: Evaporated milk with a composition of 7.9% fat and 26% Total solids is desired. What is the correct Baume hydrometer reading at 120° F?

#### **Answer:**

$$SNF = 26 - 7.9 = 18.1\%$$

Water = 
$$100 - 26 = 74.0\%$$

Sp. gr. of Evaporated milk at 
$$60^{\circ}$$
 F =  $\frac{100}{7.9} + \frac{18.1}{1.608} + \frac{74}{1}$ 

= 1.0667

Baume reading at  $60^{\circ}$  F = 145 - (145 / 1.0667) = 9.07

Baume reading at  $120^{\circ}$  F = 9.07 - (0.031 \* (120 - 60)) = 7.21

**Example:** What will be the Baume reading at 120<sup>o</sup>F if desired TS content is 31% & Fat is 9%.

# **Answer:**

Sp. gr. of Evaporated milk at 
$$60^{\circ}$$
 F =  $\frac{9}{0.93} + \frac{22}{1.608} + \frac{69}{1}$ 

= 1.195

Baume reading at  $60^{\circ}$  F = 145 - (145 / 1.955) = 13.71

Baume reading at  $120^{\circ}$  F = 13.71 - (0.031 \* (120 - 60)) = 11.85

The milk is condensed to desired composition which is evaluated by Baume reading. If the milk is standardized before it is condensed than there is no problem but some standardize it after condensing because they over condense the previously standardized milk and then add calculated amount of water to get the desired composition. All standardizing is done in storage tank.

# 9.1. Homogenization:

*Homogenization* serves to prevent creaming and coalescence. It should not be too intensive because the heat stability becomes too low. Precisely following is the effect of homogenization on the milk:

- 1. The homogenizer reduces the fat globules to such small size that majority of globules are no longer in a position to respond to force of gravity.
- 2. Homogenization increases the viscosity of evaporated milk. This further impedes particle movement other than Brownian movement, enhancing emulsion stability of fat globules.

Product should be homogenized at  $49^{\circ}$ C or above to avoid fat clumping. Two stage homogenizer is advisable at first stage  $140 - 175 \text{ kg/cm}^2$  and at second stage  $35 \text{ kg/cm}^2$ .

## 9.2. Effect of Homogenization on Viscosity of Evaporated Milk:

Homogenization increases the viscosity of evaporated milk. This means that the resulting smaller fat globules must overcome a greater resistance if they are to move upward. The cause of the increased viscosity of homogenized milk has not been fully explained, but it is believed to be due to the increased absorption of casein to the much larger surface area of the finely dispersed fat globules thereby considerable augmenting the volume of the dispersed phase and diminishing the volume of the continuous phase. A change in the physical properties of the casein may also be involved in the effect of homogenization on viscosity. Homogenization definitely increases the viscosity of evaporated milk and does so increasingly with increasing pressure.

# 9.3. Effect of Homogenization on Heat Coagulation of Evaporated Milk:

While homogenization increases the viscosity of evaporated milk, its effect on heat stability is slight in the case of milk of otherwise normal heat stability. Increase in pressure up to 211 kg/cm² slightly decreases the heat stability. But above 211 kg/cm² there is definite decrease in heat stability. In case of unstable evaporated milk however, such as milk containing developed acidity due to age or improper care of the fresh milk or high total solids milk, high homogenizing pressure definitely lowers the heat stability point and may cause serious curdling difficulties in the sterilizer.

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## Lesson-10

# Pilot Sterilization and Heat Stabilization for Evaporated Milk

## 10.1. INTRODUCTION

To ensure that the evaporated, homogenized milk does not coagulate during sterilization and at the same time does acquire a desirable viscosity, a series of sterilization tests is often done on small quantities of the evaporated milk to which varying amounts of a stabilizing salt (for the most part, Na<sub>2</sub>HPO<sub>4</sub>) are added. The tests are needed because variation occurs among batches of milk. Essentially, the addition of the salt means adjusting the pH. Because further processing must be postponed until the test results are available, this necessitates cooling the evaporated milk after its homogenization and storing it for a while. However, long-term storage should be avoided to prevent bacterial growth; moreover, cold storage of the milk increases the tendency of age thickening. The stabilizing salt is added as an aqueous solution, which dilutes the evaporated milk slightly. Therefore, the milk is often concentrated somewhat too far and is re-standardized to the correct dry-matter content during stabilization.

## 10.2. PILOT STERILIZATION

The purpose of this method of technical control of evaporated milk is to have control on sterilization process by controlling heat stability and viscosity of finished product . It makes possible

- (1) The adoption of standardized process of sterilization designed and adopted for evaporated milk of superior quality for process. This process provides a very narrow range of variation of temperature and of time exposure, in order to limit the personal factor with its inevitable uncertainties to the minimum.
- (2) A standard method of determining by means of a pilot sterilizer, a viscosimeter and a colour test, the proper viscosity and colour that the evaporated milk should have when it comes from sterilizer.
- (3) A standard method of determining the amount of stabilizer that must be added to any given batch of EM in case its properties are such that it is unsafe to subject it, without such treatment to the temperature conditions that fall within the range of the standardized process of sterilization.

## 10.3. Use of Stabilizer:

The amount of stabilizer to add to any given batch of Evaporated Milk is determined by trial. A number of sample cans are used and the test is made in the pilot sterilizer. The samples are prepared by transferring various accurately measured amount of the stabilizer solution to individual sample tins which are subsequently filled with exact weight of the evaporated milk to be tested.

From the research of Sommer and Hart, it is found that most cases of unbalanced salts in Evaporated Milk, a range of 6-62.5 gm of dry citrate or phosphate / 100 kgs of evaporated

milk will cover the amount required to restore the balance and to accomplish a satisfactory sterilizing process. But some times isolated samples also require as high as 125 gm /100 kgs of evaporated milk.

# **10.4. Preparation of Stabilizer Solution:**

The citrate or phosphate is added in the form of a solution. The strength of the solution should be such that one tenth of a cubic centimeter of a solution is equivalent to the addition of 6.25 gm of the dry salt / 100 kgs of EM. This requires a solution containing 10.63 gm of dry salt dissolved in enough water to make up 100 ml of solution that had been added to the 170 ml can, the inspection and test of which show the viscosity desired.

# 10.5. Preparation of Sample Tins

Use 1 ml pipette. In order to prevent loss of solution due to some of it being blown out with the air that escapes past the tin of pipette, it is best to add the solution to the Cans before they are filled with the milk.

# 10.6. Compensating For Effect of Dilution of Sample

The addition of a salt solution dilutes the EM. This factor alone has a slight influence on the heat coagulation point of the milk. In order to eliminate this factor it is advisable to equalize the dilution in all samples of a given series.

#### 10.6.1. Process

Milk giving alcohol negative test, after standardization to Fat/SNF = 8/18, forwarmed to >90 C for a period of 10 minutes and concentrated to 26-28% milk TS, homogenized to 100 Kg/Sq.cm at  $55-60^{\circ}$  C temperature is used for Pilot Sterilization using following procedure:

- 1. Prepare a number of sample test bottles and proceed as shown in Table 10.1.
- 2. Prepare a 10 % solution of stabilizer (by dissolving 10 gms of dry stabilizer in enough water to make 100 cc of solution).
- 3. Add stabilizer solution and distilled water to evaporated milk bottles as per table given.
- 4. Fill the sample bottles containing the measured portion of stabilizer solution with evaporated milk from the batch and seal them.
- 5. Place the sealed bottles in the Pilot Sterilizer and subject them to a heat treatment for 15 minutes at 116-117<sup>0</sup> C with a 15-20 minutes coming up time.
- 6. Cool the bottles rapidly to 24<sup>0</sup> C with help of air flow.
- 7. Examine the bottle contents for smoothness and then test for viscosity.
- 8. Select the bottle whose contents show a desirable body and smooth texture.
- 9. Determine whether stability correction is necessary.

- 10. Based on the correction corresponding to the Pilot Test (to give the best viscosity), calculate the total quantity of stabilizer required for the bulk of the evaporated milk to be prepared.
- 11. Just add enough water for complete solution of the stabilizer in a vessel. Add the mixture slowly to the evaporated milk in the storage tank, ensuring thorough mixing during addition and for several hours after its solution.
- 12. For subsequent batches of evaporated milk add a portion of the required amount of stabilizer (determined by previous day's experience) to the standardized fluid milk at the forewarmer and then complete the correction by adding the remainder to the evaporated milk in the storage tank.

# Method or preparing solution

Table-10.1: Concentrations of stabilizer solution used for Pilot sterilization test

		1	2	3	4	5	6	7
170 gm of Un-sterilized EM Plus	10.63% Na <sub>2</sub> HPO <sub>4</sub> solution in ml	0.0	0.0	0.2	0.4	0.6	0.8	1.0
	Distilled water in ml	0.0	1.0	0.8	0.6	0.4	0.2	0.0
Rate of addition required a Na <sub>2</sub> HPO <sub>4</sub> / 100 kgs of Un-s	and expressed as gm of dry	0	0	2	4	6	8	10

OR

Amounts and their concentrations of water and stabilizer to be used for Pilot Sterilization test using 195 gm Condensed milk in 200 ml bottles:

Bottle	Water,	Disodium hydrogen	DSP	DSP in Condensed	Remarks
No.	ml.	orthophosphate (DSP)	in ml.	milk, %	
		(10 % Sol.), ml			
1	Nil	Nil	Nil	Nil	
2	5	0	0	0	
3	4.5	0.5	0.05	0.025	
4	4	1	0.1	0.05	
5	3.5	1.5	0.15	0.075	
6	3	2	0.2	0.1	
7	2.5	2.5	0.25	0.125	
8	2	3	0.3	0.15	
9	1.5	3.5	0.35	0.175	
10	1	4	0.4	0.2	
11	0.5	4.5	0.45	0.225	
12	0	5	0.5	0.25	

In table, the first sample represents the evaporated milk without any addition. The second sample contains no stabilizer but represents the milk in dilute form, the rate of dilution being the same as that of all the other samples. The remaining samples represent addition of stabilizer in increasing amounts but with the dilution in all the cases equal to the second sample. By this procedure, it is possible to determine to what content the improvement in heat stability is attributed to the dilution and to the stabilizer respectively.

## **10.2.1.** Sterilizing the Sample

The sample content determining the measured portions of stabilizer solution are now filled with evaporated milk from batch. They are sealed and kept in pilot sterilizer where they are subjected to heat treatments. A heat treatment is given by bringing the temperature from 110 to 117°C which is brought up in 2 minutes. The milk is held at the temperature for 15 minutes. The bottles then are cooled rapidly by opening exhaust and cold water intake valve. As temperature influence the viscosity, it is advisable to cool it to standard 24°C. As soon as cooled, the bottles are opened, examined for smoothness and colour and tested for viscosity.

# 10.2.2. Adding the Correct Amount of Stabilizer to the Batch:

The stabilizer should be added to the evaporated milk in the form of a solution using just enough water for complete solution of the crystals. The mixture should be added slowly, keeping the evaporated milk thoroughly agitated both, during addition and for several hours after its solution

The casein – stabilizing reaction of the Na-salt is most pronounced when the added stabilizer is in the milk during forewarming and condensing. It has been found advantageous, therefore, to add at-least a portion of the required amount to the milk at the forewarmer and then complete the correction by adding the remainder to the evaporated milk in the storage tank.

# 10.3. Factors Influencing the Viscosity

# 10.3.1. Factors that increase viscosity and curdling Tendency:

- 1. Presence of developed acidity.
- 2. High Protein Content.
- 3. Excess or Deficiency of Ca<sup>++</sup> in Ca-casein combination
- 4. Presence of Bacteria producing rennet like substance.
- 5. Low forewarming temperature
- 6. High Concentration.
- 7. High homogenizing pressure especially in case of milk of inferior quality.

# 10.3.2. Factors that Decrease the Viscosity:

- 1. Absence of developed acidity.
- 2. High temperature quick heat, short holding heat treatment in forewarming
- 3. Low concentration.
- 4. High temperature quick heat, short held heat treatment of concentrated milk
- 5. Optimum Ca ++ casein balance.

# 10.4. Canning:

**Packaging** in cans is common. Evaporated milk intended for use in coffee is usually packaged in bottles that are closed with a crown cork or a screw cap. Can should meet all the sanitary standards and its metal should be non-toxic to the product or should not impart any flavour in it. The evaporated milk container must withstand the pressure changes in the process of sterilization. Its seal must be absolutely air- tight and it must be strong.

# a) Temperature of Milk:

The temperature of evaporated milk at the time of filling needs attention because it is a factor affecting the tendency of milk to foam. Foaming can seriously interfere with proper filling of the Cans. In addition, excessively low temperature increases the tendency of flapping. The most effective temperature for filling is considered to be  $4.4^{\circ}$ C.

#### b) Leak Detection:

Immediately after sealing and before they leave the last turn table of the sealing machines, the Cans are inspected for visible defective seals. In order to minimize loss of Cans and contents due to leaks in any part of Can, in the seams as well in the seals it is important to test all Cans by means of leak detector before they reach the sterilizer. Most commonly can pass submerged through hot water bath and expansion of the air in the Can cause slight pressure. In case of leaky can, the pressure expands some of excess air. The escaping air percolates upward in water in the form of air bubbles that are readily seen.

# 10.5. Sterilization of Evaporated Milk Cans:

In-bottle or in-can sterilization can be applied batch wise (in an autoclave) or continuously. Machines that have rotary air locks (to maintain the pressure) may be applied for cans and hydrostatic sterilizers for bottles.

The filled and sealed cans that have successfully passed inspection for freedom from defective seals and seams are now ready for sterilizer. If they cannot be sterilized within an hour or two, they should be held under refrigeration at  $4.4 - 7.2^{\circ}$ C. This precaution is especially important during hot weather season.

# a) Purpose of Sterilization:

- (1) The primary purpose of sterilization is to destroy all germ life and enzymes present therefore preserving the product permanently.
- (2) In addition, sterilization process gives the milk
- a. Increased viscosity
- b. Improves the body of the finished product
- c. Gives the evaporated milk a more creamy consistency
- d. Tends it a pleasing richness

- e. Assists in keeping fat in homogeneous emulsion and
- f. Lessening its tendency to separation after manufacture.

## b) Sterilizers:

There are fundamentally two types of sterilizers in use:

## 10.5.1. BATCH STERILIZER:

The batch sterilizer consists of large boiler like horizontal steam drum opening at the top or at one or both ends. Its hollow interior consists of revolving frame of spider cage into which the cans are loaded. A perforated steam distributing pipe near the bottom extends over the entire length of sterilizer drum. This pipe has a separate steam inlet to each end. Near the top, there is a water distributing pipe with connections in the water main. In the bottom of the shell, there is a drain. On the sterilizer drum are mounted also a pressure safety vent, water steam and vacuum gauges, high temperature thermometer and preferably temperature control recording and safety devices. Process consists of filling the frame with water and heating it by steam and cans are kept rotating in it; temperature maintenance and time of heat treatment are controlled.

# **Temperature of Heating & Importance of Uniform Heating:**

A uniform temperature keeping is necessary in all the parts of sterilizer to keep away defects due to not uniform treatment to all cans and all parts of each can. It will keep uniform colour, consistency etc. Rapidity and uniformity of heat transfer are enhanced by the use of water in sterilizer. Sterilizing process must provide a ratio of temperature - time exposure that is lethal to even most resistant type of spore forming bacteria.

It is recommended that the temperature should be raised from room temperature to 115.5°C and held at that temperature for not less than 15 min and not more than 20 min. It is further recommended that the rise in temperature during last 10 min of the coming up period be at about 3°C for every minute and the temperature of holding should be between 115.5 – 118.3°C and time not less than 15 minutes.

## **Shaking:**

The purpose of shaking the evaporated milk is to mechanically breakdown the curd that may have formed during sterilization to a smooth homogeneous consistency. Violent shaking causes a sharp decrease in viscosity. Excessive shaking is therefore avoided. The period of shaking also should be minimized. Usual shaking period kept is 15 sec to 2 minutes and is sufficient to break normal soft curd.

# **Cooling After Sterilization:**

Immediately after the holding time, the evaporated milk is cooled. The steam is turned off, exhaust and drain are opened and cold water is turned into the sterilizer with the real revolving and cooling is continued until the temperature of milk is reached to 21 to 27°C. It should not take more than 15 minutes time. Rapid and uniform cooling is important.

#### **10.9.2. CONTINUOUS STERILIZER:**

It is best adopted in large plants. Its advantage lies in a large reduction of packing labour, elimination of incubation and of hand inspection and automatic rejection of leakers. On the other hand the continuous sterilizer is built for a standard size cans only. It will not handle odd size and larger size cans.

The evaporated milk continuous sterilizer consists of three principal units.

- (1) Pre heater
- (2) The Sterilizer or Cooker
- (3) Cooler

Principle behind the process is to avoid excessive adversities of pressure in sterilizer to Cans. Milk is preheated just to boiling point and then in sterilizer, temperature is raised and then milk is cooled.

#### 10.9.3. UHT Sterilization

**UHT sterilization** kills bacterial spores more effectively than in-bottle sterilization. The combination of preheating and UHT treatment of the concentrate suffices to inactivate plasmin. Preheating is also required to prevent excessive heat coagulation in and fouling of the UHT sterilizer. Some heat coagulation nearly always occurs, and the subsequent homogenization also serves to reduce the size of the protein aggregates formed. Aseptic homogenization must be applied. Indirect UHT sterilization in a tubular heat exchanger allows the pump of the homogenizer to be fitted before the heater and the homogenizing valve behind it. Thereby, the risk of recontamination with bacteria is diminished. The addition of stabilizing salt can often be omitted if UHT sterilization is applied, or the amount to be added is not so critical that sterilization tests must be carried out. It implies that the whole process from preheating up to and including aseptic packaging can proceed without interruption.

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#### Lesson-11

#### **Recombined Concentrated Milks-Raw materials**

#### 11.1. INTRODUCTION

Recombination process is applied for production of most dairy products and it has helped in creating new foods and formulations. Recombination is the process of recombining milk fat and milk solids-not fat (SNF) in one or more of their various forms with or without water. This recombination must be made so as to re-establish the product's specified fat-to-SNF ratio and solids-to-water ratio. The recombined process was first used widely during World War II and then onward spreaded worldwide. The main advantages derived are:

- (1) It gives better opportunities for transfer of raw material from a surplus area via industrial manufacturing to fulfill demand in deficiency areas for local range of milk and milk products.
- (2) It supplements and supports local milk and other products such as vegetable oils and sugars.
- (3) It generates possible employment for local population at dairy as well as with related activities, and
- (4) It creates goodwill and interest with authorities.

The mixing of milk solids and other ingredients such as sugar with water directly in the proportions normally required in the final product eliminates the costly evaporation step. However, the basic processes for manufacturing remain largely unchanged.

## 11.2. Raw materials

Raw material selection is the main factor determining quality of the final product. Therefore, strictly quality criteria with respect to their usual physical, chemical and microbiological specifications along with the recommended standards for the skim milk powders, whole milk powder, butter milk powder and anhydrous milk fat for use in recombined milk and milk products are required to be maintained. In addition, they should have complete freedom from extraneous matter, scorched particles; have bland flavour and highest possible bacteriological qualities.

## 11.3. Skim Milk Powder (SMP)

The skim milk powder used has to comply with strict requirements. The suitability of SMP can be determined by pilot scale batch production of the product. The powder must have been made from skim milk that is heated so intensely (e.g., for 1 min at  $130^{-0}$  C) that the recombined concentrated milk after its homogenization is sufficiently heat stable. The count of *B* . *stearothermophilus* spores should be so low that a moderate sterilization of the evaporated milk suffices. Skim milk powders of normal composition, in association with specific functional properties, generally ensure satisfactory performance and keeping quality. Depending on storage conditions, changes may occur in properties which influence

manufacturing of recombined products even with well prepared powders. Hence, it is recommended that storage temperature should not exceed 28<sup>o</sup>C. Other requirements are:

- (1) Skim milk powder used should be medium and low heat type depending upon viscosity desired in end-product.
- (2) Good reconstitutability is another required quality parameter.
- (3) The factors like region and time of production of SMP are usually more important for recombined SCM.
- (4) Other powder properties like fines content, bulk density, solubility index affecting dissolving properties are also important.
- (5) Control of viscosity and poor solubility of powder is possible with less flexibility by blending different lots of powders. Higher viscosity can be obtained by using SMP which is made employing preheating temperature 85-125  $^{0}$ C for >4 min. Also increased true protein content with preheating intensities results in higher apparent viscosities.
- (6) Age thickening is a defect entirely determined by SMP used as raw material in sweetened condensed milk preparation. Hence a test giving idea about the behaviour of powder towards this defect which determines the Age Thickening Ratio (ATR) is suggested. Also the pyruvate content of SMP which indicates level of enzymes affecting shelf life of product should be checked.
- (7) Viscosity of recombined concentrate used for preparing recombined WMP before spray drying is an important quality factor, which is largely due to properties of proteins and proportion of SMP added. Optimum choice is a low-heat or medium to low-heat SMP of uniform total protein content (~ 37 %).

# 11.4. Full cream milk powder (WMP)

Use of full cream milk powder for reconstitution, possibly avoids need for further homogenization and offers other advantages including ease of handling, transport and storage of a single raw material.

Recent developments in spray drying techniques like agglomeration and instantization have enabled the industry to use WMP for reconstitution purposes, which was facing difficulty due to dispersion problem at high concentration, loss of solubility, foam formation and oxidized and stale flavour development during storage. Improved packaging materials and methods of gas packaging have demonstrated best stability during storage.

Full cream milk powders preheated to same level as non fat milk powders give higher viscosity in SCM and hence reduction in preheating conditions is required during preparation. Full cream milk powders having poor physical properties pose problems in dispersion and give sedimentation in SCM.

# 11.5. Buttermilk Powder (BMP)

In recombined products, only a part of SMP (10-15 %) can be replaced by BMP. It gives fuller, creamier taste due to its emulsifying properties. However >15% BMP added for recombined sweetened condensed milk may have a detrimental effect on flavour. In Evaporated milk, it improves heat stability. Looking to its susceptibility to oxidation, BMP should be stored at low temperature and be used within one year from production. Incorporation of butter milk powder improves flavour and stability after preheating of concentrate before spray drying of recombined whole milk powder.

Water activity of powder is the key factor in storage stability and hence moisture content >5% and storage temperature >30  $^{0}$ C should be avoided. For meeting the specifications and dietary needs of consumers, vitamin fortification with Vit. A in the powders is recommended. A safety margin up to 50% is kept in the addition.

## 11.6. Milk Fat Sources

A range of fat sources is available for recombination, but selection is decided by flavour quality required, ease of handling and cost. 'Filled evaporated milk' is also made. A fat different from milk fat is used.

# 11.6.1. Anhydrous Milk Fat (AMF)

It is a traditional source of fat because of its stability under ambient storage conditions. Keeping quality can be enhanced by protecting it from air  $(O_2)$  and packing under nitrogen. Greater peroxide formation in neutralized oils has been shown and hence a standard is to be fixed for maximum sodium concentration. Temperature < 40  $^{\circ}$ C gives satisfactory storage period. Though restricted legally, antioxidants addition is suggested by some workers. However, their use is unnecessary if AMF is produced and packed correctly with low levels of dissolved head space oxygen. The copper and peroxide contents of the anhydrous milk fat should be low to avoid flavor deterioration.

It is found that the preparations containing ascorbyl palmitate, tocopherol and lecithin has a synergistic effect in controlling peroxide development. The AMF presents no problem provided it meets required specifications. Looking to the importance of AMF in influencing flavour of final product, low temperature (35-40  $^{0}$ C) storage and immediate use of liquid AMF after opening the package is preferable.

## 11.6.2. Soft/Hard fraction

Soft fraction is prepared by fractional crystallization from melted fat. It contains higher proportion of low melting components and hence remains liquid at high temperature and retains dairy identity of recombined milk products but it is less stable. Use of high melting fraction of AMF for better quality recombined WMP is recommended.

#### 11.6.3. Unsalted butter

It has a superior flavour and excellent shelf-life (2 yr.) at  $< -10^{-0}$ C. However high cost in transport, difficulty in storage, longer thawing period required, possible growth of

mesophillic and thermophillic organisms causing protein precipitation (burn-on problem) in melted product and heat stable lipases and proteases limits its use.

# 11.6.4. Fresh frozen milk fat for recombining (FFMR)

Made solely from fresh cream by modified process to maximize buttery flavour, it combines best features of butter and AMF and is convenient in processing.

# 11.6.5. Emulsifiers

Carrageenan, lecithin, glycerol-monostearate and alginates in varying concentrations are used to stabilize fat emulsions.

# 11.6.6. Water

Good quality water, either natural or treated, is essential. High calcium and magnesium levels in water adversely affect protein stability of reconstituted skim milk. Sequestration of hardness of very hard waters is suggested as a method of avoiding protein coagulation.

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## Lesson-12

# **Recombined Concentrated Milks-Processing**

## 12.1. INTRODUCTION

Processes for manufacturing recombined concentrated and dried milks vary widely and basic principles are well described. Usually the powder is dispersed and fat is mixed to give a level of total solids slightly above that required in final product. Some plants prepare recombined milk at solids level of normal milk and follow the evaporation process. The main stages are:

# **12.2.** Mixing

- (1) Powder or powders for Evaporated Milk preparation are mixed to get 18-20% SNF concentration in water, heated to 45-55°C and dispersion is effected using a high speed blade, a powder funnel and centrifugal pump, a powder/liquid blender or number of other devices designed for the purpose. The non-fat milk powder used shall confirm the standards as specified. But especially heat stability of SMP used is of prime importance and hence WPNI of < 1.5 is recommended for recombined evaporated milk preparation. Care should be taken to minimize air incorporation or a deaerator can be used.
- (2) Liquid fat (55-60%) may be added before, after or with powder to minimize foaming. Uniform dispersion of fat to maintain protein/fat ratio at homogenization is required otherwise fat separation results.
- (3) The ingredients for SCM preparation are weighed. The SMP is dissolved in water heated to 40-50  $^{0}$ C first to give solids concentration of 44%. For this a high energy, high shear mixer, most commonly the "Cowles' Dissolver" type is required. Introduction of good quality powder to mixing vat through a pneumatic conveying blow line ensures sufficient dispersion. Also inline solid-liquid blenders of Tri-blender type are used for mix preparation.
- (4) After hydration time of about 15 min during which the dispersed powder completes its dissolution, the sugar can be added and dissolved (agitation stopped to minimize air incorporation and to save power). During sugar addition, mix is heated to maintain temperature at 40-50 °C. Then previously melted fat (50 °C) is added while mixing and the mix is further heated to 50-60 °C to facilitate filtration.
- (5) When full cream powder is used, highest possible level of TS (52%) is used at mixing stage to achieve maximum economy.

#### 12.3. Filtration

Filtration is carried out on duplex-type filters using a range of filter materials to remove extraneous material from milk prior to homogenization.

## 12.4. Preheating

Preheating to homogenization temperature, 50-60 <sup>0</sup>C is done. Higher temperature adversely affects heat stability of recombined evaporated milk.

## 12.5. Homogenization

From several systems, high pressure unit using shear type valve is commonly used for recombined EM preparation. Usual pressures are in range of  $140 - 175 \text{ kg/cm}^2$  for first stage, and  $35 \text{ kg/cm}^2$  on second stage. Increasing the pressure in first stage may reduce heat stability of protein. Prior to homogenization, recombined SCM is deaerated but it may not be followed if vacuum cooling is applied at the end.

Homogenization of recombined SCM is usually carried out at a temperature range of 50-60  $^{0}$ C in single stage (70 kg/cm<sup>2</sup> / 7 MPa) prior to pasteurization. However better control of product viscosity is possible if two stage homogenization (pressure up to 105 kg/cm<sup>2</sup> /10 MPa) is done after pasteurization. Use of full cream milk powder requires low pressure (2.8-0.7 MPa) two stage homogenization to control viscosity. Unhomogenized product gives high initial viscosity and accelerated rate of age thickening.

# 12.6. Pasteurization and cooling

Recombined EM may be heated to homogenization temperature or pasteurized to extend its storage life and cooled to < 5 °C. Pasteurization conditions used for recombined SCM vary in range of 80-90 °C for 30 sec to 2 min. Use of 91 °C for 30 sec gives adequate safety to product from yeasts without adverse effect on color or flavour. However, holding the product at higher temperature for longer period gives discoluration due to Maillard reaction.

The recombined SCM must be handled aseptically here on. Cooling in plate heat exchanger up to 50 °C and then by vacuum cooling to 30 °C is carried out.

# 12.7. Lactose seeding

In recombined SCM, formation of greatest number of small crystals (<10  $\mu$ ) is required to control large crystal formation giving sandy mouth feel. Addition of very finely ground seed lactose powder (300 mesh,  $\sim$  1-10  $\mu$  size) with high microbiological standards is done at a rate of about 0.05 % either as powder or as slurry. Alternatively, skim milk powder or sweetened condensed milk of previous batch can be used as seed material. After crystallization, further cooling under vacuum leads to about 10 % moisture evaporation and hence suitable allowances are made in original mix.

# 12.8. Storage, Stabilization and Standardization

The viscosity of EM is determined and solids level is adjusted to required specification. Addition of phosphates, citrates and sometimes calcium is required to stabilize proteins against heat effects during sterilization but their use should be kept to a minimum to avoid adverse effect on flavour of the product. The level of stabilizer is determined by taking trials and then whole batch can be standardized for canning.

## 12.9. Canning and sterilization

The essentials in canning recombined EM are clean, well made cans, minimum foam, appropriate head space, correct weight and efficient closure. Any type of sterilization method employed should be efficiently performed and rapid cooling should follow to avoid any defect.

Careful packaging to avoid any contamination from external sources is required for recombined SCM along with minimum headspace while filling to prevent growth of any surviving moulds.

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# Module 5

# Physico-chemical changes taking place during manufacture of Condensed milk

# Lesson-13

## 13.1. INTRODUCTION

There are numerous changes occurring in the condensed milks during their manufacture. They are because of the inherent properties of the milks, any additives like sugars, stabilizers etc. added and the processing variables. Some of the product specific changes of commercial importance are discussed here.

## 13.2. CHANGES CAUSED BY CONCENTRATION

Apart from the increase of most of the solute concentrations, removal of water from milk causes numerous changes in properties, which often are approximately proportional to concentration factor. The changes also depend on other conditions, such as heat treatment and homogenization. Some important changes in properties are:

- 1. The water activity decreases.
- 2. The Calcium ions activity increases only slightly because calcium phosphate, which is saturated in milk, turns into an undissolved state. As the water content decreases, association of ionic species increases and also ionic groups of proteins are neutralized.
- 3. The conformation of proteins changes because ionic strength, pH, and other salt equilibria change. When milk is highly concentrated, the solvent quality decreases. Thus, the tendency of the protein molecules to associate and to attain a compact conformation is increased. Coalescence of casein micelles causes them to increase in size. This increase is smaller if the milk has been intensely preheated, presumably because β-lactoglobulin and other serum proteins have become associated with casein.
- 4. Osmotic pressure, freezing point depression, boiling point elevation, electrical conductivity, density and refractive index increase and heat conductivity decreases.
- 5. The viscosity increases and the liquid become non-Newtonian and finally solid-like.
- 6. The diffusion coefficient of water decreases from approximately  $10^{-9}$  m<sup>2</sup>.s<sup>-1</sup> in milk to  $10^{-16}$  m<sup>2</sup>.s<sup>-1</sup> in skim milk powder with a small percentage of water.

## 13.3. EVAPORATED MILK

- **13.3.1.** Viscosity: Viscosity is defined as resistance to the motion of the molecules of a fluid body among themselves caused by internal frictions as opposed to mobility. This is measured by viscosimeter.
- **13.3.2. Effect of Sterilization on Viscosity of Product:** Viscosity of fresh milk changes by the preheating of milk. The condensing operation causes a slight but definite increase in viscosity. This is due to increase in concentration of milk solids. The increase in viscosity that gives the finished product its full body, however occur during process of sterilization. In

general HTST yields a thin body and low viscosity, while LTLT process yield a full body and little more viscosity.

13.3.3 . Increase in viscosity by Progressive Coagulation of Milk Proteins: In sterilization process, the rate of thickening is greatest, shortly before the occurrence of a visible coagulation. It is observed that the thickening does not proceed rapidly until above 10 min before coagulation. This is because the thickening or increase in viscosity during sterilization appears to be a part of the function of coagulation. Thickening in sterilization is in fact the beginning of coagulation. It is very slow and gradual and in normal commercial process of sterilization, covering a period of approximately 20 minutes at the full sterilizing temperature. The proportion of milk proteins coagulated while sufficient to increase viscosity, is too limited for curd to become visible. The increase in viscosity which milk undergoes toward the end of sterilizing process has to do with progressive coagulation of the milk proteins. Table-13.1. below shows the viscosity changes during successive steps in manufacture of evaporated milk:

Table-13.1: Viscosity changes during successive steps in manufacture of evaporated milk

Stage of Manufacture	Viscosity Retardation
Fresh Milk before fore-warming	15.24
Fresh milk after fore-warming	15.26
Evaporated milk before sterilizing	20.20
Evaporated milk just after sterilizing.	150.00

It is observed further that for a heavy creamy body, the heat stability of milk should not exceed 30-40 minutes. Milks with heat stability in excess of 50 min will be exceedingly thin at the end of sterilization at  $115^{0}$ C for 30 min unless an increase in solids content is depended upon to build up body. The viscosity produced during sterilization is controlled mainly by the heat stability of milk. Therefore, factors which affect the heat stability of milk will certainly affect the viscosity of milk. Following factors gives low heat stability, high viscosity and very thick body and vice-versa:

- (1) High acidity of fresh milk
- (2) Low forewarming temperature
- (3) High concentration
- (4) High homogenizing pressure
- (5) Excessive holding of evaporated milk at ordinary temperature before sterilization

Consumer desires a product with a good body that suggests richness. The heat stability sufficient to avoid visible coagulation or curdling during sterilization process is required but maximum heat stability is not desirable because such high stability results into objectionably thin milk.

## 13.3.4. Effect of Storage on Viscosity:

Evaporated milk becomes thinner with age. This loss of viscosity increases with the temperature of storage. The decrease in viscosity begins immediately. The rate at which viscosity is lost is accelerated at high storage temperature Thus it is observed that at or above 30°C, evaporated milk looses as much as 40% of viscosity (original) during first 10 days of storage. While at 15.6°C or below, the age thinning is very slight. It is reduced after which the rate of thinning is much more gradual. Age thinning of evaporated milk may therefore be definitely retarded and the attainment of final viscosity delayed by use of relatively low storage temperature. A study shows viscosity loss upon storage for 110 days of 58.75% at 26°C, 40% at 15.6°C & 11.25% at 7.2°C. But this depends upon quality of raw material (milk) used. Better grade milk lost less viscosity than the low grade initial milk with the same temperature and time of storage.s

## 13.3.5. Gel Formation on Storage

In case of some evaporated milks, the viscosity increases later in the storage period even to the gel formation. This tendency is greatest with milks that had received relatively light heat treatment and those of high solids concentration. These results of gel formation in prolonged storage are supported by commercial experiences especially with samples with high TS evaporated milks incubated at tropical temperatures.

Further it is observed that evaporated milks that have been sterilized by the HTST process (135°C for 0.5 min) thicken quickly during storage. When the gel is broken before it becomes firm, the product wheyed off. When sterilized in the conventional manner (115.6°C for 20 min), gel formation is slow and holds the water better than from the HTST process. Since long fluidity in storage is a marketable property of primary magnitude, it appears that the more severe heat treatment of commercial process (115.6°C for 20 min) that retards thickening in storage is commercially preferable to the less drastic treatment of momentary exposure to high sterilizing heat.

Neither the mechanism underlying age thinning nor that responsible for age thickening appears to be well understood. It may be due to insufficient time for the attainment of new solubility equilibrium of the Calcium and Magnesium salts when using HTST method of sterilization. When solutions of these salts are heated, it takes considerable time to attain that. Their peculiarity of being less soluble at higher temperature may be contributing to the reaction of these salts.

In the HTST method of sterilization, conditions are thus less favorable than those of the LTLT procedure (115.6°C for 20 min). This suggests the possibility that, less of Ca and Mg content is rendered insoluble. This condition might encourage the protein particles to swell and to form a gel or it may be as low continuation of the coagulation process accompanied by an operation of caseinate molecule which finally produces an irreversible gel structure.

# 13.3.6. Protein Separation during Storage

The proteins of some evaporated milks have a tendency to settle during storage. This is found more common in evaporated skim milk than evaporated whole milk. Homogenization retards protein separation in evaporated whole milk. It is observed that, fat in evaporated milk is a factor to maintain a normal dispersion of milk constituents during long period of storage. It is observed in this respect that fat acts to counter balance the protein settling to bottom.

# 13.3.7. Fat Separation in Storage

Our aim is to get Evaporated Milk of good body and to avoid fat separation. During age thinning and before age thickening sets in, there is definite tendency to objectionable fat separation. This tendency emphasizes importance of efficient homogenization that ensure reduction of at least 90% of fat globules to 2  $\mu$  or smaller. Such efficiency can be obtained by taking following care:

- § Daily check up of homogenization Valve
- § Keeping the clearance surface of valve and valve seat smooth and intact by regrinding or resurfacing.
- § Making sure of the continued accuracy of the pressure gauge.
- § Examining fat globule size.

It is observed that HTST sterilization provides little opportunity to control body hence through a heavier body, control of the extent of fat separation can be achieved. Again here the LTLT procedure (115.6°C for 20 min) proves helpful. Fat separation in this more viscous body is slower and less intense. In case of LTLT, fat layer get re-emulsified easily than in case of HTST sterilization process.

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## Lesson-14

# Physico-Chemical Changes Taking Place during Manufacture of Condensed Milk-II

#### 14.1. INTRODUCTION

During the processing of evaporated and sweetened condensed milks the physico-chemical changes that take place are further elaborated here.

#### **14.2. COLOUR**

#### 14.2.1. Relation of Caramelization to Discolouration

Browning of milk is attributed to reaction of sugar in milk. But when solution of sucrose, lactose, dextrose are heated separately, it do not show any sign of browning or caramel flavour. The colour changes in evaporated milk are not in direct relation of time, but it is the reaction of catalytic nature. Similarly, solution of sugar and proteins when heated, a marked brown colour develops and colour could not be removed by washing and also whey is clear showing no sign of any colour.

# 14.2.2. Relation of Amino Acid Sugar Complex to Browning

It appears that in the lactose - protein reaction, responsible for the darkening of milk proteins, certain amino acids are involved. The sugar combines with NH<sub>2</sub> group which is alkaline, neutralizing the alkalinity and leaving the acid group free, thereby causing a reduction of pH accompanied by the formation of highly coloured product. It is well known that sugar heated with an alkali turns brown; hence the amino acid - sugar complex which is alkaline would tend to assume a brown colour. This would be the case especially with a reducing sugar such as lactose, because of the availability of the aldehyde groups, which are naturally present in reducing sugar. This is further proved as if we add formaldehyde, (being having a great affinity for amino acid) to evaporated milk at the time of sterilization, milk remains quite white.

# 14.2.3. Effect of steps of Processing on Colour

It is found that time and temperature of heating is an important factor contributing towards colour of the milk. In addition it is noted that

- 1. Within the time of forewarming applied generally, the product suffers no decisive change in colour. If it is for long period (30'), effect is intense.
- 2. Homogenization tends to diminish the colour of the product because of a finer sub division of the fat globules which prevent such penetration of the rays of light as it would reveal the butter fat more nearly in its natural colour which is yellow.

3. Storage causes progressive darkening with increase in time and temperature. At (5<sup>0</sup>C) there is no change in colour.

It is observed that for each  $10^{\circ}$  C decrease in forewarming temperature, the holding period is increased ~ 2.5 fold without causing an increase in colour.

## 14.3. Cooked flavor

Though cooked flavour is of little significance in our country, but still its mechanism is to be studied. Heat treatment in processing tends to produce a cooked flavour in evaporated milk. This is true also of fresh milk and milk products in general. It is generally observed that cooked flavour accompanies the darkening of colour. HTST process produces less cooked flavour than LTLT process. HTST sterilization treatment (135°C for 0.5 min) also reduces the extent of sterilization, decreases the viscosity and also fat separation etc. occur. So because of these limitation factors, LTLT process is important.

## 14.3.1. Reaction Involved in Production of Cooked Flavour

The reactions involved in production of cooked flavour are not sufficiently clear. It is observed that when heating skim milk, cream, milk, EM, etc., -SH compounds are produced (provided heated to high temperature or at low temperature for longer time) from one or more proteins present. However, it is found that -SH compounds are wholly responsible for cooked flavour. Five hours heating at  $70^{\circ}$ C produces full cooked flavour. It is shown that in the reaction causing cooked flavour of milk by heat,  $O_2$  is taken up and  $CO_2$  is produced. Oxidation condition inhibits the formation of -SH group.

# 14.4. SEDIMENTS OF MINERAL SALTS

There is a tendency in some evaporated milk upon ageing for granular deposit to form in the can. This deposit has a whitish colour, it is gritty and insoluble and seemingly of noncrystalline character. On analysis these granular structure are found to contain Tri Calcium Citrate -  $Ca_{36}H_5O_7)_2$  and Tri Calcium Phoshate -  $Ca_3$  (PO<sub>4</sub>)<sub>2</sub>. These salts have peculiarity of being less soluble in hot rather than in cold solution. (C

# 14.4.1. Effect of Processing on Mineral Salts

The tendency for increased concentration of milk solids to cause an increase in the amount of sediment produced is well known to evaporated milk manufacturers. An increase in concentration necessarily increases Ca, Mg, citric acid and PO<sub>4</sub> content in the milk and thus provides possibility of precipitation. This can be controlled by addition of casein stabilizer.

# 14.4.2. Effect of Temperature of Storage on Sediment Formation

The precipitation of mineral salts in the form of white sand like deposits is an age defect of Evaporated Milk. The temperature of storage appears to be a controlling factor in this respect. Table 14.1 below shows this effect.

Table-14.1.: Effect of Temperature of Storage on Sediment Formation in Evaporated Milk

Stora	age	Relative amount of Ca-citrate sediments
Temperature	Time	Relative amount of Ca-citrate sediments
29.4 <sup>0</sup> C	30 days	Large amount of fine as well as large particles
20.00 C	78-110 days	Considerable amount of large particles
7.2 <sup>0</sup> C	4 months	No sediments

### 14.1. EFFECT OF HTST STERILIZATION

By this process, natural property of milk is retained better than that with the process of low temperature long duration. The major advantages are to avoid cooked flavour and colour changes. By HTST sterilization, heat stability will be better in concentrated product than with conventional method of sterilization, but the problems that are encountered in this process are insufficient viscosity and thin body which ultimately increase the tendency of fat separation on storage. Self stability of product is relatively small and gelation may occur during storage period. Some of the different types of processes developed are:

- (1) **Tin Sterilization**: Sterilization at 127°C-130°C for about 2 minutes to 40 seconds.
- (2) **Continuous Flow Sterilization**: Here temperature is 145<sup>o</sup>C or higher with only few seconds holding and with subsequent aseptic canning. For avoiding the problems due to this sterilization process, increase the viscosity and reduce the age thickening.

Several methods are supplementing temperature treatment which may be given before or after sterilization. They may be given to the product during coming up period or cooking period and get the advantage of avoiding cooked flavour.

# 14.2. Effect of Heat Treatment on the Acidity

When heat treatment is given to milk in manufacture of concentrated milk, the acidity measured in terms of H<sup>+</sup> concentration and titratable acidity is increased. The rate of formation of acid in concentrated product is in proportion of time and temperature treatment.

It is also related to concentration of lactose. If the milk is heated in open pan, then the acidity will slightly drop in the beginning followed by an increase in acidity which will continue till the coagulation of casein. Initial decrease in acidity is due to loss of CO<sub>2</sub> dissolved in milk which usually contributes to 0.01-0.02% of the total acidity expressed as lactic acid. The increase in acidity on further treatment is due to breaking down of casein causing cleavage of phosphorus containing acid, probably nucleic acid and to a lesser extent to oxidation of lactose. The actual increase in acidity due to conventional temperature time ratio of forewarning in SCM is very slight. This slight increase in acidity is compensated by the initial loss of acidity due to expulsion of CO<sub>2</sub>. Whereas in case of evaporated milk, the reaction of sterilization heat treatment in conventional type procedure is more and in addition,

the milk is also heated at higher temperature of forewarming. Due to this treatment, usually there is increase in acidity approximately 0.05 - 0.1% during the sterilization process than the normal acidity due to concentration.

# 14.7. Effect of Storage, Time & Temperature on Acidity

The increase in acidity is in direct relation with time and temperature of storage. The acid producing reaction due to heat treatment is continued even when the concentrated product is held in storage room. The rate of acid formation will be very slow depending upon the temperature of storage. SCM of 64.5% sugar ratio having initial acidity of 0.43% and the original bacterial count 4381/g when stored at different temperatures resulted in to increase in acidity as shown in Table 14.2:

Temperature	Time	Acidity
10.0°C	640 days	0.84%
15.6 <sup>0</sup> C	640 days	0.85%
22.2°C	640 days	1.0%

Table-14.2: Effect of Storage, Time & Temperature on Acidity of Evaporated Milk

This increase in acidity is mainly due to chemical changes as there was no increase in microorganisms during storage.

### 14.8. SWEETENED CONDENSED MILK

#### 14.8.1. Age Thickening

The main change in sweetened condensed milk during storage is presumably age thickening and, finally, gelation. Sweetened condensed milk is far more concentrated than evaporated milk. However, it does not thicken markedly faster with age. It is usually assumed that added sucrose inhibits age thickening. Sucrose increases the Ca<sup>2+</sup> + activity. A difference with evaporated milk is that an initial decrease in viscosity before age thickening is not observed. The viscosity in sweetened condensed milk increases almost linearly with time. The following main factors affect age thickening in sweetened condensed milk:

- 1. The variation in the type of milk and season occurs among batches of milk.
- 2. Higher preheating treatment yields higher initial viscosity, and gel can form earlier. Hence, UHT heating is now generally applied.
- 3. The addition of sugar at later stage in the evaporating process results in lesser age thickening.
- 4. Higher concentration factor gives more age thickening.
- 5. The influence of added salts varies widely and depends on the stage at which it is added. Salts are added up to 0.2%. Adding a small amount of sodium tetrapolyphosphate (e.g. 0.03%) mostly delays age thickening considerably, whereas adding more may have the opposite effect.
- 6. Age thickening considerably increases with storage temperature.

# 14.8.2. Maillard Browning

Ongoing Maillard reactions are inevitable. Brown discoloration is stronger as the storage temperature is higher, the milk is evaporated to a higher concentration, and more intense heating is applied. Additional Maillard reactions occur if the added sucrose contains invert sugar.

# 14.8.3. Oxidative Changes

These changes can be prevented by keeping head space oxygen to a minimum.

### 14.8.4. Lactose Crystallization

Sweetened condensed milk contains around 38 to 45 g lactose per 100 g water. The solubility of lactose at room temperature is about 20 g per 100 g water, but in sweetened condensed milk the solubility is about half as much due to the presence of sucrose. It implies that 75% of the lactose tends to crystallize, meaning about 8 g per 100 g sweetened condensed milk. Due to the high viscosity, nucleation will be slow and only a few nuclei would be formed per unit volume of milk, leading to large crystals.

Without special measures, the product will obtain a relatively high quantity of large crystals. These crystals settle and are responsible for a sandy mouth feel. Although the crystals may not be so large as to be felt singly in the mouth, they can be large enough to cause a non-smooth impression. To avoid this, they should be smaller than about  $8 \mu$  m in length.

Preventing crystallization is not possible and, accordingly, a large number of crystals should be obtained. Satisfactory results can be reached by using seed lactose. Adding 0.03% seed lactose represents 0.004 times the amount of lactose to be crystallized. The final size of the crystals in the product should not exceed 8  $\mu$  m. Consequently, the seed lactose would contain enough seed crystals (one per crystal to be formed) if its crystal size does not exceed about  $(0.004 \times 8^3)^{\frac{1}{3}}$ , i.e., 1.25  $\mu$  m. Such tiny crystals can be made by intensive grinding of  $\alpha$  -lactose hydrate.

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### Module 6

# Heat stability of Milk and Condensed milk

### Lesson-15

# **Heat Stability Of Milk**

# 15.1. INTRODUCTION

The heat-induced coagulation of milk is one of the few major classical problems in dairy chemistry awaiting complete solution. Heat coagulation is the result of a complex series of simultaneous reactions, some of which are complementary whereas others are antagonistic. Further most research has been directed towards the heat stability of unconcentrated milk, though problems related to that of concentrated milks may be of grater commercial significance.

When milk is held at temperatures above the boiling point it eventually coagulates, the higher the temperature the sooner. Coagulation time is strongly dependent on pH. The pH at coagulation is always low (< 6.2). The heat coagulation time (HCT) at 140° C of bulk milk at its pH of maximum stability is usually 20 to 30 min.

Casein-the major milk protein is not denaturable within the strict definition of the word because of its lack of secondary and tertiary structure. In fact, casein generally is considered to be a naturally denatured protein. However, it does undergo changes mostly hydrolytic, when subjected to severe heat treatment. The casein micelles aggregate and undergo heat coagulation unlike heat denaturation suffered by globular proteins (whey proteins). During relatively long period of heating at an elevated temperature many heat-induced changes occur. It is these changes that ultimately lead to coagulation, few of them have been investigated thoroughly and some have not been studied at all. Some heat-induced changes in milk likely to lead to protein coagulation are:

- 1. Decrease in pH.
- 2. Precipitation of calcium phosphate
- 3. Denaturation of whey proteins and interaction with casein
- 4. Maillard browning.
- 5. Modification of casein:
- a. Dephosphorylation
- b. Hydrolysis of  $\kappa$ -casein
- c. General hydrolysis

- 6. Changes in micellar structure:
- a. Zeta potential
- b. Hydration changes
- c. Association Dissociation.

#### 15.2. TYPES OF MILK

- **15.2.1. Type A milk**: Those milks which show a very pronounced maximum (~ pH 6.7) and minimum (~ pH 6.9) in stability in HCT/ pH curve. At pH>6.9, HCT increase again.
- **15.2.2. Type B milk**: Those milks in which stability increase progressively with increasing pH and show no maximum and minimum

The precise shape of the HCT/pH curve is characteristic of individual milks and is generally similar throughout the lactation period. Presumably dietary, environmental and genetic factors are responsible for this variation (Figure 15.1).

Fig. 15.1: Curve showing effect of pH on the heat stability on Type A, Type B milks and Serum protein-free casein micelle dispersion

Perhaps pH is the most important single influencing factor; all bulk milks and most individual cow milks exhibit a maximum at  $\sim$  pH 6.7 and a minimum at  $\sim$  pH 6.9 in the HCT/pH curve. The precise shape of the HCT/pH curve is influenced by a host of factors among which the most important are:

- 1. Whey proteins,
- 2. Other heat-denaturable proteins,
- 3. κ-casein,
- 4. Colloidal calcium phosphate,
- 5. Detergents,
- 6. Assay conditions (temperature & agitation)
- 7. Urea.
- 8. Preheat treatment,
- 9. aldehydes,
- 10. concentration

#### 11. or dilution

Approximately 20% of individual cow milks show Type B characteristics in Canada, 1% in Australia and Ireland and 70% in Japan.

# 15.2.3 . HCT/pH profile of buffalo milk (Temp 140° C, pH at 5° C)

Like Type A bovine milk, buffalo milk show maximum at pH  $\sim$  6.6, followed by a sharp decrease in stability at pH  $\sim$  6.8. Poor heat stability persist up to pH 7.3 to 7.7 resulting in broad minimum. A further increase in pH leads to an increase in heat stability. Based on these data, buffalo milk is categorized as Type A milk (Figure 15.2 and Table 15.1).

Fig. 15.2: HCT/pH curve of buffalo milk

Table-15.1: HCT at 140°C of individual milk sample at natural pH, pH of HCT – maximum and pH of HCT – minimum

рН	HCT (min)
$6.72 \pm 0.072$ (natural)	11.3 ± 5.4
$6.66 \pm 0.11 \text{ (HCT - maximum)}$	$16.4~\pm~6.6$
$6.99 \pm 0.10 \; (HCT-minimum)$	$3.0\pm0.9$
$(6.83 \pm 0.08 \text{ to } 7.35 \ \pm 0.17)^*$	

<sup>\*</sup> Refers to pH range of HCT - minimum.

#### 15.2.4. Reasons for overall shape of a Type A heat stability curve

- 1. On the acid side of the maximum, the coagulation of casein micelles is governed solely by the calcium-induced complexation of casein. As the pH is increased, the calcium ion concentration decreases and casein micelles become more heat stable; however, after a certain degree of protein polymerization and dephosphorylation (and hydrolysis of  $\kappa$ -casein) have occurred, which reduce casein stability, the lower calcium ion concentration is sufficient to induce coagulation. The occurrence of the heat stability minimum ( $\sim$  pH 6.9) is due to a combination of the formation of  $\beta$ -Lg/ $\kappa$ -casein complexes, which are sensitive to calcium phosphate precipitation, and the increase in heat induced calcium phosphate deposition.
- 2. Heat produced acid may be the principal cause of coagulation at the heat stability maximum and periodic neutralization postpones coagulation very significantly.
- 3. The transition from the coated (by denatured whey protein) to the protein depleted (depleted of  $\kappa$ -casein) form, which occurs as the pH is increased above that for maximum heat stability, results in the heat stability of the micelles first increasing (the maximum), then decreasing (the minimum) and finally increasing again over the pH range 6.4-7.0.
- 4. The occurrence of the minimum in the HCT/pH profile is due to the heat induced dissociation of micellar κ-casein, which occurs when milk is heated beyond 90°C at pH values above 6.9. The κ-casein depleted micelles are sensitive to calcium phosphate precipitation and hence the heat stability minimum occurs. The high heat

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stability at pH values above the heat stability minimum is probably due to a lack of available calcium ions (as a result of heat-induced precipitation of calcium phosphate) and the high net negative charge on the protein. Thus, to induce coagulation at pH values above  $\sim$ 7.2, a longer heating time is required to cause dissociation of sufficient  $\kappa$ -casein, hydrolysis of  $\kappa$ -casein, reduction of pH or other heat-induced changes to reduce the micellar negative charge to a value permitting coagulation.

## 15.3. Micro-structural changes revealed by electron micrographs

Heating skim milk at 130 or 140° C:

- (1) Reduction in the range of micelle size and increase in the average micellar diameter.
- (2) Below pH 6.7, heating cause changes in the micelle surfaces from smooth to a ragged appearance with many appendages.
- (a) This is due to  $\beta Lg / \kappa$ -casein complexes at the micellar surfaces
- (b) During heating more β-Lg precipitated on to the surfaces.
- (3) Above pH 7.0, β-Lg / κ-casein complexes are not formed on heating milk.
- (4) At the pH of maximum heat stability (pH 6.7), coagulum formed show that the casein micelles are aggregated in short chains in a relatively open network and many individual micelles persist.
- (5) At the pH of minimum heat stability (pH 6.9) milk coagulum show a closed network of long chains of fused micelles.

Casein micelles joins in chains and clusters gradually build into a gel network during the last few minutes before visible coagulation, but in the coagulum just after coagulation the gel network become denser and is composed of long chains of fused micelles, indicating an abrupt change at coagulation.

#### 15.3.1. METHODS OF MEASURING HEAT STABILITY

1. **Subjective heat stability test:** The test is used to determine the heat stability by heating a small sample of milk (1-1.5 ml), sealed in a narrow glass tube, in a thermo statically controlled oil bath, usually at 140° C for milk of standard concentration or at 120° C for concentrated milks, until particles of coagulated protein are observed in the following milk.

The heat coagulation times (HCT) depend on a number of experimental factors especially:

- (i) Degree of tube fill
- (ii) Head space gas
- (iii) Rocking rate during heating
- (iv) Angle of tilt and

(v) Assay temperature.

The Q 10° C for coagulation is usually ~3.

**15.3.2. Objective heat stability assay:** In this test the percentage of total sedimentable nitrogen by low gravitational forces is determined after various heating intervals. The resulting "nitrogen depletion curves" show a break at the onset of visual coagulation, samples that coagulate rapidly yielding large protein flocks, show a sharp break in the curve while those that coagulate slowly show a more gradual increase in sedimentable nitrogen.

The objective method is strongly recommended if the heat stability characteristics of a sample are to be described fully.

# 15.4. Factors affecting the heat stability of milk

# 15.4.1. Species and breed of animal

### **Species**:

- (a). Buffalo milk is less stable than bovine milk and shows a maximum in HCT/pH curve similar to Type A cow's milk.
- (b). Human milk is quite heat stable and shows Type B stability characteristics.
- (c). Ovine and Caprine milks show a marked maximum at pH 7.0 in the HCT/pH curves, but are very unstable at all higher pH values.
- (d). Sow milks are shown to have very low heat stability and show a progressive increase with increasing pH (no maximum or minimum)
- (e). Mare milk is very unstable at their original pH (7.0), HCT is <10 min at 100°C.

### **Breed:**

- (a). The values of heat stability of cow-milk from Sahiwal, Tharparkar, Red Sindhi and Crossbreds were reported to vary from 30.38 to 197 min at 120° C and 32.40 to 34.73 min at 136°C whereas the values of heat stability of milk from Murrah buffaloes at 120°C was from 99 to 109 min, 35.18 min at 136° C and 32.9 min at 130°C
- (b). Milk from Jersey cows had greater maximum and natural heat stability than milk from Friesion Cows. Maximum heat stability declined with the age of the cows.
- (c). It is known for long time that heat stability of buffalo milk is much lower than cow milk. But contradictory reports have appeared in the literature on heat stability of buffalo milk.

## 15.4.2. Genetic Variants

Maximum heat stability of  $\kappa$ -casein is observed in genetic variants (B>AB>A) and  $\beta$ -lactoglobulin (B, AB>A): whereas natural heat stability is affected only by  $\kappa$ -casein genetic variants (B>AB>A).

- a. Maximum and natural heat stability at 120°C is correlated positively with  $\beta$ -casein and  $\kappa$ -casein concentrations and is negatively correlated with  $\alpha S_1$  casein and  $\beta$ -Lg concentrations.
- b. Natural and maximum heat stability is correlated positively with urea concentration.
- c. The pH of skim milk samples is associated with  $\alpha S_1$ -casein, genetic variant, age of cow, stage of lactation and concentration of  $\gamma$ -casein.

# 15.4.3. Stage of lactation and dietary factors

### a. Lactation:

There is a general, erratic tendency for milk to become more stable with advancing lactation. Season/stage of lactation cause variability in heat stability due to differences in composition particularly urea and other factors involved in colloidal stability: Ca<sup>2+</sup> activity, casein micelle size, phosphate content of micelles, and their voluminosity.

#### b. Feed:

The type of feed influences heat stability characteristics. Cows produce Type A milk when grazed on good pasture but some cows produce Type B milk, or milk intermediate between A and B, when grazed on dry, withered pasture.

#### 15.4.4. Milk constituents

# (a) Milk Salts

- § H<sup>+</sup> and polyvalent cations and anions (ca<sup>2+</sup>, Mg<sup>2+</sup>, PO<sub>4</sub><sup>3-</sup> and Citrate<sup>3-</sup>), especially the concentration of these ions in the serum phase, play an important role in the heat stability.
- § Milk salts contribute upto 65% of total buffering capacity of milk. The distribution of salts in milk results from complex temperature dependent equilibrium between dissolved and colloidal phases.
- § The soluble salts are the principal factors influencing heat stability but they apparently act only in the presence of  $\beta$ -Lg. However, both soluble calcium and phosphate (SP) have a major effect on the shape of the HCT/pH curve.
- § Although the HCT/pH curve can be readily altered by modifying the concentration of soluble and colloidal salts, natural variation in HCT are not correlated with the concentrations of indigenous salts.
- § Ca, P and citrate ions show negative correlation with HCT of buffalo milk and its concentrate, while Mg is found to have positive correlation with their HCT.
- $\S$  All the four polyvalent ions Ca, Mg, P and Citric acid exhibit a negative correlation with HCT of cow milk at 140 ° C.

## (b) Caseins:

#### κ-Casein

- (1)  $\beta$ -Lg /  $\kappa$  case in interaction determines the shape of HCT/pH curve.
- (2) The ratio of  $\kappa$  -casein (on the surface of casein micelles) to b -Lg determines whether a milk is of Type-A or Type-B
- (3) Since buffalo casein micelles are larger than bovine micelles, it is probable that the former will have lower quantity of surface  $\kappa-$  casein than the latter and because buffalo milk contains 23% more b -Lg than bovine milk, a very low  $\kappa$ -casein / b -Lg ratio, may be partly responsible for the broad minimum in buffalo milk.
- (4) Buffalo casein micelles have lower net negative charge than bovine casein micelles and this may also contribute to the broad minimum observed in buffalo milk.
- (5) Increase in casein micelle diameter (from  $\sim$ 25 nm to above 200 nm) on heating skim bovine milk at 140 ° C for 10 min may be due to.
- a. Precipitation of denatured whey protein on to the surface of the casein micelles via formation of b -Lg/ $\kappa$  casein complexes
- b. Heat induced precipitation of calcium phosphate on to casein micelles, thus attaching small and shattered micelles to intact micelles.
- c. Heat induced aggregation of small micelles leading to the formation of larger micelles.
- d. Since  $\kappa$ -casein is believed to be located mostly at the micellar surfaces, it is present at a higher concentration in small casein micelles than in larger casein micelles. Therefore, there are more  $\kappa$ -casein molecules available on the surfaces of small micelles for interaction with denatured  $\beta$ -Lg than on larger micelles.

#### c) a s2 -Casein

Addition of a s2-casein to milk has no significant effect on heat stability at pH values acid to the maximum but causes slight destabilization in the minimum pH range and extends the minimum over a wider pH range.

#### d) a s1 - and b -caseins

b -casein plays an important role in micellar structure and heat stability although natural variation in the a s1: b - ratios are relatively small.

# e) Whey Proteins:

Serum proteins exercise a major effect on the shape of the HCT/pH curve.

### f) b -lg:

b -lg is one of the major whey proteins found to exist in dimeric form in milk. Under the action of heat, dimer is cleaved into monomeric form, and there is formation of reactive free SH-groups on account of marginal group containing cysteine/cystine. The cleavage of b -lg favours polymerization and also the reaction between b -lg and  $\kappa$ -casein. The other observations are:

- (a) The interaction between b -kg & κ-casein is confined to a narrow pH range of 6.7 to 7.0.
- (b) At lower pH, direct denaturation of whey proteins occurs.
- (c) In addition to disulphide bonds, hydrophobic bonds also take part in the interaction between  $\kappa$ -casein & b -lg in the colloidal phase thus, enhancing the heat stability of milk in the pH range of 6.7 to 7.0.
- (d) An increase in pH of milk (below pH 6.8) could be induced by the addition of b -lg during heating. Thus it is possible to counteract the reduction of pH caused by heating, and increase in heat stability.
- (e) The ratio of b -lg to  $\kappa$ -casein may be more important in heat stability than the concentration of these proteins, suggesting that surface characteristics of the micelle are critical in heat stability.
- (f) The maximum in the HCT/pH curve is a consequence of low stability at pH ~6.9 which is due to heat-induced precipitation of calcium phosphate on casein micelles sensitized by complexation with heat-denatured whey protein.

# g) a -la:

Although a -la contains no sulphydryl groups, it does possess 4 disulphide bonds per mole. a -la and b -lg have essentially the same effect on heat stability and are about equally effective on a weight bases. a -la & b -lg can form intermolecular complexes, presumably via disulphide interchange.

#### h) Ovalbumin:

It causes destabilization of milk in the pH range 6.5 -7.4 and 6.4 – 7.6 respectively.

### i) Lysozyme:

It markedly reduces the stability of milk at unadjusted pH values.

# j) Colloidal calcium phosphate:

Colloidal calcium phosphate (CCP) is an integrating factor in the casein micelle. Native micellar structure is necessary for the occurrence of a typical maximum/ minimum in the HCT/pH curve. Calcium ion and CCP play at least partially similar and interchangeable roles in heat stability.

- § Removal of CCP increases stability at all values throughout the pH range 6.3-7.0 and the minimum in the HCT/pH curve disappear when greater than 30% of the CCP is removed. The effect is reversible provided not more than 30% of the CCP is solubilized.
- § The destabilizing influence of CCP below pH 7.1 may be due to masking of organic phosphate groups.

### k) Lactose:

- Enrichment of milk with lactose decrease stability. Lactose acts as a destabilizing
  factor by virtue of acting as a source of heat-induced acids, in the presence of oxygen.
  Lactose reduces the stability and modifies the HCT / pH curve. It is involved in
  Maillard browning and apparently urea increases stability only in the presence of
  lactose.
- Protein-carbohydrate interactions, on the contrary, increase the heat stability of milk according to the reactivity of the carbonyl group attached to the carbohydrate moiety
- When 50-70% hydrolysis of the lactose in milk using the enzyme b -galactosidase is carried out, heat stability increase is maximized.

#### l) Urea:

- § The seasonal variation in heat stability is significantly correlated to changes in the naturally occurring level of urea in milk. Between 72 and 90% of the variation of heat coagulation time, measured at the original pH of the milk, is accounted for by changes in milk urea alone.
- The lesser amount of urea in buffalo milk (17.5 mg / 100 ml) as compared to cow milk (40 mg/100 ml) is one of the factors responsible for lower heat stability of buffalo milk.

# 15.5. Additives

#### a) Urea and other amides:

- § Addition of urea prior to heating has a marked stabilizing effect. At low concentrations (< 7mM) urea does not alter stability in the pH region of minimum stability (Type A) but does stabilize throughout the pH range 6.4- 7.4 at higher concentration (>15mM). Type B milks are stabilized throughout the pH range even by low concentrations of urea.
- § Added urea has been reported to have no effect or produce a small decrease in heat stability in concentrated bovine milk systems.

Thus, urea may perform at least two functions:

- (i) To react chemically to prevent cross-link formation and aggregation
- (ii) To diminish pH drift, thereby modifying the rate of formation of chemical cross-links.

### b) Other amides:

Urea, biuret, triuret, methyl- and ethyl-urea have similar effects on the HCT/pH curve and are about equally effective on an equimolar bases. They all are

- (i) Capable of forming homocitrulline from lysine
- (ii) Capable of involvement in Maillard browning with lactose and
- (iii) They reduce the rate of pH decline during heating

However, pH buffering is considered to be the most likely mechanism for heat stabilization.

# c) Aldehydes and sugars :

Formaldehyde, glycoal, glycol aldehyde, glyceraldehyde, erythrose and 2–deoxyribose stabilize milk and concentrated milk but several hexoses have no effect. In contrast to urea, which has no effect on the stability of concentrated milk, aldehydes are effective on both standard and concentrated milk. The reaction of aldehydes with  $\epsilon$ -NH<sub>2</sub> groups or the cross-linking of polypeptide chains may be involved.

- § Most of the sugars increased stability over the pH range 6.5 7.1, but at pH 7.2 7.4 had no effect.
- § Glyoxal, as well as simple aldehydes, is a very effective stabilizer of concentrated milk.
- $\S$  Urea and glyceraldehydes have synergistic effect of on the HCT pH curve of buffalo milk.

# d) Gums & Alginates:

κ-Carrageenan at concentrations upto 0.05% (w/v) stabilizes milk in the region of the minimum without a significant effect on stability at the maximum pH. Higher concentrations (especially >0.4%) cause marked destabilization throughout the pH range 6.4-7.4. Other hydrocolloids, e.g. carageenan K-100, CMC and Manucol esters, have little effect on heat stability at pH values below ~ 7 but cause destabilization at higher pH values.

# f) Detergents:

- § **Sodium dodecyl suphate**: Increasing additions of SDS upto 0.5% progressively shift the HCT / pH curve of milk to more acidic values without significantly altering its shape, and markedly increase maximum stability but have very little effect on stability in the minimum pH range. Casein micelles dissociate at higher concentrations of SDS.
- § Cationic detergents: Shift the HCT / pH curves to more alkaline values without significantly altering the shape but causing slight stabilization. Cetyltrimethyl ammonium bromide is particularly effective. The hydrophobic bonding may be involved.
- § The non ionic detergents: Triton X and Tween 60 added to milk at levels upto 3% (w/v) have no significant effect on heat stability throughout the pH range 6.4 7.4.

# g) Acid casein:

Casein addition does not have stabilizing effect on the fluid milk due to its effect on the salt balance.

### h) Chemical stabilizers (salts):

Salt balance is regarded as one of the most important factor in heat stability of milk. The effects of different salts on heat stability of cow & buffalo milk are:

- § Primary effect of stabilizers is a consequence of their influence on the pH of milk.
- § The heat stability of buffalo milk could be increased either by the addition of phosphate or citrate which reduce the ionic calcium content or by the replacement of 15% of calcium by sodium through electro metathesis.
- § Calcium has a drastic effect in destabilizing milk.
- § Addition of calcium chloride, sodium chloride and potassium phosphate significantly decrease the heat stability of buffalo milk.
- § Na<sub>2</sub>HPO<sub>4</sub> cause a significant decrease in the heat stability of buffalo milk, which is attributed to a significant increase in milk pH. On the other hand, addition of Na<sub>2</sub>HPO<sub>4</sub> to acidic milk restores its pH and heat stability. Sodium phosphate causes a shift in the pH of maximum stability towards the acidic side of fresh milk.
- § Ortho phosphates are generally the most effective stabilizers and CaCl<sub>2</sub>, which reduces intrinsic heat stability, should be used in moderation.
- § The point in the process at which stabilizers are added is not important and no single predictive test for the minimum level of stabilizer or combination of stabilizing salts required to optimize heat stability is apparent.

\*\*\*\*\*\*

# Lesson-16.

# Heat stability of Condensed milk

#### 16.1. INTRODUCTION

Heat stability of milk, concentrated and dried milks is of commercial importance. The term heat stability of evaporated milk refers to the relative resistance of the milk to coagulations in the sterilizer. Webb Bell and Deysher defined heat stability as "the time necessary to initiate coagulation at 115° C (239° F)".

### 16.2. Importance

- 1. Heat stability is important in the processing operation of concentrated product manufacture.
- 2. In the manufacture of recombined evaporated milk from anhydrous milk fat and skimmed milk powder, heat stability adjustment is important before sterilization. This is because of the convenience with which milk may now be stored and transported in the form of anhydrous milk fat and skimmed milk powder.
- 3. In the manufacture of recombined UHT milks
- 4. In the manufacture of beverages e.g., coffee whiteners must not coagulate or cause "feathering" when added to coffee usually under adverse conditions of temperatures and pH.
- 5. In the manufacture of low heat/high heat milk powders and used in admixture with fresh fluid milks.
- 6. In the manufacture of skim milk powders, stability of 'instant' dried skim milk when added to hot coffee showed that the formation of insoluble material was inversely related to the maximum coagulation time of the reconstituted powder measured at  $140\,^{\circ}$  C and directly related to the casein number of milk protein (i.e. % of milk protein precipitated at pH 4.6).

# 16.3. Heat Stability & Concentrated Milks

- § Although evaporated milk is produced in quantity, the process is very arbitrary.
- § Sommer & Hart (1926) found that apart from the destabilizing affect of albumin, the heat stability of the product is controlled by the ratio of calcium and magnesium to phosphate and citrate. Adjustment of the ratio by the addition of these salts, mainly phosphates, permits successful sterilization of the product in most cases. This 'salt balance' theory still remains as the major basis of the commercial sterilization of evaporated milk.

§ Powders made from milk which had not been preheated and which had a high level of undenatured whey protein nitrogen generally have a lower heat stability on reconstitution than had preheated powders. This reduction is even more marked when the powder is recombined as 18 % SNF, 8 % fat evaporated milk. In contrast, when the powders are reconstituted to the total solids level of the skim milk from which the powder was derived, the reverse effect is obtained and the non-preheated milk gives higher heat stability times. This effect is significant in the selection of powders for the preparation of sterilized milks.

§ The heat stability of the recombined product is, therefore determined by the skimmed milk powder, homogenization, and the final pH adjustment using phosphate stabilizers before sterilization. This creates a more complex situation as the combined heat stability characteristics of both the imported skimmed milk powder and the local milk supply will determine the suitability of the final product for sterilization.

 $\S$  In general, heat stabilization of 15 min at 120  $^{\circ}$  C can be taken as guide indicating adequate stability for commercial sterilization.

# 16.4. Optimum Heat Stability

The problem is not of achieving maximum heat stability at the cost of other defects but is to obtain optimum heat stability as to keep desired viscosity, body and texture. The maximum heat stability will eliminate the danger of curdling during sterilization but it would yield evaporated milk so lacking in body, i.e. viscosity that it would cause the milk to be "Rough" after sterilization. The stability may range from a few minute beyond the time held at sterilizing temperature to slightly over double the time held at sterilizing temperature.

# 16.5. Factors Influencing Heat Stability

The factors that influence heat stability of evaporated milk are related to the inherent properties of the milk such as chemical composition and freshness and the process of manufacture. The more important factors are:

### **16.3.1.** Acidity

In case of average normal milk, any increase of developed acidity has adverse effect on the heat coagulation of milk or heat stability of milk. This is true of the titratable acidity and of pH. It is further proved that the optimum acid reaction for maximum heat stability is dependent upon its relation to several variables of milk, such as salt balance and amount of milk proteins. Hence the optimum acid reaction for one lot of milk may or may not apply to any other lot of milk. The normal range of titratable acidity of freshly drawn milk is 0.15 to 0.16%. The sensitiveness of heat stability of milk and evaporated milk emphasizes the importance of getting fresh milk at condenser.

# 16.6. Effect of Concentration and Sterilizing Process on Acidity

The acidity of evaporated milk is higher than that of fresh milk from which it is made. The titratable acidity of evaporated milk before sterilization is approximately equal to that of fresh milk times the ratio of concentration. The acidity of milk after sterilization is more than that of before sterilization. This is attributed to the formation of acid resulting from the action of processing heat on some of the milk constituents. The pH of evaporated milk likewise is

lower than of fresh milk. Fresh milk averaged approximately pH 6.6 while evaporated milk falls in range of pH 6.15 to 6.3

#### 16.7. Milk Proteins

The nitrogenous constituents of milk are the pivot on which heat stability centres. They are the substances which are coagulated. The relation of all other ingredients or factors to the heat coagulation of milk is important only in so far as they influence the sensitiveness of the major proteins of milk to heat. Proteins of greatest importance here are casein and lactalbumin. Both are coagulated by heat. It is reported that 68.6% of total albumin found in fresh milk is precipitated in evaporated milk after sterilization. In normal evaporated milk of correct viscosity, the casein coagulated does not exceed 10% of total present.

- § The casein is of primary importance. It represents 80% of the total proteins in milk. It curdles at ordinary temperature by acid. In fresh sweet milk, it will coagulate at 136°C or higher but even at flash pasteurizing temperature it loses its power to react normally to rennet. The condensing of milk lowers heat coagulation point of casein. The heat stability of casein is greatly influenced by the balance of mineral salts of milk. Webb stated "the problem of heat coagulation of milk is in reality a problem of heat stability of calcium caseinate system"
- § The lactalbumin is present to the extent of 15% of the milk proteins. It is partially coagulated by heat in normal milk and completely in the presence of an acid medium. It is neither affected by rennet, nor by acid at ordinary temperature nor, by the mineral constituents of milk.
- § When forewarming is kept above 90°C, the initial viscosity and the increased viscosity are in direct relation to the amount of albumin added. But when forewarming temperature is held below 60°C, the initial viscosity remains the same and the changes which are small have no relation to the albumin content of milk.
- § Sommer demonstrated that even a small increase in lactalbumin content greatly reduces the time of sterilization to coagulate the evaporated milk.
- § Both the casein and albumin content of milk vary with season of year due to change in lactation period of animals. Variation is greater in case of albumin content as compared to casein. The percent of casein and albumin is higher at the beginning and towards the end of lactation period. Colostrum milk tends to decrease the heat stability of the milk.
- § Thus, the effects of protein composition specifically concern the ratio between κ-casein and  $\beta$ -lactoglobulin. The larger the amount of  $\beta$ -lactoglobulin, the higher the maximum HCT and the lower the minimum. This is explained by  $\beta$ -lactoglobulin enhancing the dissociation of κ-casein at pH >6.7, which results in formation of more strongly depleted micelles. The higher maximum HCT at pH 6.6 may result from an increased association of  $\beta$ -lactoglobulin with the micelles, which may enhance colloidal repulsion.

# 16.8. Products of Bacterial origin other than Acidity

Bacterial contamination of milk may lower the heat coagulation point of evaporated milk through activities other than acid formation. Certain species of bacteria produce rennin and rennet like enzymes. Heavy contamination with this type of organisms is capable of lowering the heat coagulation temperature. Thus, bacteria causing sweet curdling when present in large number will lower heat stability of milk.

#### 16.9. Mineral Salts Balance

The temperature at which evaporated milk curdles in the sterilizer is affected, and to a larger extent controlled by the balance of the milk salts.

# 16.10. Sommer and Hart's Theory of the Salt Balance:

They were first to demonstrate that aside from albumin, salt balance has a greater effect on heat stability and salt balance is readily changed by other changes like acidity, thus affecting, coagulation temperature.

- § It was observed that casein has maximum heat stability when in combination with a definite optimum amount of calcium. When the Ca<sup>++</sup> content available for the calcium casein complex is above or below this optimum combination, the casein is less stable to heat. The calcium contained in the milk distributes itself between casein, phosphates and citrates. In addition, the Mg present reacts by replacing the Ca in the PO<sub>4</sub> and citrates. The effect of calcium and Mg being basic radicals is opposed to the effect of PO<sub>4</sub> and citrates which are acid radicals. The calcium casein combination is at its optimum of heat stability when above two groups of mineral salts are in balance, hence the term salt balance. An excess or deficiency of either group accelerates heat coagulation.
- § If coagulation in heat test is due to deficiency of Ca and Mg, it can be prevented by the addition of proper amount of soluble Ca or Mg salts such as Ca and Mg acetates or chlorides. Such milk may also be stabilized by a slight increase in acidity because the increased acidity changes secondary PO<sub>4</sub> to primary PO<sub>4</sub> and the primary PO<sub>4</sub> have little or no effect on the salt balance. This change diminishes the amount of PO<sub>4</sub> that ties up the Ca and more Ca<sup>++</sup> is available to satisfy the Ca<sup>++</sup> casein equilibrium. In such cases slight increase in acid thus improves the Ca-casein balance and actually raises the heat coagulation point.
- $\S$  If troublesome heat coagulation is due to high  $Ca^{++}$  and  $Mg^{++}$  it can be prevented by addition of the proper amount of  $PO_4$  or citrate such as di-sodium phosphate or sodium citrate.
- § It is found in most cases that low heat stability is due to excess of Ca and Mg. Hence the heat stability is mainly controlled by exclusive addition of salts of  $PO_4$  and citrates. And since  $PO_4$  is cheaper of the two, it is preferred over citrate.
- § The variation in concentration of the ions such as buffer salts as PO<sub>4</sub>, citrate especially those of citrate have a greater effect on the coagulation temperature than slight variation in the pH of normal milk within range of 6.58 to 6.60. But both factors must be considered.
- § It is concluded that each lot of milk represents a separate colloidal system and that for each system there is an optimum combination of salt balance, due to such factor as pH with which optimum heat stability is attained.

 $\S$  Thus, the main effect of salt composition is through the calcium and phosphate contents. The addition of a certain salt to milk can strongly disturb all salt equilibriums involved. Addition of calcium and phosphate to milk, up to the concentrations that are found in concentrated milk, causes its heat stability at pH > 6.8 to be equal to that of concentrated milk, i.e., zero.

#### 16.11. Factors related to Process of Manufacture

In view of the problems associated with the concentrated and dried milks, the more important processing operations affecting the heat stability of these products are described below:

# 16.11.1. Forewarming

The temperature at which the fresh milk is forewarmed is an important factor in the control of heat coagulation of evaporated milk. For unconcentrated milk, preheating mainly causes a shift of the heat coagulation curve to lower pH values, and the stability at the pH optimum is hardly affected. But a very intensive preheat treatment (e.g. a few minutes at 150  $^{\circ}$  C) causes heat stability to increase over the whole pH range and the minimum in the curve disappears. Perhaps, the second heat denaturation of b -Lg, which occurs near 140  $^{\circ}$  C, is involved.

Preheating of milk at 80  $^{\circ}$  C, 90  $^{\circ}$  C and 120  $^{\circ}$  C in the presence of aldehyde and sugar significantly increased heat stability of buffalo milk. Maximum heat stability of concentrated buffalo milk can be achieved by preheating milk at 80  $^{\circ}$  C.

Concentrated (evaporated) milk hardly can be sterilized without preheating the milk before concentrating. The heat stability maximum is shifted to lower pH values and becomes higher. The pH of the milk before preheating also affects the heat stability of the evaporated milk, its optimum being, for instance, 6.45. The beneficial effect of preheating must be caused at least partly by reactions of the b-Lg, addition of this protein to the milk decreases the heat stability of evaporated milk, but this decrease can be eliminated largely by proper preheating.

Preheating at ultra-high temperatures for short times produces a 'concentrate' which is very stable to subsequent heat processing but the product has a low viscosity and is therefore unsuitable for the manufacture of satisfactory evaporated milk. The stability of recombined, evaporated milk is considerably improved if the original milk is preheated at 120 ° C for 2 min. The maximum stability occurs at the acid side of the natural pH and pH manipulation might best be done using a mixture of NaH<sub>2</sub>PO4 and Na<sub>2</sub>HPO<sub>4</sub> rather than HCI.

In practice, the pre-heating temperature for the manufacture of heat – stable skimmed milk powder, temperatures of 85-90  $^{\circ}$  C with holding periods of 10-20 min have been commonly used. However, there is evidence now of the use of higher temperatures in excess of 100  $^{\circ}$  C with shorter holding periods.

# 16.11.2. Homogenization

 $\S$  Homogenization of skim milk has little effect on its heat stability, but the stability of fat – containing products is decreased by homogenization, the magnitude of the effect being dependent on the temperature at which homogenization is performed, the extent of destabilization is greatest at 60  $^{\circ}$  C and least at 80  $^{\circ}$  C.

§ Homogenization has no significant influence on the heat stability of buffalo milk. However, after concentration, the heat coagulation time of homogenized milk decrease significantly. Homogenization causes a significant increase in the molar ratio of (Calcium + Magnesium) / (Phosphate + Citrate) which in turn destabilize homogenized concentrated milk. A reduced stability with increasing homogenization pressure from 3.5 to 34.5 MPa is observed. The destabilizing effect of homogenized milk could be partly offset by two stage homogenization (20.7 MPa followed by 3.5 MPa) or by the addition of phosphate stabilizer (0.08% w/v) or by homogenizing at high temperature (65°C).

#### 16.11.3. Concentrated milk:

- § Homogenization has little effect on milk, but it renders concentrated milk far less heat stable. The detrimental effect can be offset for a considerable part by preheating milk before concentration.
- § Heat stability of fat containing concentrated milk products is found to decrease by homogenization.
- § Concentration (31 % TS) before homogenization result into more stable product.
- $\S$  The heat stability of concentrated milk could be enhanced to a greater extent by high temperature fore warming (145  $^{\circ}$  C, 5 sec), two stage homogenization and addition of sodium phosphate.
- § Homogenization tends to slightly lower the heat stability of evaporated milk. This tendency increases with increasing homogenization pressure, and as the level of heat stability determined by the forewarming treatment drops.
- a. With milk of high heat stability such as results from HTST forewarming at 120°C for 4 minutes, the pressure of homogenization has no effect on heat stability.
- b. With the milk of normal heat stability such as result from forewarming at  $90^{\circ}$ C for 10 minutes, the homogenizing pressure lowers the heat stability only slightly.
- c. With milk of low heat stability, such as results from forewarming at 65.5°C, the heat stability drops considerably with increasing homogenizing pressure.

### **16.11.4.** Homogenizing Pressure :

Milk forewarmed at 90°C for 10 minutes having total solids 26.44% have heat stability as shown in Table 16.1 below:

Table-16.1: Heat stability and the corresponding Temperature and Pressure of Homogenization.

Homogenization Temperature	300	C	45	° C	60	°C
Homogenization Pressure, kg/cm <sup>2</sup>	105	175	35	175	105	175
Heat Stability at 115°C	16	15	14	11	13	10

Excessive homogenizing pressure that is more than 210 kg/cm<sup>2</sup> definitely lowers the heat coagulation temperature of evaporated milk.

### **16.11.5. Storage**

Normal milks can be stored at 4 ° C for at least one week without significant change in heat stability, suggests that the activity of indigenous milk proteinase is of little consequence to heat stability, at least under normal circumstances. The growth of proteolytic psychrotrophs in milk on prolonged storage at refrigeration temperature does not have a significant effect on the heat stability of milk until they reach populations of ~10 CFU/ml.

#### 16.11.6. Concentration Process

- § The concentration of evaporated milk has a marked effect on heat stability. As the concentration increases, the heat coagulation temperature drops. Particularly with 25.9% TS, a forewarming temperature slightly below the boiling point is suitable. At higher concentration, however, the evaporated milk becomes increasingly curdy.
- § The titratable acidity of evaporated milk after sterilization calculated as lactic acid, definitely exceed that contained in the fresh milk times the ratio of concentration.
- § The increased concentration of acid, albumin and casein respectively tends to lower the heat coagulation. Sommer and Heart suggested that with the higher acid reaction in the concentrated milk, there is an appreciably smaller fraction of secondary phosphates, which in turn has the effect of accentuating the unfavourable influence of the usual excess of calcium in the milk.
- § For every percent of difference in concentration within the limits of 16-26% SNF, there was a change of 1.25 to 1.50<sup>o</sup>C in temperature of heat coagulation.
- § A concentration below 14% SNF and high forewarming temperature (95°C) causes to lower the heat coagulation point while with concentration above 14% SNF, the same forewarming temperature increases the heat stability.
- § "Low-heat stability can no longer be considered a factor which might limit evaporated milk to 26% solids content" because an improvement in the heat stability can be obtained by keeping optimum forewarming temperature at 120°C with a holding time of 3-4 minutes.
- § Concentrated buffalo milk is less stable than concentrated cow milk.
- § Concentration of buffalo milk result in decrease in pH and in the proportion of calcium, magnesium, phosphate and citric acid in dissolved phase and increase in the ratio of calcium / phosphate and (calcium + magnesium) / (phosphate + citric acid).
- § The stability of the calcium caseinate phosphate complex to various coagulating agents declines on concentration.
- § Concentration induces major changes in milk system such as:
- (i) closer packing of casein micelles

- (ii) A higher concentration of denaturable whey proteins (native whey protein > 0.9% decreased HCT)
- (iii) Precipitation of calcium phosphate which also causes a significant decrease in pH. If the pH of a 9% SNF milk is 6.6, that of a 26% solids concentrate will be  $\sim$  6.2 and of a 40% concentrate  $\sim$  6.0.
- (iv) Due to water binding by proteins, lactose and salts there is very little free water in 3:1 concentrate.
- (v) Milk concentration by ultrafiltration (rather than by evaporation) is much more heat stable than evaporated milk.
- (vi) Concentration increases the rate of dephosphorylation, preheating has no effect on rate of dephosphorylation of unconcentrated milk but reduce the rate for concentrated milk.

### **16.11.7.** Additives

#### Urea and lactose:

- § Urea does not significantly increase the stability of concentrated milk (25% TS) although it does act synergistically with aldehydes.
- § Addition of simple aldehydes brings about large increase in heat stability of milk over a comparatively wide pH range. Some sugars which react readily as an aldehyde on heating are also shown to stabilize concentrates at high temperature (120 ° C).

### Alkali and salts

- § Sodium hydroxide @ 0.05 to 0.10% and trisodium phosphate @ 0.075% (additions before fore warming) increase the heat stability of 18.5% to 22.5% total solids skim milk concentrates but calcium chloride has the reverse effect.
- § In order to ensure concentrated milk to be heat stable, it is essential to employ stabilizing techniques before homogenization. The primary effect of stabilizer is a consequence of influence on the pH of the milk.

#### Salts in buffalo milk concentrates:

Maximum increase in heat stability is noticed in preheated ( $120\,^{\circ}$  C, no holding) buffalo milk concentrate (35% TS) when disodium hydrogen phosphate is added @0.3% level. Addition of trisodium citrate at the same level is also found to increase stability but to a lesser extent. Added casein has some stabilizing effect on HCT of buffalo milk concentrate (1:2).

# 16.11.8. Effect of Heating the Milk after Concentration

The stabilizing effect of HTST is not confined to forewarming the fresh milk before condensing. The special heat treatment may be applied either before or after concentration of the milk but the temperature of heat treatment that reduces maximum stability is found to vary. Thus when employed as a part of forewarming process, a temperature of 120°C for 3-4

minutes yield maximum heat stability. When HTST heating is applied to the milk after concentration, a temperature of 150°C with no holding period produces maximum heat stability.

# 16.12. Heat Stability of Recombined Concentrated Milks

- § Heat stability for recombined EM is of utmost importance. It may be defined as the time taken for milk / milk concentrate to thicken or coagulate when heated under standardized conditions. It is a particular problem in evaporated product because of the concentrated nature of the product and need for sterilization.
- § The main factors affecting heat stability of recombined concentrated milks are similar to those for concentrated milks. However, concentration of milk and addition of butter oil during recombination has complicated relation with heat stability which is not yet fully understood.
- § Correction for improvement of heat stability is achieved by the similar methods as listed for the concentrated milks. However, heat treatment or addition of stabilizing salts such as phosphates together with dried buttermilk is in practice and regarded as most successful.
- § As heat stability of concentrated milks manufactured from whole milk or powders varies with season and stage of lactation, attempts have been made to extend the period over which heat stable powders can be manufactured.

The addition of sweet cream buttermilk powder before heat treatment increases overall heat stability, perhaps due to its high phospholipids content. Similarly, incorporation of soya lecithin has marked positive influence on heat stability of recombined evaporated milk.

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### Module 7

# Physico-chemical properties of condensed milk

# Lesson-17

#### 17.1. INTRODUCTION

Concentrated milks are liquid milk preserves with considerably reduced water content. Water is removed by evaporation. Preservation is achieved either by sterilization, leading to a product called *evaporated milk*, or by creating conditions that do not allow growth of microorganisms. The latter is generally realized by addition of a large quantity of sucrose and exclusion of oxygen. The resulting product is called *sweetened condensed milk*.

These products were initially meant for use in regions where milk was hardly or not available. The milks were packaged in small cans. The contents were often diluted with water before consumption to resemble plain milk. Currently, alternative products are used more often, such as whole milk powder or recombined milk. For the concentrated milks, some alternative forms of use are developed, and processing and packaging have been modified. The consumption of sweetened condensed milk has greatly declined.

#### 17.2. PRODUCT PROPERTIES

### 17.2.1. Flavour & Colour

§ Maillard reactions are of paramount importance for the flavor and color of evaporated milk. The temperature and duration of the heat treatment during manufacture determine the initial concentration of the reaction products, but ongoing Maillard reactions occur during storage, especially at a high temperature. The milk eventually develops a stale flavor also due to Maillard reactions. The flavor after a long storage time differs considerably from that directly after intense heating. This is because the complicated set of reactions involved leads to different reaction products at different temperatures. A sterilized milk flavor may be appreciated by some people when the milk is used in coffee. Off-flavors due to autoxidation need not occur.

§ When the milk is used in coffee, the brown color is often desirable to prevent the coffee from acquiring a grayish hue. The brown color depends greatly on the Maillard reactions, although the color of the fat plays a part.

# **17.2.2.** Viscosity

The *viscosity* of evaporated milk is often considered an important quality mark. Many consumers prefer the milk to be viscous. However, it should pour as like thin cream. This can be achieved by sterilization in such a way that visible heat coagulation is barely prevented. UHT evaporated milk is always less viscous and, therefore,  $\kappa$  -carrageenan is often added.

# 17.2.3. Age Thinning

If the original milk contains bacterial lipases and proteinases due to growth of psychrotrophs, these *enzymes* may remain active in the evaporated milk and lead to strong deterioration, i.e., soapy-rancid and bitter flavors, and to age thinning. Evaporated skim milk may even become more or less transparent due to proteinase activity.

#### 17.2.4. Nutritive Value

The *nutritional value* of evaporated milk can be significantly decreased as compared to that of plain milk. In-container sterilization can destroy up to 10% of the available lysine, about half of the vitamins  $B_1$ ,  $B_{12}$ , and C, and smaller proportions of vitamin  $B_6$  and folic acid. All of these changes are far smaller when UHT heating is applied.

The nutritive value of the sweetened and unsweetened condensed milk is very high. Both of them are rich in fat and fat soluble vitamins viz. A, D, E & K and body building proteins, bone forming minerals and energy giving lactose. While sweetened condensed milk is especially high in energy giving sucrose because of the added sugar, evaporated milk is suitable for infant feeding since it makes a soft curd and is easily digested.

#### 17.2.5. HEAT STABILITY

Concentrated milk is very less stable during sterilization than non-evaporated milk, and the fairly intensive homogenization applied decreases the heat stability further. Moreover, evaporated milk should increase in viscosity during sterilization. Essentially, the viscosity increases by incipient coagulation. A subtle process *optimalization* is needed to meet these requirements.

Therefore, the milk must be preheated before evaporation in such a way that most serum proteins are denatured. Otherwise, the evaporated milk forms a gel during sterilization due to its high concentration of serum proteins. Preheating is, for example, for 3 min at 120 °C.

### 17.2.6. Effect of Stabilization

The pH should always be adjusted. Preheating and evaporation have lowered the pH to about 6.2 or 6.1, and that is far below the optimum pH. In practice,  $Na_2HPO_4$ .  $12H_2O$  is usually added, but NaOH can also be used.

#### 17.2.7. Effect of Homogenization

Homogenization of evaporated milk does not lead to formation of homogenization clusters. It is often observed that a slight homogenization increases HCT, which cannot be easily explained.

UHT heating of evaporated milk after homogenization is not possible. Even traditional sterilization is difficult if the milk is highly concentrated or if the evaporated milk is intensely homogenized. There are some other factors affecting heat stability. It can be improved by lowering the calcium content of the milk before evaporation by means of ion exchange. Addition of 0.05%  $\rm H_2O_2$  or about 15  $\mu$  mol  $\rm Cu^{2+}$  (from 0.5 to 1 mg  $\rm .~kg^{-1}$ ) after preheating but before evaporation tends to increase the heat stability.

#### **17.2.8. CREAMING**

Creaming of evaporated milk eventually leads to formation of a solid cream plug that cannot be redispersed. This may be due to bridging of adjacent fat globules because of 'fusion' of the fragments of casein micelles in their surface layers. Accordingly, intensive homogenization is necessary. A higher viscosity of the evaporated milk often involves a slower creaming, but the relations are not straightforward. Generally, a high viscosity is due to approaching heat coagulation. The homogenized fat globules tend to participate in this coagulation and hence to form clusters, which will cream rapidly.  $\kappa$  -carrageenan is often added to decrease creaming rate. The homogenization has an adverse effect on the heat stability and, consequently, the homogenization pressure should not be high. The largest fat globules exhibit creaming, and therefore the aim should be to have the relative diameter of the globule size distribution as small as possible. The width is greatly affected by the type of homogenizer used.

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# Lesson-18

# **Age Thickening and Gelation of Condensed Milk**

#### 18.1. INTRODUCTION

When evaporated milk is kept, its viscosity may initially decrease slightly. This may be explained in terms of the casein micelle aggregates formed during sterilization which changes from an irregular to a spherical shape, and as a result the effective volume fraction decreases. Subsequently, the viscosity increases, and it becomes strongly dependent on the shear rate, the milk displays a yield stress, and a gel is formed that firms rapidly.

#### 18.2. MECHANISM

The mechanism involved in the age thickening or gelation is not quite clear. However, in most cases, gelation is not caused by proteolytic enzymes or Maillard reactions, although the latter parallel the gelation. Also, gelation is not related to heat coagulation. It does not depend significantly on the pH, and its rate increases rather than decreases after lowering of the calcium content.

Electron microscopy reveals that thread-like protrusions appear on the casein micelles, which eventually form a network. It is likely that a slow change in the micellar calcium phosphate is at least partly responsible for the changes observed, but a definitive explanation is still lacking. Age thickening and gelation occur faster in UHT-evaporated milk. It may then be due to proteolysis caused by enzymes released by psychrotrophs, but also if such enzymes are absent, fast age gelation occurs. A more intense sterilization after evaporation delays gelation whereas it is faster in more concentrated milk and at a higher storage temperature.

It is observed that addition of sodium polyphosphate delays gelation considerably. Addition of citrate or orthophosphate often accelerates gelation, presumably because of binding of calcium. Polyphosphates may be hydrolyzed to yield orthophosphate, especially during heating. Consequently, addition of polyphosphate does not counteract gelation of in-bottle-sterilized evaporated milk.

Conventional evaporated milk only gels if kept for a long time at a high temperature. Extensive Maillard reactions also occur. Rapid gelation can occur if the evaporated milk before its sterilization is kept refrigerated at 4 ° C for a few days.

#### 18.3. SWEETENED CONDENSED MILK

The sweetened condensed milk is highly concentrated. Its mass concentration ratio is very high and because of this and the high sugar content, the product is highly viscous, i.e.,  $\eta_a$  is approximately 2 Pa . s, about 1000 times the viscosity of milk. The product is somewhat glassy in appearance because the fat globules show little light scattering as the refractive index of the continuous phase is almost equal to that of fat. The turbidity of the product is largely due to lactose crystals. Most of the lactose crystallizes because of its supersaturation.

# 18.4. RECOMBINED CONCENTRATED MILKS

# **18.4.1. Viscosity**

Viscosity of recombined EM is one of the important aspects in consumer acceptance which is decided by original powder and conditions of processing and stabilization. Age thinning is a problem when high storage temperatures are employed. High total solids and HTST method of sterilization give age thickening or gelation defect.

Viscosity, the most important property of recombined SCM, should be ~ 35 poise in fresh product which can increase up to 3 times, yet giving good acceptance. Too low viscosity poses air incorporation at filling and fat separation problems. Too high initial viscosity accelerates rate of age thickening.

Means to control viscosity of recombined SCM, in order of importance are:

- 1. Manufacture,
- 2. Selection of milk powder,
- 3. Homogenization,
- 4. Pasteurization and
- 5. Storage temperature.

#### 18.4.2. Colour

The color of recombined EM varies with use of anhydrous milk fat, powder and level of heat treatment. Colour and flavour of recombined SCM are affected by raw materials used and severe pasteurization conditions employed. Homogenization is found to lighten the colour.

# **18.4.3. Flavour**

Flavour of the product can be improved by use of buttermilk powder due to higher phospholipids content. Holding the product for some period, at least for 7 days after production and before release, allows post manufacturing hydration of dry ingredients and normalization of flavour characteristics.

The most preferred flavour in recombined WMP results when fresh whole milk, commercial additives and fresh buttermilk powder contributing number of flavour compounds lost during the preparation of anhydrous milk fat are included in the mix. Incorporation of distilled monoglycerides or buttermilk powder provides best flavour stability on extended storage of recombined WMP. Powders with moisture level below 3% provide best flavour initially and during storage. Moisture content >5% leads to crystallization of lactose resulting in lumpy powder, increased free fat and increase in colour development within 2-3 months.

# 18.4.4. Fat Separation

The efficient homogenization, absorption of casein on fat globule, product viscosity and minimum fat globule clustering controls the fat separation in recombined EM. High ambient temperature increases the defect. Addition of carrageenan gives best control of fat separation

but have greater tendency towards gelation. Formation of a stable fat globule from anhydrous milk fat and SNF is very important for recombined evaporated milk.

Homogenization reduces fat globule size as well as increases viscosity thereby reduce fat separation tendency. Commonly employed pressures are up to 10 MPa, about 3.5 MPa being most common. Homogenization of reconstituted skim milk with milk fat (at 50 kg/cm² or 150 kg/cm²) increases viscosity, while recombined SCM made using recombined cream (0.7-0.9 % protein, 20.2-20.9 % fat) age thickens slowly. Age thickening is also accelerated by higher storage period and temperature as well as by higher proteins and lower whey protein nitrogen with changed casein fraction. The pasteurization conditions can be used to control viscosity but is less convenient to use.

# 18.4.5. Lactose Crystallization

The second important quality determining factor for recombined SCM is lactose crystallization which can be controlled by storage temperature and using dry seeding procedure.

# 18.4.6. Other factors important for quality aspects

- § Fortification of recombined SCM with 1000-3000 IU of Vit-A and 30 mg of ascorbic acid is recommended.
- § Holding period of up to 2 weeks before release of recombined SCM in the market is desirable to allow post manufacturing hydration of dry ingredients and normalization of flavour characteristics.

High temperatures preheat treatment of milk, as part of manufacture of full-cream milk powder; can reduce the susceptibility of product to oxidation during storage as a result of development of sulfhydryl compounds. However, such heat treatment is not always compatible with the development of required specific functional properties.

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### Lesson-19

### **Defects in Sweetened Condensed Milks**

#### 19.1 INTRODUCTION

The major defects that are characteristics of unmarketable sweetened condensed milk are discussed here under:

# 19.2. Sandiness

The relative smoothness of texture of sweetened condensed milk is determined mainly by the size of the lactose crystals it contains. Sandiness is due to the presence of relatively large, coarse crystals. For a product of the desired smoothness, the crystal should average  $\leq 10\mu$  in length.

## 19.2.1. Sandiness Due to Sucrose

When the sucrose ratio of finished product exceeds 64.5 %, it approaches the status of a saturated solution of sucrose. When such condensed milk is subjected to low temperature such as in cold storage or otherwise on its journey from factory to consumer, some of the sucrose that is present in excess of saturation will crystallize. They give the condensed milk a coarse, sandy texture. Incomplete solution in the pan has also been found to be a contributing factor of sandiness.

When sweetened condensed milk that has been allowed to become too cold for packaging is re-warmed in the presence of agitation, the development of sandiness is unavoidable. Vigorous agitation while warming tends to cause the smaller lactose crystals to redissolve. This increased super-saturation promotes diffusion of the dissolved lactose. The numbers of crystals present is thus much reduced, so the remaining crystals have relatively much dissolved lactose to grow, forming sufficiently large aggregates to give the milk an objectionable sandy character as can be seen from Table 19.1.

Table-19.1: The quality of sweetened condensed milk and the numbers and size of lactose crystals

No. of Crystals	Size of crystals in μ	Quality
400000	Less than 10	Excellent
300000	Less than 10	Good
150000	More than 10	Slightly Pasty
50000	More than 10	Mealy
25000	More than 10	Fine Sandy
5500	More than 10	Very Sandy

#### 19.2.2. Sugar Sediment or Sugar Down

Deposits of sugar sediment on the bottom of can or barrel of sweetened condensed milk are not an uncommon occurrence. The microscopic examination as well as chemical analysis

shows such sediments to consist predominantly of lactose crystals. The main cause is due to the difference in specific gravity between the crystallized lactose and the remainder of the condensed milk. The specific gravity of lactose hydrate at 68°C is 1.5453. The average specific gravity of sweetened condensed milk is 1.3085.

It is reported that sugar sediment in sweetened condensed milk of a given viscosity increases or decreases respectively with an increase or decrease of the crystal size. Condensed milk of normal viscosity in which the lactose crystals do not exceed  $10\mu$  in length generally remains free from objectionable sugar sediments.

# 19.2.3. Effect of Viscosity on Sugar Sediment

The greater the viscosity, greater is the resistance to the force of gravity. Hence lesser will be the ability of the lactose crystals to drop to the bottom. In many cases of sediment trouble, the finished product is definitely too thin to hold the crystals in permanent suspension. In such cases reasonable increase in concentration may avoid sediment, provided that crystal size is dependably controlled.

## 19.2.4. Effect of Homogenization

Homogenization increases the viscosity of milk. This in turn decreases the tendency for the finished product to drop the lactose crystal. This effect increases with increasing pressure.

# 19.2.5. Effect of Super Heating

Due to super heating, there is swelling of casein which gives the product more body and tends to retard the sugar sediment.

# 19.3. Age Thickening

It is a common defect of sweetened condensed milk. It may be due to:

- 1. Bacterial Activity
- 2. Physico chemical reactions

Bacterial thickening is caused by extremely heavy bacterial contamination with the causative species of micro-organisms, which results from lack of efficient sanitary supervision in manufacture. It can be prevented by proper sanitation and efficiently controlled forewarming temperature. In the presence of causative organism; it can be controlled by increasing the sucrose in water ratio to nearly 64.5 %.

The exact reactions involved in Sweetened condensed milk age thickening due to physicochemical causes have not yet been fully explained but it may be due to:

- 1. Hydration of proteins mainly the casein which adsorbs water and thickening is increased
- 2. The influence of season on age-thickening is very marked.
- 3. Proper adjustment of fore-warming temperature prevents the defect age thickening.

#### 19.3.1. Effect of Sucrose Content

An increase of the sucrose in water ratio decreases the tendency to age thickening. An increase in addition of sucrose to the fresh milk used in the manufacture of sweetened condensed milk reduces the viscosity of the concentrated milk and retards or inhibits age thickening. The ratio should be adjusted according to the seasonal stability or instability of the product.

### 19.3.2. Effect of Time of Adding the Sucrose

Fore warming the milk and sugar syrup separately, cooling to pan temperature (55°C), then mixing sugar syrup and milk and drawing the mixture to pan decreases the tendency of age thickening. Adding sugar near the end of the condensing period tends to cause age thinning and may cause fat separation.

# 19.3.3. Effect of Pan Temperature

Condensing the milk at high temperature tends to destabilize the product and increase the tendency to age thickening. Lowering of the pan temperature near the end of the condensing period is effective in lessening the viscosity and thus decreasing the tendency to age thickening.

# 19.3.4. Effect of SNF on Age Thickening

An increase in the % of SNF and especially in the ratio of SNF–in-water increases the tendency to age thickening. So, SNF content should be adjusted before processing.

# 19.3.5. Effect of Ratio of Fat to SNF

The reasonable ratio should be provided such as 8.5% fat to 19.5% SNF provided by the U.S. Standard which has tendency to ameliorate the effect of influences that cause colloidal swelling of the proteins. The fat lessens the intensity of reaction. It acts as a softener and dilutant of the protein suspension, yielding product with plain and smoother texture than the product made from skim milk. The age thickening tendency is more in case of sweetened condensed skim milk.

# 19.3.6. Effect of Concentration

High concentration – high viscosity – more age thickening. Too low concentration – Fat separation and sugar deposits.

# 19.3.7. Effect of Acid Reaction

Fresh and good sweet milk has good stability and good keeping quality of condensed milk. If acidity is lowered by the addition of an alkali, there is improvement in storage stability of the product. Addition of  $\sim 0.2-0.4$  kgs of NaHCO<sub>3</sub>/100 kgs of raw milk is sufficient to stabilize the milk that is unstable towards age thickening.

# 19.3.8. Effect of Storage Temperature

In case of unstable milk, the age thickening tendency increase with the storage temperature. At 15°C, if the product is stored, there is decrease in age thickening. At 21°C the tendency of age thickening increases. It is observed that the rate of thickening approximately double with a rise in temperature of every 6°C.

#### 19.4. Flavour Defects

#### 19.4.1. Rancid Flavour

It is not a frequently occurring defect of sweetened condensed milk. It can be controlled by taking following precaution:

- (1) Fore warming the fresh milk at temperature above 76°C.
- (2) Avoid leaks of raw milk in to the batch during fore warming and between the forewarmer and vacuum pan.
- (3) Do not add sugar during fore-warming process.
- (4) Use unpolluted water and keep all the equipments in sanitary condition.

#### 19.4.2. Metallic Flavour

It may be caused due to the use of copper equipment. It may give pickery coppery taste of copper salts. Use stainless steel equipments.

# 19.4.3. Tallowy Flavour

It is due to the autoxidation of milk fat. Now-a-days it does not occur, but it may occur if the finished product is exposed for a considerable period of time to air and light and in plants that are still using copper vacuum pans and copper forwarmer.

# 19.4.4. Fat Separation

It is the result of abnormally low viscosity and thin body. In such milk at rest, there is definite tendency of some of the fat to rise to the surface forming a layer that has deeper yellow colour than fresh milk. This defect can be controlled by proper processing and proper concentration of total solids.

### 19.4.5. Dark Colour / Brown Discolouration

The immediate causes of the discoloration are the intense heat treatment during fore warming and high storage temperature. At low temperature, the colour is not noticeably affected by aging.

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# Lesson-20

# **Defects in Evaporated Milks**

#### 20.1. INTRODUCTION

The major defects that are characteristics of evaporated milk are discussed here under:

#### 20.2. NON-BACTERIAL DEFECTS IN EVAPORATED MILK

Evaporated milk, in order to be marketable, must be free from symptoms of biological fermentation. It must be sterile. In addition, it must have certain physically important properties such as full of body that symbolizes richness and prevents objectionable fat separation, smooth and homogeneous texture, a pleasing natural milk flavour and a light creamy colour.

Much attention is to be paid to this group of non-bacterial defects such as curdiness due to heat coagulation, fat separation, mineral deposit, browning, bloats due to freezing, change in altitude and chemical action by milk acids upon base metal of container because it involves defective process itself.

# 20.3. Curdiness Due to Heat Coagulation

For the dependable prevention of curdiness and other physical defects that dissipate the marketable properties of the finished product, it is necessary to efficiently control the viscosity of the evaporated milk. The causes and prevention of curdiness due to heat coagulation are briefly summarized as follows:

- A. Curdy evaporated milk is due to too low heat stability in the presence of heat treatment used for sterilization.
- B. The standard commercial sterilizing process of holding at 115.6 °C for 20 min or other temperature time ratios with equal lethal effect represents the minimum temperature time ratio that will insure a sterile finished product.
- C. When the cooking time (minutes held at full sterilizing temperature) approaches the heat stability time, there is danger of permanent curdiness in the finished product.
- D. If the heat stability of the evaporated milk is too low to ensure freedom from curdiness, it may be increased and curdiness prevented by use of higher forewarming temperature or by the proper use of casein stabilizer.
- E. Too old or otherwise inferior raw milk as well as holding the evaporated milk too long or at too high a temperature before sterilization are not the frequent causes of curdy evaporated milk.
- F. Higher homogenizing pressure such as pressure between 211 & 281 kg/cm<sup>2</sup> or higher lowers the heat stability and increases the tendency to curdiness. Raw milk of inferior quality

or condensed milk held improperly before sterilizing or homogenizing pressure in excess of 175 kg/cm<sup>2</sup> increases the tendency to a curdy finished product.

# 20.4. Fat Separation in Storage

Adoption of homogenization as an integral function of manufacturing of evaporated milk eliminates the defect of fat separation as a serious problem. Yet even in the homogenized product, the distribution of fat globules is not permanently uniform, on progressive storage of homogenized product, it becomes higher and richer on top layer.

If objectionable intensity of fat separation is to be avoided, it is necessary in addition to efficient homogenization (the fat globules uniformly to a diameter of 2  $\mu$  or less) to

- (1) Aim not for maximum heat stability but for heat stability sufficiently moderate to give evaporated milk a full body and a viscosity adequate to definitely retard fat separation in storage.
- (2) To accomplish this, the processor aims at a concentrate that possesses heat stability a few minutes longer than about twice that of the sterilizing period.
- (3) Varying the sterilizing process, yields the optimum viscosity for maximum fat emulsion stability.

# 20.5. Mineral Deposit

Under certain conditions, there is a tendency for a whitish gritty deposit to form near the bottom of the container. This deposit consists chiefly of tri calcium citrate. Its causes & prevention are as follows:

- (1) Citric acid is believed to be present in milk combined with Ca and Mg. The citric acid content of milk has been found to be greatest when the cows are on green pasture.
- (2) Higher the concentration of evaporated milk, the greater the tendency for appearance of a mineral deposit during storage.
- (3) The temperature of storage is a controlling factor. The higher the temperature, the larger the amount of deposit. At  $7.2^{\circ}$ C there is no mineral deposition.

# 20.6. Browning of Evaporated Milk

The reasons for browning are discussed somewhere else. However, the causes and prevention are summarized as follows:

- (1) The darkening of colour is due to reaction in the presence of sterilizing heat, between the sugar of milk and certain amino acid. This reaction causes discoloration of the proteins.
- (2) The Browning effect is much less intense in the case of high temperature short time heat treatment then LTLT.

(3) The colour of evaporated milk darkens with age. The rate of darkening increases with storage temperature. At 5<sup>o</sup>C or below there is no change of colour during storage.

# 20.7. Bloats of Evaporated Milk

The bloats are caused due to

- (1) Freezing,
- (2) Change in altitude and
- (3) Chemical actions.

Bloats resulting from these causes are frequently occurring. When freezing, the content expands sufficiently to cause the cans to buldge at their ends, giving the impression of bloats. When subsequently transferred to temperatures at which the milk melts, the buldging ends almost invariably flip back to normal.

Change to high altitude may cause tins to buldge. This will result especially when cans are sealed at low altitude and are then transferred to high altitude regions. The danger in such cases is intensified, if the evaporated milk happens to be cold at times of filling and atmosphere at high altitude is warmer causing the air and milk in can to expand. The pressure difference between inside and outside of tins thus causes the can ends to buldge out.

Chemical action of milk acid upon the base metal of the container affects the appearance of bloats.

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## Module 9

# Microbiological qualities of Sweetened condensed and evaporated milks

## Lesson-21

# **Microbiology of Sweetened Condensed Milk**

## 21.1. INTRODUCTION

Sweetened condensed milk is not sterile. It contains living microbes and spores. The low water activity (~ 0.83) or, rather, the high sugar content prohibits growth of most but not all microorganisms.

Deterioration usually occurs by osmophilic yeasts, most of which belong to the genus *Torulopsis*. The yeasts often cause gas formation (bulging cans), a fruity flavor, and coagulation of protein. Coagulation may result from ethanol production. As a result, the product becomes unacceptable. The yeasts do not start easily, especially if the sugar concentration is high. It may thus take several weeks for incipient growth to be perceptible.

Some micrococci may grow in sweetened condensed milk, although slowly, especially if water activity and temperature are high. Presumably, the presence of oxygen is required. It may happen that they grow to reach a colony count of, say,  $10^5$  ml  $^{-1}$  and then stop growing, without causing noticeable defects. If they keep growing, coagulates eventually form and several off-flavors develop.

Some molds, especially strains of *Aspergillus repens* and *A. glaucus*, can grow as long as oxygen is present. If so, fairly firm colored lumps are formed and an off-flavor develops. One spore in one air bubble can cause such a lump.

Obvious remedies for microbial spoilage include the killing of all saprophytes and mold spores in the milk and in the sugar. Bacterial spores cannot germinate in sweetened condensed milk. Growth of harmful microorganisms in the dairy plant should be rigorously avoided. No sugar and residues of the milk should be left about. Satisfactory hygienic standards must therefore be maintained, especially in the packaging room. Harmful microorganisms cannot grow during concentrating, but the machinery must be thoroughly cleaned, immediately after evaporation. Mold spores can be removed by air filtration. The packaging machine should fill the cans very accurately with a safety margin of 1 g. Too little condensed milk in the cans means that more air is left, which increases the chance of growth of molds and micrococci. If the cans are overfilled, the milk may spill over the side and encourage growth of osmophilic yeasts.

## 21.2. Spoilage due to Bacteria

Sweetened condensed milk has better keeping quality than plain condensed milk because high sucrose concentration retards quality damaging bacterial development. The bacterial count has been found to increase with age in some cases and decreases in others. In normal milk, these changes are very gradual, depending considerably on the temperature of the storage. In the case of heavy contamination leading to definite bacterial defects however, the rate of increasing of the causative organism is greatly expended. The chief bacterial defects observed in sweetened condensed milk are:

## 21.3. Mold Button Formation

This defect is generally absent in fresh product. The number and size of button will depend upon the type of causative organisms, age of the product and temperature of the storage and time. The button formation is mostly due to the mold *Aspergillus repens*. Buttons in sweetened condensed milk have also been traced to the molds.

## 21.3.1. Aspergillums Glalicus

The growth of mold is stopped as soon as oxygen is removed or decreased. But the size of the colonies will go on increasing because the enzyme produced by mold continues activity after the death of the mold. Due to this defect in condensed milk we may find lumps of variable size, cheesy consistency and whitish yellow to reddish brown colors. They constitute firm, self contained units that do not emulsify into the body of the milk. The buttons themselves have a stale cheesy taste and they develop in the milk a stale odor. These molds do not survive the customary range of forewarming temperatures used. The appearance of buttons is due to the contamination after forearming.

#### **Control**

Proper sanitation and following precautions in manufacturing and packing avoids recontamination of the product.

- 1. Keep as minimum air space as possible in tins
- **2.** Seal the product in vacuum of 51cm, below this mold will not grow.
- **3.** Store the product at 15<sup>o</sup>C or below.
- **4.** Inversion of condensed milk tins at regular intervals.

## 21.4. Bacterial Age Thickening

It is very common defect in storage. In most cases, this may be due to physico-chemical changes. However, there are certain micro-organisms which may cause age-thickening. These problems arise when raw milk is of very high count. When sweetened condensed milk has age thickening defect due to micro organisms, then taste of the product is changed and often acidity is increased. It is due to micro organisms of bacillus type, which may survive on heat treatment. There are both acid producers and enzyme producers which involve in age thickening. The thickening is invariably due to cocci type of organism. They are:

- 1. Micrococcus lactic albidus
- 2. Micrococcus freudenreichii

This defect is usually accompanied by a high germ count, a disagreeable stale and often cheesy flavor and taste. When the thickened milk is diluted with water and heated, the curd separates. Large number of *S. pyogenes*, *Staphylococcus aureus*, *B. subtilis* and *Bacillus megentricus* and yeasts are also present in such milk. It is observed that the acidity factor is non-important and the thickening is due to the production of rennin like enzyme.

#### **Control Measures:**

- 1. Elimination of sources of contamination by cleaning and sterilization.
- 2. Selecting milk of good quality
- 3. Raising sucrose concentration. The sugar ratio should not exceed 64.5 %, other-wise it may involve problem of sucrose crystallization.
- 4. Storing at low temperature

#### 21.5. Bloat

This defect is mainly due to gaseous fermentation and this defect is considered from consumer's point of view. It is a severe defect, people will not like to have even if little defect is developed. In case of canned milk, resulting pressure is so much that it may cause ends to bulge out and in extreme cases, it may burst out. In case of barrel, the head may be blown off. In case of sweetened condensed milk the aerogenous type of bacteria and certain type of yeast mainly named as Torula lactis condensi ferments the sucrose forming alcohol and CO<sub>2</sub>. Many investigators found the causative micro organisms to be budding yeast capable of fermenting sucrose in concentrated solution.

## 21.5.1. Mechanism of Gaseous Fermentation

The sucrose is inverted by the enzyme invertase contained in and secreted by yeast cells. In this inversion, the molecule of sucrose takes on one molecule of water and breaks down into 2 molecules of monosaccharide. The monosaccharides are readily fermented by this yeast.

Yeasts cells require  $O_2$  for metabolism and growth. They must be present in invigorated form and in sufficient numbers to expedite the air utilization in the condensed milk and in the sealed container before this limited  $O_2$  supply is exhausted by micro-organism other than sucrose fermenting yeast. Contamination must be sufficiently heavy to make available enough invertase to initiate inversion at once. When once started, inversion and fermentation proceed rapidly at or above room temperature.

Concentrated sucrose solution because of its relatively high osmotic pressure, tend to retard, if not prevent, the activity of microorganism. This effect is intensified with increasing concentration. A minimum sugar - in water ration of 62.5% will hold in check most of the species of micro organisms that are normally present in sweetened condensed milk produced under reasonable average attention to temperature control in manufacture, factory sanitation and quality of supplies. Under such conditions, danger of gaseous fermentation is remote. But not so in the presence of heavy contamination with invigorated sucrose fermenting yeast. Here, the sucrose concentration fails to prevent the development of bloats even when the sucrose is present in saturated solution. As the yeasts are generally insufficiently resistant to

the pre-heating temperatures applied, their presence must be due to contamination. It has been found that sugar may be source of contamination, especially when moist.

#### **Controls**

- 1. The most likely and most direct cause of bloat of sweetened condensed milk is contaminated sugar. Keep the sugar sealed in its original package until needed.
- 2. Use barrels in preference to sacks. Keep it protected from dampness and insects such as bees, flies cockroaches. Insects plus dampness cause inversion and fermentation.
- 3. Adding the sugar to the milk in the pan in the form of concentrated syrup in boiling hot water (~ 65% sugar-in-water) is a dependable safe guard.
- 4. Sanitation of all equipments, from hot well to filling mechanism, and protecting the condensed milk from prolonged exposure to air by filling into the final container as soon as possible after manufacture, are important additional precautions. Container should be filled as full as reasonable allowance for heat expansion will permit.
- 5. Keep less air space.
- 6. Store below room temperature.

#### 21.6. Flavour Defects

The usual bacterial flavor defects encountered are rancid, stale, tallowy etc. Generally it is due to hydrolysis of fat.

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# Lesson-22

# Microbiology of Evaporated Milk

## 22.1. INTRODUCTION

In the manufacture of Evaporated milk, the sterilization is primarily aimed at killing all bacterial spores — reduction to, say,  $10^{-8}$  spores/ml — and inactivating plasmin. Lipases and proteinases from psychrotrophs should be absent from the raw milk because these enzymes would be insufficiently inactivated. The most heat-resistant spores are those from *Bacillus stearothermophilus*. This bacterium does not grow at moderate temperatures but may do so in the tropics. At  $121^{0}$ C, the D value of the spores is some 4 to 7 min. The preheating suffices for a sterilizing effect almost equal to 1, whereas the sterilization gives almost equal to 3 at most and, hence, added together giving less than or equal to 4. Contamination by these spores should therefore be slight, and growth of the organism occurring in the evaporator, possibly followed by sporulation (e.g., during intermediate cold storage), should rigorously be avoided. If the sterilizing effect is adequate for B. *stearothermophilus*, then B. *subtilis*, *Clostridium botulinum*, and C. *perfringens* are also absent.

Thus, normal evaporated milk is sterile. However following observations are made:

- (a) It appears that evaporated milk, as purchased in the local market is not only free from pathogenic micro organisms but may, for all practical purposes be considered sterile.
- (b) From bacteriological stand point, evaporated milk may be considered a safe good for use in infant feeding.
- (c) Common bacterial defects of evaporated milk are
- a. Bloat caused by gaseous fermentation
- b. Bacterial coagulation and
- c. Off flavors due to bacterial causes of which bitter flavour is most frequently encountered.

# 22.2. Bloats of Evaporated Milk

Bloats caused by gaseous fermentation are commercially the most serious bacterial defect of evaporated milk. The responsible organism is commonly not always responsible. It may be due to post processing contamination of evaporated milk.

# 22.2.1. Characteristic Organisms Causing Bloats

Characteristic epidemics of bloats in commercial manufacture of evaporated milk that involve the great majority of serious out-breaks of bloats are caused by microorganisms of superior heat resistance. Such fermentation is due to anaerobic bacteria belonging to the

butyric acid group and in most cases, though not always the putrefactive prevail. Gas formation is very vigorous and is accompanied by putrid odour (foul odour).

The responsible organisms as reported are *Plectridium foetidium*. It has flagella, is spore forming bacteria and motile. It resembles to *B. Tetani*. At 32°C, it ferments milk in four days under anaerobic conditions. The milk first curdles and curd gradually digests, leaving a clear yellow liquid similar in appearance to butter oil. The fermentation is accompanied by the evolution of penetrating, foul, putrefactive odour suggesting H<sub>2</sub>S. The organisms survive 15 min at 118°C. Its thermal death point lies between 118-121°C.

## 22.2.2. Sources of Organism & Prevention of Defect

Plectridium foetidium belongs to a group of butyric acid group bacilli found abundantly in cultivated soil on field crops and on small grains. These organisms are more prominent in milk from stable fed cows than in milk from pasteures. They are found in cow manure. Prevention of defect thus involves improved sanitation in production of milk on the farm, or adjustment of temperature and time ratio of sterilization of the evaporated milk as will destroy these organisms if present in milk.

## 22.3. Coagulation of Evaporated Milk

Bacterial coagulation is probably the most common bacterial defect in evaporated milk. The coagulum found in these outbreaks has varied from the sweet curdling type to a sour curd and an intense bitter curd. Some reported curd to be sweet and no bitter taste when no acid was developed and curd was of varying firmness and complete absence of wheying off. Causative organism identified is *Bacillus cereus*. *Bacillus coagulans* was separated and identified from commercial coagulated milks. These organisms were not destroyed by a 10 min exposure to 80°C but failed to survive 112°C.

## 22.3.1. Bitterness in Evaporated Milk

Many instances of bacterial coagulum showed intense bitterness, suggesting protein breakdown and formation of peptone and other decomposition products of known bitter flavour. The coagulum was firm and striking whiteness. The content had wheyed off. This was practically clear. Acidity was normal 0.35 to 0.4%. Inoculation with sterile milk yielded pure culture of very small spore forming rods. Organism was facultative anaerobic with an optimum incubating temperature 32°C but failed to survive 110°C for 15 minutes.

## 22.3.2. Prevention of Defect

The extreme whiteness of contents of defective cans suggested insufficient heat treatment in sterilization as the cause of defect. Non-uniformity of heat distribution while sterilization, seems the main cause which should be prevented.

## 22.3.2. Fishy Flavour

Coagulated milk with fishy flavour development is reported by the causative organism *Proteus ichthyormis* in evaporated milk.

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## **Module 10**

# Freeze and membrane concentration

## Lesson-23

## **Freeze Concentration**

# 23.1. INTRODUCTION

Freeze concentration is an excellent alternative to evaporation and reverse osmosis for concentration of many liquid foods. Product quality is generally high since low temperatures are used and no vapor-liquid interface occurs. However, current commercial freeze concentration technology is not economically competitive with the more established alternatives. Freeze concentration is applied where focus is on aroma retention and high quality products. It is specially suited for heat sensitive products. Freeze concentration is used for coffee extracts, fruit juices, vinegar, beer, wine and practically any other aqueous solution

# 23.2. Process

§ Freeze concentration has been practiced for centuries. In its earliest form it was as simple as leaving a barrel of liquid outside in the cold winter night. Water would crystallize and grow as a thick layer of ice along the inside walls of the barrel. The next day they would simply cut a hole through the ice shell and drain the now concentrated product. The water (now ice) was simply discarded.

§ Understanding the principles by which ice crystals grow in fluid foods would aid in furthering freeze concentration technology. In particular, if optimal heat balance conditions can be maintained throughout the freeze concentration process, large, easily separated crystals can be grown in short times. Modern freeze concentration processes consist of a crystallization section, where part of the water is converted into solid ice crystals using a refrigeration system. The ice crystals are then separated by filters, centrifuges or using the wash columns

§ Now, through a process of innovative engineering, process simplification and component standardization, the patented technology has reduced both equipment costs and energy usage significantly making Freeze Concentration a practical option for the constantly growing number of applications throughout the food and drink sector.

## 23.3. Freeze Drying

Freeze-drying (also known as lyophilization or cryodesiccation) is a dehydration process typically used to preserve a perishable material or make the material more convenient for transport. Freeze-drying works by freezing the material and then reducing the surrounding pressure and adding enough heat to allow the frozen water in the material to sublime directly from the solid phase to gas. Freeze drying is a superior preservation method for a variety of

food products and food ingredients. The plant sizes available ranges from pilot scale to large industrial batches and continuous plants. During the freeze drying process deep-frozen products are dried at temperatures below -18°C in freeze dryer. No thawing of the product takes place, which ensures a high quality product.

## 23.4. Advantages of Freeze drying

The modern plants provide high-quality products for customers as well as unrivalled financial and operational advantages for the company by eliminating product loss, reduced energy costs and maximizing plant reliability and ease of use. Other benefits are:

- § The plants offer an advanced technology and efficient design to ensure the preservation of excellent quality in a wide range of food products, such as vegetables, temperate and tropical fruits, fish, meat, TV-dinners, coffee, flavour essences and several other products.
- § The original flavour, proteins and vitamins are preserved.
- § The products will retain their original shape, colour and texture.
- § Re-hydration is rapid and complete.
- § The process results in stable products with long shelf life.
- § The products are durable at a wide range of temperatures, eliminating the need for complicated cold chain distribution systems.
- § The low weight and easy handling of freeze dried products reduce shipping costs dramatically.

## 23.5. Applications of freeze-drying

Freeze-drying is a relatively expensive process. The equipment is about three times as expensive as the equipment used for other separation processes, and the high energy demands lead to high energy costs. Furthermore, freeze-drying also has a long process time, because the addition of too much heat to the material can cause melting or structural deformations.

- 1. Therefore, freeze-drying is often reserved for materials that are heat-sensitive, such as proteins, enzymes, microorganisms, and blood plasma. The low operating temperature of the process leads to minimal damage of these heat-sensitive products.
- 2. Freeze-drying is used to preserve food and make it very lightweight.
- 3. The process has been popularized in the forms of freeze-dried ice cream, an example of astronaut food.
- 4. It is also popular and convenient for hikers because the reduced weight allows them to carry more food and reconstitute it with available water.
- 5. Instant coffee is sometimes freeze-dried, despite high costs of freeze-dryers. The coffee is often dried by vaporization in a hot air flow, or by projection on hot metallic plates.
- 6. Freeze-dried fruit is used in some breakfast cereal.
- 7. However, the freeze-drying process is used more commonly in the pharmaceutical industry.

8. In bacteriology freeze-drying is used to conservate special strain.

# 23.6. Freezing

- § The freezing process consists of freezing the material.
- § In a lab, this is often done by placing the material in a freeze-drying flask and rotating the flask in a bath, called a shell freezer, which is cooled by mechanical refrigeration, dry ice and methanol, or liquid nitrogen.
- § On a larger-scale, freezing is usually done using a freeze-drying machine. In this step, it is important to cool the material to the lowest temperature at which the solid and liquid phases of the material can coexist. This ensures that sublimation rather than melting will occur in the following steps:
- o Larger crystals are easier to freeze-dry.
- o To produce larger crystals, the product should be frozen slowly or can be cycled up and down in temperature.
- o This cycling process is called annealing.
- o However, in the case of food, or objects with formerly-living cells, large ice crystals will break the cell walls.
- o Usually, the freezing temperatures are between -50 °C and -80 °C.
- o The freezing phase is the most critical in the whole freeze-drying process, because the product can be spoiled if badly done.
- o Amorphous (glassy) materials do not have a eutectic point, but do have a critical point, below which the product must be maintained to prevent melt-back or collapse during primary and secondary drying.
- § Large objects take a few months to freeze-dry.

## 23.7. Primary Drying

- 1. During the primary drying phase, the pressure is lowered (to the range of a few millibars), and enough heat is supplied to the material for the water to sublimate.
- 2. The amount of heat necessary can be calculated using the sublimating molecules' latent heat of sublimation.
- 3. In this initial drying phase, about 95% of the water in the material is sublimated.
- 4. This phase may be slow (can be several days in the industry), because, if too much heat is added, the material's structure could be altered.
- 5. In this phase, pressure is controlled through the application of partial vacuum.

- 6. The vacuum speeds sublimation, making it useful as a deliberate drying process.
- 7. Furthermore, a cold condenser chamber and/or condenser plates provide a surface(s) for the water vapour to re-solidify on.
- 8. This condenser plays no role in keeping the material frozen; rather, it prevents water vapor from reaching the vacuum pump, which could degrade the pump's performance. Condenser temperatures are typically below -50 °C.
- 9. In this range of pressure, the heat is brought mainly by conduction or radiation; the convection effect can be considered as insignificant.

# 23.8. Secondary Drying

- 1. The secondary drying phase aims to remove unfrozen water molecules, since the ice is removed in the primary drying phase.
- 2. This part of the freeze-drying process is governed by the material's adsorption isotherms.
- 3. In this phase, the temperature is raised higher than in the primary drying phase, and can even be above 0°C, to break any physico-chemical interactions that have formed between the water molecules and the frozen material.
- 4. Usually the pressure is also lowered in this stage to encourage desorption (typically in the range of microbars, or fractions of a pascal). However, there are products that benefit from increased pressure as well.
- 5. After the freeze-drying process is complete, the vacuum is usually broken with an inert gas, such as nitrogen, before the material is sealed.
- 6. At the end of the operation, the final residual water content in the product is around 1 to 4%, which is extremely low.

## 23.9. Properties of freeze-dried products

- 1. If a freeze-dried substance is sealed to prevent the re-absorption of moisture, the substance may be stored at room temperature without refrigeration, and be protected against spoilage for many years. Preservation is possible because the greatly reduced water content inhibits the action of microorganisms and enzymes that would normally spoil or degrade the substance.
- 2. Freeze-drying also causes less damage to the substance than other dehydration methods using higher temperatures.
- 3. Freeze-drying does not usually cause shrinkage or toughening of the material.
- 4. In addition, flavours and smells generally remain unchanged, making the process popular for preserving food. However, water is not the only chemical capable of sublimation, and the loss of other volatile compounds such as acetic acid (vinegar) and alcohols can yield undesirable results.

## CONDENSED AND DRIED MILK

Freeze-dried products can be re-hydrated (reconstituted) much more quickly and easily because the process leaves microscopic pores. The pores are created by the ice crystals that sublimate, leaving gaps or pores in their place.

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# Lesson-24

## **Membrane Concentration**

## 24.1. INTRODUCTION

Industrial membrane filtration plants were introduced in the dairy industry in the beginning of the 70's. The basis for using membrane filtration in the dairy industry is that the dry matter components in milk and whey particles consist of different sizes as shown in Table 24.1. By selecting filters/membranes of different pore sizes and applying pressure on the product to be filtered, it is possible to divide the milk and whey in different fractions. What passes the filter/membrane is permeate and what does not pass is retentate as shown in Figure 24.1.

# Fig. 24.1: Different Membrane Filtration techniques and molecular size permeation

## 24.2. MEMBRANE TYPES

- 1. **Spiral Membrane:** Due to their compact layout and large amount of membrane area per element, spirals are typically used for high-flow applications with minimal or no suspended solids. Their advantage is low hardware and operating costs.
- 2. **Ceramic Membrane:** Ideally suited for value-added or sanitary products, as well as applications requiring selective separations from fluid streams containing aggressive components such as solvents.
- 3. **Stainless Steel Membrane:** Rugged design, especially effective for demanding applications with extreme process conditions or feed streams with elevated particulate solids and/or high viscosity.
- 4. **Tubular Membrane:** Highly resistant to plugging when processing streams with large amounts of suspended solids or fibrous compounds.
- 5. **Hollow Fiber Membrane:** Extremely high packing density and open channel design; offers the possibility of backwashing from the permeate side, particularly suited for low solids liquid streams.
- 6. **Plate and frame:** Open channel design allows it to be used for products with very high viscosity, particularly suited for high solids pharmaceutical and food applications.

## 24.3. FILTRATION TYPES

## 24.3.1. Reverse Osmosis

Reverse Osmosis is used to remove water from a product to increase the solids content; evaporator condensate is often 'polished' by reverse osmosis, so that it can be used elsewhere in the dairy. Reverse osmosis is a high-efficient technique for dewatering process streams, concentrating/separating low-molecular-weight substances in solution, or cleaning wastewater. It has the ability to concentrate all dissolved and suspended solids. The permeate contains a very low concentration of dissolved solids. Reverse Osmosis is typically used for the desalination of seawater.

In order to describe Reverse Osmosis, it is first necessary to explain the phenomenon of osmosis. Osmosis may be described as the physical movement of a solvent through a semi-permeable membrane based on a difference in chemical potential between two solutions separated by that semi-permeable membrane.

The example given in the Figures 24.2 serves to demonstrate and clarify this point. A beaker of water as shown in Figure 24.2 is divided through the center by a semi-permeable membrane. The black dotted line represents the semi-permeable membrane. We will define this semi-permeable membrane as lacking the capacity to diffuse anything other than the solvent, in this case water molecules.

# Fig.24.2: Reverse Osmosis

Now, when a little common table salt (NaCl) is added to the solution on one side of the membrane (Figure 24.2) the salt water solution has a lower chemical potential than the water solution on the other side of the membrane. In an effort to equilibrate the difference in chemical potential, water begins to diffuse through the membrane from the water side to the salt water side. This movement is osmosis. The pressure exerted by this mass transfer is known as osmotic pressure.

The diffusion of water will continue until one of two constraints is met. One constraint would be that the solutions essentially equilibrate, at least to the extent that the remaining difference in chemical potential is offset by the resistance or pressure loss of diffusion through the membrane. The other constraint is that the rising column of salt water exerts sufficient hydrostatic pressure to limit further diffusion. By observation then, we can measure the osmotic pressure of a solution by noting the point at which the head pressure impedes further diffusion.

**Reverse Osmosis**: By exerting a hydraulic pressure greater than the sum of the osmotic pressure difference and the pressure loss of diffusion through the membrane, we can cause water to diffuse in the opposite direction (Fig. 24.2), into the less concentrated solution. This is reverse osmosis. The greater the pressure applied, the more rapid the diffusion. Using reverse osmosis we are able to concentrate various solutes, either dissolved or dispersed, in a solution.

## 24.3.2. Nanofiltration

Nanofiltration is used to remove mainly the monovalent ions from whey. A partly demineralization and water removal is obtained. Nanofiltration is selected when Reverse Osmosis and Ultrafiltration are not the correct choice for separation. Nanofiltration can perform separation applications such as demineralization, color removal, and desalination. In concentration of organic solutes, suspended solids, and polyvalent ions, the permeate contains monovalent ions and low-molecular-weight organic solutions like alcohol.

Like Reverse Osmosis, the mass transfer mechanism in Nanofiltration is diffusion. Though generally quite similar in terms of membrane chemistry, the Nanofiltration membrane allows the diffusion of certain ionic solutes (such as sodium and chloride), predominantly monovalent ions, as well as water. Larger ionic species, including divalent and multivalent ions and more complex molecules are highly retained.

Since monovalent ions are diffusing through the Nanofiltration membrane along with the water, the osmotic pressure difference between the solutions on each side of the membrane is not as great and this typically results in somewhat lower operating pressure with Nanofiltration compared with Reverse Osmosis.

Some typical applications for Nanofiltration are:

- Desalination of food, dairy and beverage products or byproducts
- Partial Desalination of whey, UF permeate or retentate as required
- Desalination of dyes and optical brighteners
- Purification of spent clean-in-place (CIP) chemicals
- Color reduction or manipulation of food products
- Concentration of food, dairy and beverage products or byproducts
- Fermentation byproduct concentration.

#### 24.3.3. Ultrafiltration

Ultrafiltration is a selective fractionation process using pressures up to 145 psi (10 bars). Ultrafiltration is widely used in the fractionation of milk and whey, and in protein fractionation. The whey proteins are separated to form a product with 35, 60 or 80 % Whey Protein Concentrate. If ultra filtration is applied to skim milk, then Milk Protein Concentrate is obtained. It concentrates suspended solids and solutes of molecular weight greater than 1,000. The permeate has low-molecular-weight organic solutes and salts. The protein fractions are typically evaporated in multi-effect evaporators with either TVR or MVR recompression to save steam, before spray drying.

#### 24.3.4. Microfiltration

Microfiltration is a low-pressure cross-flow membrane process for separating colloidal and suspended particles in the range of 0.05-10 microns and as such used for bacteria removal, fermentation broth clarification and biomass clarification and recovery.

## 24.4. Application

Membrane filtration is today used in the Food & Dairy industry and likewise in other process plants. Membrane filtration can be used to meet very distinct liquid separations.

Table-24.1: Milk constituents and their sizes

No.	Particles Diame			
1	Fat globules (and bacteria)	500-10,000 nm		
2	Casein particles	10-300 nm		
3	Whey Protein	3-6 nm		
4	Lactose	$\sim 1 \text{ nm}$		
5	Salts	0.4-1 nm		
6	Water	~ 0.3 nm		

Cross-flow membrane filtration has opened the doors to a variety of new and innovative dairy products. Nowadays, the mechanical separator is not the only means of harvesting a component of milk. Today, not only the cream can be separated but virtually every major component of milk through membrane filtration can be separated. Membrane filtration technology has rapidly gained prominence in the processing of dairy ingredients. Microfiltration, Ultrafiltration, Nanofiltration and Reverse Osmosis, is making it possible to produce products with very unique properties and functionalities.

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## Module 11

# Dried milks: History and status in India and abroad

## Lesson-25

## 25.1. INTRODUCTION

Dry milk production has become an increasingly important segment of the dairy industry which is expected to grow further because of its features such as better keeping quality, less storage space, and lower transportation costs which result in attractive economics. Nonfat dry milk serves the same purpose for milk solids-not-fat that traditionally butter has done for milk fat. The ultimate aim of the industry is to obtain dry products which if recombined with water give little or no evidence of detrimental change compared to the original liquid product. Various names have been applied to the same dry milk product. For example, nonfat dry milk also has been called skim milk powder, dried skim milk, nonfat dry milk solids, and dehydrated skim milk. Dry whole milk, dry cream, dry buttermilk, and others frequently are called dried or powdered whole milk, etc., according to their identities.

#### 25.2. HISTORY OF DRIED MILKS

Dry milk has been known in some parts of the world for many centuries.

- § Marco Polo in the 13<sup>th</sup> century reported that soldiers of Kublai Khan carried a dried milk on excursions. The belief is that part of the fat was removed from the milk before drying and dehydration was accomplished by solar heating.
- § The first usable commercial production of dried milk was invented by the Russian chemist M. Dirchoff in 1832.
- § In 1855 T.S. Grimwade took a patent on a dried milk procedure, though William Newton had patented a vacuum drying process as early as 1837.
- § In 1909, Nicholas Appert, a Frenchman, developed dried milk in tablet form by air-drying of milk solids concentrated to a "dough" consistency.
- § During the last half of the 19th century, attempts to produce a dried milk involved the addition of other dry products to concentrated milk. Sugar, cereal products, and sodas, singly or in combinations, were added.
- § In 1850, Birdseye concentrated milk with added sugar until a solid was obtained.
- § A British patent was granted in 1855 to Grimwade who developed a modified dry product from highly concentrated milk to which sodium (or potassium) carbonate and sugar were added. This semi-solid material was extruded into thin streams and dried in trays.

- § Others who pioneered in methods of moisture removal from milk were Heine, Newton, Horsford, Dalson, Gallois and Deauve.
- § Patents and reports which emphasized processes for dry milk manufacture without the addition of other products began about 1898.
- § In 1901, Campbell of the United States and Wimmer of Denmark dried concentrated milk on trays.
- § In 1902, Hall obtained a patent on a new system of manufacturing dry condensed milk.
- § Today, powdered milk is usually made by spray drying nonfat skim milk, whole milk, buttermilk or whey. Alternatively, the milk can be dried by drum drying. Another process is freeze drying, which preserves many nutrients in milk, compared to drum drying.

# 25.3. OBJECTIVES AND REQUIREMENTS OF MILK POWDERS

- 1. The main purpose of the manufacture of milk powder is to convert the liquid perishable raw material to a product that can be stored without substantial loss of quality, preferably for some years. Decrease in quality mainly concerns formation of gluey and tallowy flavors due to Maillard reactions and autoxidation, respectively and decreasing nutritive value especially decrease in available lysine. If the water content becomes very high and the storage temperature is high, caking due to lactose crystallization and enzymic and even microbial deterioration can occur. However, such problems can be avoided.
- 2. The powder should be easy to handle. It should not dust too much or be overly voluminous. It should be free-flowing, i.e., flow readily from an opening, and not stick to the walls of vessels and machinery. The latter requirement is especially important for powder used in coffee machines, etc.
- 3. After adding water the powder should be reconstituted completely and readily to a homogeneous mixture, similar in composition to the original product. Complete reconstitution means that no undissolved pieces or flakes are left and that neither butter grains nor oil droplets appear on top of the solution. 'Readily reconstituted' means that during mixing of powder and water no lumps are formed, because these are hard to dissolve. In the ideal situation the powder will disperse rapidly when scattered on cold water; this is called *instant powder*. Special processing steps are needed to achieve this property.
- 4. According to its intended use, the reconstituted product should meet specific requirements. If the use is beverage milk, the absence of a cooked flavor is of importance. If the powder is to be used for cheese making, the milk should have good clotting properties. If used to make recombined evaporated milk, satisfactory heat stability is necessary. So there are several widely divergent requirements that cannot be reconciled in one powder. For instance, it is not possible to make whole milk powder that has no cooked flavor and at the same time develops no oxidized flavor during storage. With respect to the intensity of the heat treatment, milk powders are classified as low-, medium-, or high-heat.
- 5. The product must be free of health hazards, be it toxic substances or pathogenic organisms. Besides general hygienic measures and checks prevailing in the dairy industry, there are some specific considerations.

The approximate *composition* of some types of powder is given in Table 25.1. There are other kinds of powders also. All of these products have specific requirements. Because the composition of the raw material varies, the composition of powders also varies. Accordingly, one has to tolerate a certain margin. This offers the possibility for adulteration; for instance, buttermilk powder or whey powder can be added to (skim) milk powder. The presence of a foreign powder can mostly be detected microscopically, but admixture of a small percentage of another liquid before the drying generally cannot so easily be established. Because whey is cheaper than skim milk, this kind of 'adulteration' sometimes occurs.

Table-25.1: Approximate Composition (% w/w) of Some Types of Powder

No.	Constituents	Powder Form							
		Whole Milk	Skim Milk	Whey	Sweet Cream				
					Butter Milk				
1	Fat	26	1	1	5				
2	Lactose	38	51	72-74	46				
3	Casein	19.5	27	0.6	26				
4	Other Proteins	5.3	6.6	8.5	8				
5	Ash	6.3	8.5	8	8				
6	Lactic Acid	-	-	0.2 - 2	-				
7	Water	2.5	3	3	3				

## 25.1. USES

M ilk Powder is a manufactured dairy product made by evaporating milk to dryness. An array of milk powders is now available. They are shown in Table 25.2 below:

Table-25.2: Different types of milk powders available in market

<ul> <li>Nonfat dry milk</li> </ul>	<ul> <li>Dry skim milk vegetable fat products</li> </ul>
<ul> <li>Dry whole milk</li> </ul>	<ul> <li>Malted milk Powder</li> </ul>
<ul> <li>Dry buttermilk</li> </ul>	• Cheese powders (Cheddar, Blue, etc.)
<ul><li>Dry whey</li></ul>	<ul> <li>Sweetened chocolate flavored non fat dry milk</li> </ul>
<ul> <li>Dry ice cream mix</li> </ul>	<ul> <li>Coffee creaming products</li> </ul>
<ul> <li>Dry creams (sweet or sour)</li> </ul>	<ul> <li>Sodium or Calcium Caseinates</li> </ul>
<ul> <li>Dry high acid milks</li> </ul>	<ul> <li>Partly skim milk powder</li> </ul>
<ul> <li>Infant milk food</li> </ul>	<ul> <li>Lactose powder</li> </ul>
<ul> <li>De-lactose milk powder</li> </ul>	

§ Miscellaneous products that have been processed experimentally or commercially in small amounts are: dry creamed cottage cheese, dry butter, and dry yogurt. Modified equipment and special methods may be required for their successful manufacture.

- § Milk Powder is frequently used in the manufacture of infant formula, confectionery such as chocolate and caramel candy, and in recipes for baked goods where adding liquid milk would render the product too thin.
- § Powdered milk is also widely used in various sweets.
- § Milk Powder is also a common item in UN food aid supplies, fallout shelters, warehouses, and wherever fresh milk is not available.
- § It is widely used in many developing countries because of reduced transport and storage costs (reduced bulk and weight, no refrigerated vehicles). As with other dry foods, it is considered nonperishable, and is favored by survivalists, hikers, and others who require nonperishable, easy-to-prepare food.

# 25.1. DEVELOPMENT OF DRYING EQUIPMENT

- § **Drum / Roller Drier**: Just (1902) was among the first inventors to receive patent rights on a drum drier with two rolls. Hatmaker of England improved Just's model. Vacuum drum driers were designed by Ekenbery, Sweeden 1839; Passburg (1903), Germany; and Govers (1909).
- § **Spray Drier**: Among the early inventors of spray drying equipment was Percy, who in 1872 combined atomization of a fluid and heated air. Stauf, a German, received a US patent on an improved design based on this principle in 1901.

## 25.2. IMPROVEMENT OF RECONSTITUTABILITY / AGGLOMERATION

As a result of pioneering research to improve the reconstitutability of nonfat dry milk in water, Peebles was issued patents in 1936, 1955, and 1958. In the Peebles method, regular spray dried milks (usually nonfat dry milk) are reprocessed by rewetting the surface of the particles in turbulent air which causes the wetted particles to collide, forming clusters. In the next stage, the moisture content is reduced with hot air. Products treated by this system became known as an "instantized" product. Instantized nonfat dry milk from the Peebles process appeared for general distribution on the retail markets in 1954.

## 25.3. FOAM SPRAY SYSTEM

The US Department of Agriculture's foam spray drying technique is an important contribution to the industry. Besides providing the advantage of one step processing, the method is more satisfactory for products sensitive to heat damage during dehydration. Foam spray dried dairy products have very good dispersibility, but poor sinkability.

## 25.4. NUTRITIONAL VALUE

- § Milk powders contain all standard amino acids the building blocks of proteins and are high in soluble vitamins and minerals.
- § According to USAID the typical average amounts of major nutrients in the un-reconstituted milk powder are: 36% protein, 52% carbohydrates (predominantly lactose), 1.3% calcium,

- 1.8% potassium (by weight). Storage of powder at high relative humidity and high ambient temperature can significantly degrade the nutritive value of milk powder.
- $\S$  Commercial milk powders are reported to contain oxysterols (oxidized cholesterol) in higher amounts than in fresh milk (up to  $30\mu g/g$ , versus trace amounts in fresh milk). The oxysterol free radicals have been suspected of being initiators of atherosclerotic plaques.
- § Adulteration can occur in powders, e.g. melamine adulterant in infant formula, added to show higher protein content.

## 25.9 WORLD MARKET

Milk powders are next to ghee and butter in market size. Some years back milk powders had a larger market size than that for butter. Due to relatively slow growth, the market for milk powders has lagged behind. However, it has shown a consistent positive growth on a year to year basis.

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# **Module 12**

# Grading and quality of raw milk for dried milks

## Lesson-26

## **26.1. INTRODUCTION**

The grading and quality of milk for dried milks is based on the bacterial quality of original milk, acidity of the milk and properties of milk concentrate used for drying which affect the final dried product.

# 26.2. BACTERIAL QUALITY OF THE ORIGINAL MILK

In deep-cooled milk, psychrotrophic Gram-negative rods can develop during prolonged storage (e.g., *Pseudomonas* spp.). These bacteria do not survive even a mild heat treatment. Proteinases and lipases formed by these rods may survive and become incorporated into the powder. Prevention of the growth of these bacteria is possible using refrigeration, limiting the storage time, and thermalization process. Contamination and growth during storage of the thermalized milk should be avoided.

Heat-resistant bacteria and bacterial spores are of great importance. They survive low pasteurization (72  $^{0}$ C for 15 s), and most are not killed during evaporation and drying. Due to concentrating, the powder contains about ten times as many bacteria per gram as the milk immediately after preheating. A more intense pasteurization will kill the heat-resistant cocci (e.g., *Enterococcus faecalis*, *S. thermophilus*) and in a high-quality medium-heat or high-heat milk powder only bacterial spores and *Microbacterium lacticum* can originate from the original milk.

Among the aerobic and anaerobic spore-forming bacteria, Bacillus cereus and Clostridium perfringens are especially important to the powder quality. If the reconstituted milk is to be used for cheese making, a very low count of gas forming anaerobic spore formers (C. tyrobutyricum and C. butyricum) may be essential. All of these bacteria are likely to originate mainly from contamination during milking (dung, soil, and dust). A low count of anaerobic spore formers points to a good-quality silage. But the pathogenic C. perfringens C. perfringens C. perfringens . Likewise, the total count of aerobic spore formers is not always an indication of the spore number of B. cereus. This probably is because (1) contamination with B. cereus is heavier on pasture, and (2) at higher environmental temperatures B. cereus can develop and sporulate in imperfectly cleaned and disinfected equipment outside the operating periods. To kill bacterial spores, heat treatment at 90 to  $110^{\circ}$  C for 10 to 20 s is insufficient; hence, UHT treatment should be used. usually does not originate from the silage, though it may from the dung. Hence, a low count of anaerobic spore formers need not be an indication of the absence of

#### 26.3. NEUTRALIZATION OF ACIDITY OF MILK

Fresh, sweet milk contains no lactic acid although mixed milk may have a titratable acidity ranging from 0.10 to 0.15% due to milk proteins, carbon dioxide, citrates, and phosphates. Neutralization refers to the practice of reducing the acidity of the fluid products. If skim milk has become acid, neutralization slightly decreases the titratable acidity of the reconstituted product.

In countries where neutralization of milk is necessary, and for the neutralization of a high acid product, such as whey, food grade alkalis are used. The acidity in these cases should be reduced to a pH of 6.8 (range of 6.6 to 7.0). The specific titratable acidity in this pH is dependent upon the composition of the product and type of alkali used.

Temperature used for neutralization should be 35  $^{0}$ C or less and at least 15 min is allowed for the reaction before heating. The selection of alkali may be influenced by intended use of the dry product and the processing method. Sodium bicarbonate causes foaming on a drum so should not be used for the drum drying. A sodium type such as sodium hydroxide is preferred for products requiring a maximum solubility. Care should be exercised to minimize the development of defects in the milk; otherwise adverse effects of the chemical may result.

# 26.4. PROPERTIES OF MILK CONCENTRATE FOR DRYING

## 26.4.1. Concentrate

It is essential to evaporate milk correctly to achieve the required feed characteristics. Inappropriate evaporation can cause increased viscosity and product instability that may make further processes such as drying more difficult or even damage the product irreversibly. Many functional powder properties, such as solubility, heat stability and the WPNI index are set at the evaporation stage. Some properties such as the denaturization of whey proteins, and colour and flavour development, are irreversible.

The correct properties are achieved by managing all the heating and handling processes within the evaporator plant to ensure the desired result. These properties can include: low heat powder for minimum product damage, medium heat powder (e.g. for baking applications), high heat (heat stable) powder with the correct properties to accept UHT treatment after reconstitution, and whole milk powder with the desired WPNI levels for easy reconstitution. Also, air in the concentrate should be avoided by any means, as it upsets the whole concentrate pre-treatment and drying.

## **26.4.3.** Solubility

Measurement of Insolubility Index is usually performed on milk powder. If it is not possible to trace the problems with too high Insolubility Index by changing the drying parameters, it is recommended to perform the test on the concentrate. The amount of concentrate to be used is calculated as follows:

g concentrate = g powder x 100 / %TS of concentrate

g powder = 10 g skim milk, 13 g whole milk, or 6 g whey powder. The rest of the procedure is as described for powder.

## **26.4.4. Viscosity**

- § The concentrate leaving the last effect of the evaporator is liquid. The concentrate may however have different viscosity depending upon the composition, heat sensitivity of the proteins, pretreatment, temperature and solids content.
- § Whole milk concentrates are generally less viscous than skim milk concentrates, and as a general rule the viscosity should not exceed 60 and 100 cP, respectively, if the atomization should be optimal. Higher viscosities can be handled in the dryer, but may loose the capacity by bad atomization or big droplets and an inferior product will be the result.
- § The composition of the concentrate will influence the viscosity, especially on the protein (P) content in relation to the lactose (L) content. When the ratio P:L is high, the concentrate will get a high viscosity. The ratio P:L can be adjusted by adding lactose. As a general rule, a higher fat and lactose content will give lower viscosity. Higher protein content will give higher viscosity.
- § When milk is exposed to a high heat treatment, especially in indirect pasteurizing systems, prior to the evaporation, the viscosity of the concentrate will be higher.
- § The concentrate temperature will naturally have a direct influence on the viscosity and higher temperature means lower viscosity.
- § The solids content of the concentrate will have a very significant influence on the viscosity, and the higher the concentration, the higher the viscosity.
- § However, above parameters shows the direct influence on the viscosity. One of the main influences on the viscosity is the time, i.e. the viscosity is a function of time, also known as age thickening. This means that the viscosity will increase if the concentrate is left for some time. The increase is depending on composition, mainly proteins binding to each other, temperature and concentration. The age-thickening is only partly reversible by agitation.
- § A temperature increase will result in a viscosity drop; but as the age thickening is more pronounced at higher temperatures, the viscosity will soon increase to the same level and further on as the time passes.

The quality of the concentrate for spray drying should comply with the following:

## 26.4.5. Skim milk

Skim milk having 48-50% TS should have maximum viscosity of 100 cP at 40°C. For manufacture of instant products, the WPNI should be 2.5-3.5 mg and for high bulk density powders, the WPNI should be max. 1.0 mg. It should not show any measurable amount of scorched particles as well as solubility index and Sieving test should show no visible insoluble particles.

#### **26.4.6.** Whole Milk

Whole milk having 48-50% TS should have maximum viscosity of 60 cP at 40°C. For manufacture of instant products, the WPNI should be 2.5-3.5 mg and for high bulk density powders, the WPNI should be max. 1.0 mg. It should not show any measurable amount of scorched particles as well as solubility index and Sieving test should show no visible insoluble particles.

## 26.4.7. Whey

Whey having 52% TS should have maximum viscosity of 100 cP at 40°C. It should have >75% crystallized lactose with mean crystal size ranging from 30-50  $\mu$ . It should have maximum 25% denatured whey proteins and should not show any measurable amount of scorched particles as well as solubility index and Sieving test should show no visible insoluble particles.

# 26.5. EFFECT OF PRE-CONCENTRATION OF MILK ON THE QUALITY OF THE RESULTANT POWDER

The chemical and physical properties of a concentrate with high total solids affect essentially the properties of the resultant powder.

§ The major effect of concentration is on the viscosity of the milk. The results depicted in Table 26.1 demonstrate the increase in viscosity of skim milk when concentrated. Although viscosity increased with concentration, the effect was not linear, was most marked when the concentration of milk solids exceeded 45%. The large viscosity increases above 45% TS are due to the concentration of casein micelles occupying over 30% of the total volume of the milk.

§ Table 26.2 shows stages of product concentration in the manufacture of milk powders. It is evident from the table that with increase in concentration sensitivity to heat denaturation of protein increases which in turn influences the quality of milk powder, especially the solubility.

Tables 26.3, 26.4 and 26.5 demonstrate influence of the dry matter content of the concentrate on the various properties of skim and whole milk powders.

Table- 26.1: The effect of concentration on the viscosity of skim milk

Total solids (%)	Viscosity (cP)
18.6	4.4
23.8	5.6
29.3	8.8
32.3	16.6
39.8	59.3
46.4	1280.0

 $Table \hbox{-} 26.2 \hbox{: Stages of product concentration in the manufacture of milk powders} \\$ 

TS (%)	Water (%)	pН	Viscosity (cp)	Sensitivity to heat	Remark
13	87	6.7	1	Low	Liquid
26	74	6.6	10	Moderate	Concentrate for Roller Drying
40	60	6.1-6.3		High	Concentrate for Spray
45	55		100 to	_	drying
50	50		10000		
80	20	solid	solid	Very high	Critical solids for Spray Drying
88	12	solid	solid	Highest	Maximum sensitivity for insolubilization
94	6	solid	solid	High	Straight through process
97	3	solid	solid	Low	Final moisture

Table-26.3: Relation between the degree of preconcentration and physical properties of  $\overline{\text{SMP}}$ 

No.	Properties		% TS	in concenti	rate
	_	32.1	37.3	42.6	47.6
1	Specific Vol. (ml/100g)	196	156	135	121
2	Entrapped air (% of particle vol.)	33	29	26	19
3	Particle size	22.2	23.9	24.4	24.4
4	Flowability (angle degree)	55.0	50.7	45.2	43.8
	Solubility Index				
5	Immediately after production	0.03	0.05	0.09	0.32
6	After storage for 12 m (30°C)	0.07	0.08	0.09	0.32
7	Viscosity of concentrate (cP)	35	125	328	1761

Table-26.4: Relation between the degree of preconcentration and the physical properties of WMP

No.	Properties	% T	% TS in concentrate			
		42.8	45.5	48.8		
1	Viscosity of concentrate (cP)	145	558	5097		
2	Specific Vol. (ml/100g)	185	170	162		
3	Entrapped air (% of particle vol.)	21	19	18		
	Solubility Index					
4	Immediately after production	0.13	0.13	0.13		
5	After storage for 12 m (30°C)	0.16	0.27	0.34		
6	Extractable fat (% of Total Fat)	5.8	5.5	5.1		

Table-26.5: Influence of the dry matter content of the concentrate on various characteristics of whole milk powder (Concentrate – Non-homogenized and Concentrate – Two-stage homogenized)

Product	Characteristics		Co	ncentra	te –		Concentrate –					
		Non-homogenized					Two-stage homogenized					
Concentrate	Dry Matter, %	42.3	45.6	50.7	52.6	55.4	37.9	42.7	47.0	51.5	52.7	
	Viscosity cp	20	31	71	101	197	22	25	126	348	643	
	Degree of homogenization, %	18	21	25	30	32	85	84	82	76	77	
Powder	Moisture Content, %	3.38	3.31	3.48	3.64	4.37	3.40	3.46	3.53	3.66	3.80	
	Viscosity, cp	890	1200	2570	2900	5350	1290	1190	1780	3600	4160	
	ADMI Solubility Index (ml)	0.05	0.05	0.42	0.56	2.06	0.05	0.05	0.10	0.55	0.60	
	Degree of homogenization, %	38	45	64	73	76	88	80	77	77	76	
	Free-fat content, %	24.0	20.5	10.3	7.5	6.1	11.0	7.9	4.5	4.1	4.7	
	Bulk Density, g/cm <sup>3</sup>	0.54	0.56	0.61	0.66	0.68	0.51	0.54	0.57	0.59	0.61	
	$N_2$ penetration $ cm^3/100  g$	8.79	7.38	4.38	2.70	2.46	8.65	5.41	3.49	3.65	3.24	
	Mean particle density	0.94	0.98	1.04	1.08	1.10	0.91	0.95	0.98	1.00	1.02	
	Vacuole volume cm <sup>3</sup> /100 g	28.1	24.6	18.8	14.7	13.2	32.1	28.4	24.0	21.8	20.3	
	Y 10 Permeability factor	31.2	29.9	23.3	18.2	18.5	26.9	19.0	14.4	16.6	15.9	

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## Module 13

# **Technology of dried milks**

## Lesson-27

# **Drum or Roller Drying**

## 27.1. INTRODUCTION

Milk and skim milk are dried commercially by the spray, drum or foam spray method. Today most milk and skim milk are dried by the spray method. In the past, the drum dried dairy products constituted a high percentage of the total amount produced. But since World War II decreased economic advantage of processing by drum compared to the spray process and its characteristic scorched flavour and poor solubility due to the severe heat treatment have drastically reduced the volume of drum dried milks. Now only a relatively small amount is manufactured by the drum process.

## 27.2. DRUM OR ROLLER DRYING

Milk or milk products can be dried in a thin film ( $\sim 0.1$  mm) on an internally steam heated rotating drum. Often, two drums are set up side by side at a very small distance apart. The water evaporates within a few seconds, which is possible due to the high drying temperature ( $>100^{0}$ C). On a drum drier, the product enters as a liquid and leaves as a solid. The product is removed from the exterior of the drum by a "doctor" blade. The dried film is scraped off from the drum by means of a steel knife, collected, and ground.

The process is usually called drum drying, or in the dairy industry, more often called "Roller Drying." The process is also known as "Film Drying". Drum drying requires less space and is more economical than the spray drier for the small volume.

The major disadvantage of drum drying is that the dry product may have a scorched flavor and solubility is much lower because of protein denaturation. Considerable product damage due to heating occurs, mainly because scraping off is always imperfect and, accordingly, a part of the milk is repeatedly wetted and dried. The quality of the powder can be improved by using a vacuum roller drier, in which the milk is dried at a lower temperature, but this method is expensive. Nowadays the roller drying process is little used.

# **27.3.** Classification of Drum Dryers:

Drum dryers may be classified as shown in Table-27.1.

Table-27.1: Classification of Drum Dryers

1	Number of hollow drums	(a)	Single drum
		(b)	Twin drum, or
		(c)	Double drum units
2	Pressure surrounding the product	(a)	Atmospheric
		(b)	Vacuum
3	Direction of turning of a twin or double drum unit	(a)	The drums may turn up at the center and away at the top as in a twin drum unit, or
		(b)	The drums may turn at the center and together at the top, as in a double drum unit
4	Method of placing product on the	(a)	Trough or reservoir above for top feed
	surface of drum	(b)	Spray or splash feed
		(c)	Sump below for dip feeding, or
		(d)	Trough below for pan feeding
5	Method of obtaining vacuum for	(a)	Steam ejector, or
	a vacuum drum drier unit	(b)	Vacuum pump
6	Material of construction	(a)	Steel
		(b)	Alloy steel
		(c)	Stainless steel
		(d)	Cast iron
<u> </u>		(e)	Chrome, or nickel plated steel

Generally cast iron is usually used. The wear is excessive on stainless steel drums. The metal used for the knife should be softer than the drum. The double drum atmospheric drier is most commonly used in the dairy industry. Vacuum drum driers are essentially the same as, atmospheric units except that the drums are enclosed so that a vacuum can be pulled on the product during drying. The single drum with top feed is more commonly used for vacuum. A thicker film is obtained with the top feed.

#### 27.4. Flow of Product

The product may be placed in its natural form or condensed in a vacuum pan or evaporator before it is fed to the drum drier. Milk is usually pre-condensed for single drum units. The product is usually preheated and placed in a reservoir between the upper portions of the drums. Other devices may be used to provide thin film over the turning drums. The doctor blade, a sharp hard flexible knife, scrapes the dried material from the drum. The blade sits at an angle of 15 to 30° with the surface. The film of dry milk forms a continuous sheet from the knife to the auger trough which is about level with the bottom of the drum. The auger for each drum discharges the product into elevators, then to the hammer mill which pulverizes the product, after which it may be sized. After sizing, the dried product is packaged and stored, or moved to market. These preceding steps are carried out in one continuous operation. In some vacuum units, the product is accumulated in a tub or tray and removed

periodically first from one side of an enclosed chamber (separate from the drying chamber) to the other side, while maintaining a vacuum on the drying unit.

Water vapor above the drier has a lower density than the air surrounding the unit, and will rise. A hood must be placed over the drums with a stack for vapour to move out of the area.

# **27.5. Drums**

The atmospheric double drum drier is most commonly used for milk drying.

- § **Vacuum:** The vacuum drum drier is used for foods and milk. The inside of the drum is heated with steam with a low pressure. For vacuum drum drying, the vacuum between the drum and the housing surrounding the drum is maintained at 68.5 to 73.6 cm Hg vacuum. The product temperature approaches the temperature of the steam heating medium.
- § Size & Speed: The drums used for a drier are 61 to 122 cm in diameter, and up to 3.6 m in length. Drums must be carefully machined, inside and outside, otherwise a difference in thickness will alter, heat transfer and drying will not be uniform. The speed of the drums is adjustable usually being from 6 to 24 rpm; however, the range of speed may be from 1 to 36 rpm. Speed of the drums is important as it affects (a) the thickness of film, and (b) the time the product is on the roll. The speed of the drums may be varied according to the concentration of the product and the dryness desired.
- § **Product Contact:** The product is removed after  $3/4^{th}$  to  $7/8^{th}$  of a revolution of the drum has taken place. The product is in contact with the drum for about 3 s or less.
- § **Mounting:** One drum of a double drum drier is mounted on a stationary bearing; the other drum is mounted on a bearing which can be moved to provide the desired clearance between drums.
- § **Spacing:** The spacing between drums of a double cylinder drier is about 0.05 to 0.1 cm when the drums are cold. The level of milk between the drums affects the capacity of the unit and is designated by the distance above the centerline of the drums. The product is contained over the drums with end plates.
- § Uniform Film Feeding: A single drum drier may be equipped with upto five spreader rolls to increase the uniformity of thickness of the film on the drier. The concentrated product is fed onto the drum next to the spreader rolls. As the drum turns, a thin film is formed, and an additional film then forms on the next spreader roll.

## 27.6. Moisture Removal

Moisture is removed by circulation of air which carries away the moisture above the unit. A hood above the unit directs the water vapors from the drier. A product entering the reservoir above a double drum with 9% solids would leave at about 12 to 16 % total solids. A hood should be placed over, the sump or reservoir which is between and above the drums. Adequate airflow must move over the surface of the drums to carry away moisture. An excessive amount of airflow should be avoided since this causes low efficiency due to rapid cooling of the drum surfaces and of the product. In a vacuum drum drier, moisture must be removed from the chamber in much the same procedure as done in the evaporators.

#### 27.7. Steam Flow

Steam is fed into the center of the drum at one end of the shaft through the hub. Steam pressures up to 6.3 kg/cm<sup>2</sup> are utilized with dry saturated steam up to 149 <sup>0</sup>C. Superheated steam should be avoided because as heat is removed from the steam, the vapor condenses to a liquid. The liquid condensate moves to the bottom of the drum and must be removed by a pump or siphon. Flooding of the inside of the drum with the condensate reduces the rate of heat transfer.

# 27.8. Factors Affecting Production from a Drum Drier

- (1) **Milk Feed Temperature**: As the milk feed temperature is increased, the rate of drying increased proportionally at about 2.2 % for each 5.5  $^{0}$ C increase in feed temperature with little increase above 71  $^{0}$ C.
- (2) **Height of Milk**: Increasing the height of milk over the drum from 25 to 30 cm gives an increase in drying rate of 10%.
- (3) **Drum Gap**: The roller gap should be adjusted between 0.05 and 1.09 cm. If the drums are farther apart, leakage will occur. The thickness of the film on the drum is directly related to the distance between the two drums.
- (4) **Drum Speed**: Only a slight increase in drying rate occurs when the drum speed is increased. As the speed is increased, the film becomes thinner so that the production, both in kilograms of water evaporated and kilograms of dry product produced remain approximately the same with only slight increase in drying rate as the speed is increased.
- (5) **Steam Pressure**: With an increase in steam pressure, the temperature is increased and thereby the rate of drying increased. Too high a steam pressure or super heated steam must be avoided because scorching of the film will result. Increasing the steam pressure from 3.87 to 4.57 kg/cm<sup>2</sup> (150 to 155  $^{0}$ C) increases the production of dry product by approximately 10%.

The product may be damaged and scorched if there is uneven milk supply, incomplete removal of film, imperfect roller alignment, rough roller, too high a temperature of the product caused by too high a steam temperature or too slow a drum speed.

## 27.9. Operation and Maintenance

Several items must be checked periodically to assure maintenance of quality of the product at sufficient, rated capacities or output. Following factors must be considered.

- (1) Drums must be properly aligned, particularly for double drum or twin drum units and have identical characteristics of speed, heat transfer, wear, etc.
- (2) Knives must be reground regularly (~ every 100 hr) and be of uniform sharpness.
- (3) Knives must be flexible, machined on both edges, have uniform thickness and be easily adjusted. Uniform knife pressure against the drum must be maintained. Excessive pressure of blade on the drum increases the energy to operate the drum and danger of metallic shavings in the product.

- (4) Drum surface must be kept smooth. It may be necessary to resurface the drums after 1000 to 3000 hr of operation. The drum should receive a film of oil or paraffin wax when not used regularly to prevent rust.
- (5) Condensate must be promptly removed from inside the drum.
- (6) Drum must be vented of air to assure that all of the interior heat transfer surface of the drum can be utilized for steam.
- (7) Processing operations before drum drying consist primarily of removal of moisture, normally 2:1 concentration to 16 to 18% solids, in an evaporator or vacuum pan. Clarification helps to some extent. Homogenization before drying on a drum does not affect the rate of drying, but will help quality for whole milk.
- (8) Starting the drier is accomplished by
- (a) Lifting the knives,
- (b) Starting the drums
- (c) Turning on the steam, and
- (d) Placing the product on the drums.

The very first powder is not of as high a quality as the remainder because of the tendency to be either over or under dried. The drums should be set in motion when the heat is applied by gradually turning on the steam to prevent warping.

- (9) Preheating of product is desirable, especially if intended for bakery use.
- (10) End plates or end drums, which are spring loaded and contain the product above double drums, must be properly adjusted to (a) prevent leakage and (b) accumulation of solids.
- (11) The level of milk in the reservoir must be uniform and holes kept open in the distribution tubing. Change in the milk level affects the film thickness on the drum and thus the steam requirements.
- (12) High moisture in the product is due to low temperature, thick film, high total solids, and fast speed (rpm.)

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# Lesson-28

# Freeze, Vacuum and Foam Drying

## 28.1. INTRODUCTION

The processes other than heat evaporation of milk for manufacture of dried milks are also in practice for some specific purposes. They are freeze, vaccum and foam drying.

# 28.2. Freeze drying

The process consists of freezing the product, supplying heat so moisture is removed without passing through the liquid phase, or as is usual, by maintaining a vacuum in the vaporizing chamber. The vapours are removed before they reach the vacuum pump.

#### **28.3.** Method

A batch processing or a continuous operation in a high-vacuum belt drier can be applied. Freezing can be done in a separate or the same chamber in which drying is done. Freezing is done to -28.8 °C or lower. Multiple chambers are used for commercial operations with the complete drying process in each chamber. A vacuum approaching 2.54 cm Hg absolute is used. The air and vapor are passed over a refrigerated coil to condense the vapor. The quantity of heat supplied must be limited to avoid damage to the product particularly as the conduction properties change with drying. Electric heating platens, which can be decreased in temperature as drying progresses, are usually used for batch processes. Rapid freezing is desirable to provide formation of small ice crystals. Small crystals provide least change in the properties of the product and a product which will reconstitute easily. Low temperature processing is desired to provide a product with minimum change. A thin layer of the liquid is frozen, whereupon the ice is sublimated under a high vacuum. The heat or sublimation must be provided to vaporize the ice. The heat of vaporization is equal to the sum of the latent heat of fusion, sensible heat of liquid, and latent heat of evaporation. Moving heat to the product in the vacuum chamber is a major problem. Heating can be done by radiation, conduction, or convection. A voluminous cake is left (the space of the ice crystals is now occupied by holes) and is subsequently ground.

## 28.3.1. Advantages

- 1. Damage due to heating does not occur but that is also true of spray drying if skillfully performed.
- 2. Suitable for processing in small quantities and is applied to lactic starters, etc.

## 28.3.2. Disadvantages:

1. The method is expensive.

2. The fat globules are subject to partial coalescence; this causes freeze-dried whole milk powder to show segregation after its reconstitution. Freeze drying costs about 5 to 10 times the conventional drying for foods.

## 28.3. VACUUM DRYING

One method of vacuum drying is to meter nitrogen into concentrated whole milk after homogenization. The product is then cooled to 1.7  $^{0}$ C in a scraped surface heat exchanger. The product is placed on a solid stainless steel horizontal belt 30.5 cm wide. It goes through first a heating drum, then a cooling drum, each 61 cm diameter for heating and cooling. Heat is supplied by 19 banks of 2 KW radiant heaters. A vacuum of about 50 mm Hg absolute is supplied. Scraper blades remove the product from the belt.

#### 28.4. FOAM DRYING

Foam spray drying, is new and has caused interest in new products. Common dairy products, skim milk, whole milk, buttermilk, sweet and sour cream (up to 3:1 fat to SNF) whey and emulsified cheese slurry can be foam spray dried. Air is commonly used as the added gas for making foam spray nonfat dry milk. Nitrogen is commonly used for making foam spray dried whole milk.

The product to be dried is first stabilized. Air is fed to the product in a closed mixer where a porous product is formed. The air-product mixture is extruded onto perforated drying trays. The tray of foam passes over air jets catering the foam to increase drying surface. The trays then move into and upward in the drying chamber in the same direction as the air moves. Air at 104.4 °C with a velocity of 111 to 203 cm/s is used, traveling co-currently with the tray. At the top section of the drier, after the warm drying air is emitted, cool air enters to cool the product. The dry product is produced in 1.5 to 15 min, is porous and readily soluble.

A foam film up to 40 mm thick is placed on a stainless steel belt. The product is exposed for a minute and temperature does not exceed 76.6  $^{\circ}$ C. The system can produce 5 to 7.5 kg of dry product per sq.m of belt.

## 28.4.1. Advantages:

- 1. Using most conventional spray drying equipment for drying liquids up to a maximum of 60% total solids as compared to 50% on a particular drier.
- 2. The powder quality can be excellent due to the low drying temperature applied.
- 3. It can be applied to inhomogeneous products.
- 4. Drying special products, such as malted-milk and cottage cheese whey. But difficulty can be encountered because of sticking to the drier. A product of low density, excellent flow properties, highly hygroscopic, and rapid solubility is obtained using this method.
- 5. By holding the product, a product with more crystalline lactose is obtained. Holding 52% TS condensed nonfat milk at 35°C for 15 min provides 7.7% crystalline lactose in the powder; for 35 min, 17.1%, for 60 min, 35%. Conventional agglomerated dry milk has from

- 5 to 25% crystalline lactose. The greater the percentage of crystallized lactose, the less hygroscopic is the dry product.
- 6. Obtaining an instant type powder, but with characteristics different than the product prepared by agglomerating processes normally used following the spray direr.
- 7. Provides a procedure for increasing the capacity of conventional equipment. By removing the water ahead of the drier, the cost of operation can be decreased. Approximately twice as much product can be handled by the drier with a feed of 60% TS as compared to 42.5%. The capacity of a conventional drier can be increased appreciably by using higher total solids in the input.
- 8. Foam spray nonfat dry milk has a bulk density of about 0.35 gm. per ml. or less which is about the same as agglomerated or conventional instantized powder.
- 9. The product can withstand considerable handling and can be compressed into smaller volumes without appreciably affecting the dispersibility.
- 10. A more uniform particle size is obtained with foam spray drying.
- 11. Good dispersibility during reconstitution enhances its prospects for home use.

#### 28.4.2. Limitations:

- (a) The product on reconstitution remains on top of water for an extended period and needs to be mechanically mixed to form a solution.
- (b) During reconstitution, considerable foam is formed.
- (c) Providing a liquid milk product at 60 % TS requires special consideration for heat exchangers and evaporation. In general, larger heat exchanger surfaces or continuous forced flow of the product through the heat exchanger is required.
- (d) As the holding time increases, the viscosity increases and makes handling difficult and dispersibility decreases.

The product acceptance may be slow for industrial uses because of the low bulk density, hence higher packaging costs.

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# Lesson-29

## SPRAY DRYING

## 29.1. INTRODUCTION

The spray dryer design and technology provide the powder producer with the ability to control the powder form through the spray drying process. The spray dryers are designed to produce properties such as particle size, bulk density, moisture content, solubility, dispersibility etc. exactly as desired by the user. The products are applicable to all pumpable products, whether heat or non-heat sensitive.

#### 29.2. SPRAY DRIER

Figure 29.1. gives one, considerably oversimplified, example of a drier layout. Actually a wide range of configurations is applied, according to type of raw material used, product specifications, and local possibilities (e.g. resources). Naturally, minimization of operating expenses is desired; this is not simple when the drier is to be used for a range of different products.

As the atomized product is introduced into the drying chamber, heated air is forced through the chamber. The air furnishes heat for the evaporation of the moisture and the air is a carrier for moisture to be removed from the drier. The air may be forced through the drier by either a pressure or suction system. The product and air must be separated after drying. The product is then cooled and packaged. Controls maintain the proper adjustment of the variables involved in drying. Additional operations may be utilized to provide a product that will dissolve rapidly. The capacity of commercial driers may vary from 180 to 4000 kg per hr and higher of dried product. Proper management of a drier operation is important to provide good quality solid products from good quality liquid products.

**Fig. 29.1:** Simplified diagram of the spray-drying process.

## 29.3. OBJECTIVES

- § To remove the moisture
- § To reduce the cost of transportation
- § To improve storage of the product, and
- § To provide a product fit for many food manufacturing operations.

## 29.4. ESSENTIAL PROCESSES

The use of the spray drier has increased in recent years and it is the most important method of drying liquids to solids for milk and milk products. The spray drier is flexible and can be used

for many different food products. The system, incorporating various components of equipment, is complicated, even though the principle of operation is simple. There are several variants, but the following are essential process steps that are always involved:

## 29.4.1. Heating of the air

Generally heat exchangers are used. The classical medium is steam, but a pressure over 9 bars, hence a temperature over  $175\,^{0}$  C, is hard to reach. Consequently, hot gas is now often used, obtained by burning natural gas, predominantly methane. Another possibility is hot oil. Direct electric heating of the air can also be applied. Direct burning of natural gas in the drying air is very economical, but is undesirable (and generally illegal) as it causes some contamination of the powder with nitrogen oxides. The air is heated to  $\sim 200\,^{0}$  C and leaves the drier at  $\sim 100\,^{0}$  C.

# 29.4.2. Atomizing the concentrate in the air

A spinning disk is used for atomization of the liquid. The drops leave the disc in a radial and horizontal direction. The drying chamber must be wide to prevent droplets reaching, and thereby fouling the wall of the chamber. Currently, spraying nozzles are more commonly applied. Except for very small driers, several nozzles, arranged in one or more clusters, are installed. The drops leave the nozzles in a roughly downward direction. The air inlet is generally in the center of a cluster, also directed downward. The drying chamber can have a smaller diameter and often has a greater height, as compared to driers with disk atomization. The shape of the chamber is designed in such a way that the mixing of air and droplets and the course of drying of the droplets are (presumed to be) optimal. This produces such small droplets that they will dry very quickly, with either a spinning disk or a pressure nozzle.

## 29.4.3. Mixing hot air and atomized liquid

Drying occurs correspondingly. Air and liquid usually enter the drying chamber co-currently and are mixed so intensely that the air cools very rapidly. Consequently, the larger part of the drying process occurs at temperatures not much over the outlet temperature. The air inlet is generally tangentially, causing a spiral-like downward motion of the drying air.

## 29.4.4. Separating powder and consumed drying air

This is primarily achieved with cyclones, arranged in various ways. In a drier as seen from above, the air strongly rotates and the lower conical part of the drying chamber acts as a cyclone, and most of the powder is discharged below. The powder often is cooled before packaging. So-called fines, i.e., the smallest powder particles present, are returned to the drier, often close to the region of atomization. The air has to be cleaned before it is returned to the atmosphere. Commonly bag-shaped cloth filters are used. Another option is wet washing: the air stream is led through a falling spray of water, which is recirculated. The outlet air is still hot and part of the heat can be transferred to the inlet air in a heat exchanger.

# 29.4.5. Aggregation of powder particles

This can be achieved by nozzle spraying when the nozzles are arranged in such a way that the sprays overlap. Another method is to return the fines in a region where the drops are still

fairly liquid. A third way is rewetting in a fluid bed. In all these situations, sticky powder particles are caused to collide with each other.

## 29.4.6. Second-stage drying

It can be achieved in various configurations. In the drying of a liquid, several stages can be distinguished, e.g. a stage in which the liquid turns into a more or less solid mass and a stage in which the solid mass obtained decreases further in water content (final drying). In milk products, a solid-like material is obtained at a water content near 8% (the product obtained is no longer sticky and appears to be dry), whereas a powder with, say, 3% water is desired. Traditionally, one process step is included in both drying stages. In spray drying, advantage is often taken of separating the final drying, which is generally achieved in a *fluid bed*, apart from the main process.

## 29.5. DRYING FUNCTIONS

The spray drier utilizes a product which is first condensed in a vacuum pan or an evaporator. The product is then atomized inside a drying chamber of a drier. The drying functions include:

- § Moving the air
- § Cleaning the air
- § Heating the air
- § Atomizing the liquid
- § Mixing the liquid in the hot air
- § Removing the dry material from the air
- § Additional drying of the product
- § Cooling the product, and
- § Pulverizing and Sizing the product.

#### 29.5.1. CLASSIFICATION OF SPRAY DRYERS

The classification of spray dryers according to their different arrangements and types are shown in Table 29.1.

Table 29.1: Classification of Spray Dryers

No.	Criteria for Classification		Types
1	Method of atomizing spray	(a)	High pressure nozzles
	material	(b)	Centrifugal spinning discs
		(c)	Two fluid systems - air and steam
2	Method of furnishing heat	(a)	Steam
		(b)	Gas
		(c)	Fuel oil
		(d)	Electricity
3	Method of heating air	(a)	Direct-gas or fuel oil
		(b)	Indirect utilizing heat exchanger plates
			coils.
1	Position of drying chamber	(a)	Vertical
		(b)	Horizontal.
5	Number of drying chambers	(a)	One drying chamber
		(b)	Two drying chambers-main and secondar
			drying chamber.
5	Direction of airflow in relation to	(a)	Countercurrent
	product flow	(b)	Right-angle.
7	Pressure in drier	(a)	Atmospheric (usually a very slig
			pressure)
		(b)	Vacuum.
3	Method of separation of powder	(a)	Cyclone
	from air	(b)	Multi cyclone
		(c)	Bag filter.
9	Treatment and movement of air	(a)	Recirculation of air
		(b)	Dehydration of air
		(c)	Conventional-atmospheric air used an
			exhausted after use.
10	Removal of powder from drying	(a)	Conveyor
	chamber	(b)	Vibrator
		(c)	Sweep conveyor
		(d)	Air conveyed to cyclone.
11	Method of heat transfer	(a)	Convection
		(b)	Radiation.
12	Kind of atmosphere in drying	(a)	Nitrogen
	chamber	(b)	Air
		(c)	Other gas, usually inert
13	Position of fan providing	(a)	Pressure in chamber
		(b)	Suction in chamber
14	Direction of airflow in chamber	(a)	Updraft
		(b)	Downdraft
		(c)	Horizontal
		(d)	Mixed
15	Shape of drying/chamber	(a)	Silo or cylindrical
	. , ,	(b)	Boxlike, square cross-section
		(c)	Tear-drop
16	Product being dried	(a)	Milk
-		(b)	Other milk product
		(c)	Egg
		(d)	Other food products
		(e)	Chemicals
		(f)	Detergents

#### 29.5.2. CHARACTERISTICS OF SPRAY DRIERS

The acceptance of the spray drier is attributable to its favorable characteristics, particularly for a good product obtained and the economics of operation. These characteristics are:

- (1) Continuous operation
- (2) Little labour required to operate
- (3) Handle many different products
- (4) Heat contact to product is short during drying and removal (quality is more likely to be preserved as overheating is less likely)
- (5) Thermal efficiency is low compared to other types of driers
- (6) Operation dependent on large surface area of product which is obtained by different methods of atomizing
- (7) Separation of air and product after drying is important
- (8) Unit is easy to clean if operating properly
- (9) Economy of moisture removal improved by condensing product before drying
- (10) Product properties and quality may be effectively controlled

Normally, product does not contact drier surfaces until dried, (However, sticking of the dried or semi- dried material to the surface of the drier is a problem if the heat source fails and the product continues to enter the drier)

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# **Air Heating**

#### 30.1. INTRODUCTION

In the process of spray drying, air is playing a major role which enters the drier through air filter, air heater and finally drying the milk.

#### 30.2. AIR FILTER

Different types of air filters may be used. Close woven filters are unsuitable because of the heavy, extra load which their resistance places upon the suction fan. In some plants, the incoming air is washed by drawing it through a spray of water or through a series of screens of wire mesh or gauge over which a film of water trickles. The water spray or film of water is limited in its efficiency as an air filter. It removes the coarser mechanical impurities but it fails to purify the air from the grease and turbidity of city atmosphere.

The filter, most commonly used is a type in which oil is used as filtering element. It embodies the adhesive impingement principle of removing dust from the air. This type of filter consists of multiple cells filled with expanded metal. The cells are dipped in oil which coats the entire filter with adhesive film consisting of odourless oil that is sufficiently heavy to prevent being blown off. A plant with an average capacity of  $\sim 100$  kgs of dried skim milk per hour requires about 230.5 m<sup>3</sup> of air per minute.

In the presence of air of average purity, it is a good practice to wash the cells at least every two weeks. In order to maintain a satisfactory average condition of the filter, the rotation system of cleaning is generally preferred. The washing is done by soaking the cells overnight in hot water, containing an alkaline cleaning compound followed by blowing live steam through the cells for a period of 15 to 30 minutes. The clean cells are recharged by dipping them in oil and allowing them to dry in12 hours.

# 30.3. Air Heater

The air may be heated directly or indirectly or semi-direct such as by means of a furnace with hot air or by indirect means whereby the air passes over banks of fin type steam coils.

## 30.2.1. The Direct and Semi Direct Heating Systems

# Advantages:

- (1) These systems have greater thermal efficiency provided the escape of the furnace gases at uneconomically high temperature is prevented.
- (2) They attain higher temperature than are possible with indirect heating.
- (3) It consumes much of the oxygen  $(O_2)$  that is contained in the air and to that extent lessens the tendency of tallowy flavour in the milk powder.

## Disadvantages:

- (1) There is danger of getting black specks due to the presence of particles of extraneous material such as entrained furnace spot and dust, and scales of rust breaking loose from the flues.
- (2) Less temperature control.

## **30.2.2.** The Indirect Heating System:

This is the system commonly used in spray drying of the milk. The incoming current of fresh air passes over fin type of steam coils installed in the heating system. Heat control is facilitated by separate valve for each coil section. Fuel is saved by using exhaust steam in the first coil section and high pressure steam in subsequent section. Other media that may be used for heating the air in the spray drying system are hot oils and electric current.

## Advantages:

- (1) Easy control of temperature
- (2) Thermal loss can be avoided by placing insulated lines between the heating devices and drying chambers.

## **30.3. Drying Chambers**

The drying chambers vary in shape and size. Shape and size of the chamber varies according to the capacity of the unit and manufacturing firms. Main aim is to remove the powder as soon as it is dried. Some drying chambers are rectangular, other cylindrical, some have straight vertical sides, and others are cone shape slopping downward. Drying chambers are usually made of stainless steel and provided with observation glass. In modern times, dryer has been developed to produce instant milk powder, which will have tall tower type chamber.

For a reason of economic efficiency, the relationship of position of milk inlet into the drying chamber and air inlet and discharge should be such as to accomplish maximum saturation of drying air and maximum utilization of heat contained therein. For best preservation of important marketable properties of the resulting powder (quality and solubility), milk inlet and air inlet and discharge should be so located as to accomplish maximum rapidity of evaporation and uniformly low moisture content of the finished product.

#### 30.4. Counter Current versus Parallel Current Flow

In general, the milk and air either enter at opposite side (at top and bottom respectively) or flow in countercurrent toward each other or they both enter in the same region to get in parallel or co-current flow. In counter-current principle the points of inlet and direction of flow are so organized as to cause the incoming air (which is driest and hottest at this point) to meet the milk particles at the point of their lowest humidity and to cause the outgoing air (which is most nearly saturated) to leave the drying chamber at the point where the milk is wettest (which is near the atomizer).

## **30.5.** Effect of Parallel Flow on Uniformity of Particle Size

In the parallel flow principle, milk and air enter in the same region and travel concurrently. The seeming logic of the counter current principle, in which the drying milk passes successively through zones of dry air increasingly and finishes in the incoming air which is hottest and driest at the point, suggests maximum dryness of the finished product. It is conceivable, however, that the head on collision between air current and milk fog causes countless numbers of atomized milk particles to collide and fuse, forming big droplets, dissipating uniformity of particle size and impeding the speed of drying. In the parallel flow principle, milk fog and air currents move in the same direction, eliminating the danger of violent collision between milk particles and preserving the uniform fineness of particle size.

## 30.6. Effect of Parallel Flow on Rapidity of Drying

The parallel flow of milk fog and air current gives maximum rapidity of drying. The set up here is based on the theory that the high temperature and very low humidity of the incoming heated air in the presence of moisture content and tremendous surface area of the freshly atomized milk particles, cause maximum rapidity of the evaporation. The dry hot air at this point is at its maximum capacity for instantaneous absorption of the vapours released by the vast surface area of the drying milk. Simultaneously, the dried milk particles drop to the bottom while the saturated air currents exhaust through the top of the chamber.

It is of interest to point out here the tendency of the newer designs of air drying chambers is definitely in the direction of the parallel flow intake of milk dryers. Maximum rapidity of drying favours high solubility and keeping quality of finished product.

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#### **Atomization**

#### 31.1. INTRODUCTION

Atomization is aimed at forming droplets fine enough to dry quickly, but not so fine as to escape with the outlet air after having been dried. Moreover, a very fine powder has undesirable properties because it is hard to dissolve. Thus, t he objective of atomizing is to reduce the milk to a particle size so small that due to the tremendously increased surface area, the resulting mist of milk projected into the current of heated air, surrenders its moisture nearly instantly. The minute particles of milk are dried before they reach the side walls or floor of the drying chamber. The average particle size of the milk fog provided by efficient atomization has a diameter of  $\sim 50\mu$ . It is estimated that 0.5 lit of milk so atomized contains approximately 5625431140 particles, which represent a surface area of about 24586 m<sup>2</sup>. The milk must be sprayed into the hot air in form of very small particles as nearly uniform in size as possible because

- (1) In a mixture of sizes, the smallest particles might be prematurely dried and overheated whilst the largest might be only partially dried
- (2) In addition, uniformity assists toward better control of the initial mixing with the stream and easier separation of the powder from it afterwards.
- (3) The individual particles should also contain a minimum of trapped air.

In practice, completely uniform particle size has not yet been obtained but individual atomizers produce a majority of particles within a particular size range. Atomizer is a unit which distributes milk in form of very small droplets.

# **31.2.** Advantages of Atomization:

- (1) Rapid evaporation
- (2) Less effect on the quality of the product.

## **31.3.** Types of Atomizers:

- (1) Pressure jet type
- (2) Pneumatic type
- (3) Centrifugal type
- a. Hanging-Bowl type
- b. Disk type

## c. Ring jet type

# 1) Hydraulic Pressure Jet Type:

In this type, the original form of atomizer forces the milk at a high pressure ~ 141-211 kg/cm<sup>2</sup> through a very small orifice, which may be only 0.125 mm in diameter, in a hard steel disk. Modern types impart swirl velocity in addition to axial velocity by tangential entry of the milk to a swirl chamber preceding the orifice.

The atomizer produces a hollow cone spray with the droplets dispersed in an annular ring around the air cone and with a decreasing particle size towards the outer edge. Although drop size is not uniform, it is possible to produce a range of size. Drop size is reduced by increasing the pressure, reducing the nozzle diameter, or reducing the liquid velocity, but this type of atomizer is unsuitable for producing a large proportion of drops below 30µ diameter. The viscosity of the concentrated milk influences the possible pressure with a given orifice, and in general a high degree of concentration is not satisfactory.

#### Advantages:

- (1) Its construction is simple,
- (2) It is possible to adjust the angle of the cone-shaped spray of the atomized liquid thereby allowing a relatively small diameter of the drier and low vacuole content in the powder particles.
- (3) When drying milk, the fat globules are disrupted into much smaller ones, about as they would be in a homogenizer as the pressures applied are comparable.

#### Disadvantages:

- (1) Choking of the jet orifice
- (2) Alteration of the spray due to an increase in the orifice diameter as a result of erosion, spare nozzles must always be available
- (3) This atomizer is not suitable for handling special mixtures containing milk with other solids in suspension
- (4) High pressure pumps and milk lines are necessary.

## 2) Pneumatic type of Atomizer

A stream of compressed air is used to disintegrate a jet of milk. The air impinges at high velocity onto a film of liquid projected at right angle to it, or alternatively several air jets impinge on the milk stream from an angle. In this method

- (1) It does not require high pressure pumping of the milk
- (2) Erosion of jet orifices is reduced

(3) A more concentrated liquid can be sprayed as compared with the pressure jet type.

A very high velocity is achieved and the spray travels a long distance before breaking. This atomizer is the best type for producing very small particles and is not satisfactory when the size range is required to exceed 50  $\mu$ . More energy is required per unit of surface area created than with the other two types.

## 3) Centrifugal type Atomizer

No pressure is applied to the milk, the velocity being imparted by centrifugal force in a rotating device. The milk leaves the periphery of the atomizer as either in thin sheet or filaments which break up into spherical droplets in an umbrella-shape spray. There are several types of disks but, essentially, the liquid falls on a disk and is flung away at a high speed, e.g.,  $100 \text{ m} \cdot \text{s}^{-1}$ . Different types are:

- (a) **Hanging inverted bowl:** The concentrated milk is fed to the underside of the bowl, which rotates at a high speed. The milk is thrown outwards by centrifugal force, spreads evenly round the circumference, and leaves the rim as a film of uniform thickness.
- (b) Flat wheel shaped hollow disk: It is having openings around the periphery or a hollow slotted basket. The concentrated milk is fed to the centre of the rotation device and is discharged through the opening around the circumference. The speed of rotation is usually high varying between 200 to 300 revolutions per second according to the diameter of the bowl or disk and the peripheral velocity desired.

The drop size depends upon the velocity (decreasing with increasing velocity) and can therefore be varied by altering the speed of rotation or the disk diameter. This permits the production, within certain limits, of powders of different bulk densities. In general however, centrifugal atomizers tend to produce powders of larger average particle size than do jet types. The normal range is from 50 to 250  $\mu$ .

The centrifugal atomizer is usually incorporated in modern plants because of the following characteristics:

- (a) There are no small orifices to become chocked and, when necessary, materials containing suspended solids can be handled.
- (b) High pressure pumps and pipes are eliminated; the atomizer is often supplied by a simple gravity feed, although a timing pump is an advantage.
- (c) Highly concentrated milk can be sprayed.
- (d) In addition to the thermal economy which results, the bulk density of the powder particles increased whilst the volume of entrapped air is reduced as more concentrated milk is sprayed. This is of considerable importance in connection with both packaging and keeping quality of dried milk.
- (e) Moreover, powder containing, a high proportion of large particles readily obtainable with centrifugal atomizers tend to dissolve in water the most easily.

- (f) It has also been claimed and later disputed that a greater degree of uniformity of particle size is obtainable.
- (g) Disk atomization is still practicable at high viscosity; highly evaporated milk can thus be processed.

## Disadvantages:

- (a) Many vacuoles are formed in the particles
- (b) The droplets are flung away perpendicularly to the axis of the disk and, accordingly, the chamber has to be wide to prevent the droplets from reaching the wall. Roughly speaking, the distance covered by the droplets in a horizontal radial direction is at least 104 times the droplet diameter.

The modern preference is for the milk to leave the atomizer in definite streams (i.e. from opening in a disk or special channels in an inverted bowl.) rather than as a flat sheet.

(c) Ring – jet Atomizer: A "ring-jet" atomizer operates in two stages which combine centrifugal force and a blast of cold air. The concentrated milk is fed to the centre of a centrifugal spray disk which rotates inside an air nozzle, arranged so that a ring space is formed between the nozzle outlet and the periphery of the disk. The disk rotates at a relatively low speed (3000 rpm with a direct driven motor) and spreads a jet of milk into a fine film and coarse spray with an average droplet size of about  $300\mu$ . The ring-shaped blast of cold air then shatters the drops around the periphery of the disk and reduces their size to about  $50 \text{ to } 100\mu$ . A close control of the droplet size is possible; the latter varies with the intensity of the air blast which is governed by the air supply pressure. Moreover, the air content of the particles is greatly reduced. The ring jet is arranged to give n downward spray and the hot drying air also passes vertically down the drying chamber.

It is possible to produce heavy powders containing little trapped air and regulating the particle size to about 100μ. Powders of very good wettability can be obtained.

#### 1. Droplet Size Distribution

Determination of the size distribution of the formed droplets is difficult and none of the methods available for particle-size determination is fully reliable. Usually, the produced powder is taken as a basis, but that involves several uncertainties like the uneven shrinking of droplets, presence of vacuoles, and possible agglomeration of the powder particles.

To obtain smaller drops, the concentrate is often heated to decrease its viscosity. However, this should be done immediately before atomization because viscosity increases rapidly within a minute at a high temperature, especially for concentrated skim milk.

#### 2. Vacuoles

During the atomizing of a liquid, some air is always trapped in the droplets. This generally concerns some 10 to 100 air bubbles per droplet when a disk is used, whereas the number is far less when applying a nozzle, often 0 or 1 air cell per droplet. During the drying of the droplets, water vapor enters the air bubbles, causing them to expand. This is because the

water vapor can more easily diffuse to the vacuoles than across the external layer of the drying droplets, which has already been concentrated and has become rigid. Therefore, the vacuoles are only partly filled with air. Raising the drying temperature increasingly expands the vacuoles and enlarges the vacuole volume. Figure 31.1 shows the morphology of powder particles with vacuoles.

Fig. 31.1: Cross sections of powder particles obtained by spray drying of (A) evaporated whole milk, nozzle; (B) evaporated whole milk, disk; (C) evaporated skim milk, disk; (D) skim milk, disk (in sequence). The broken line shows the outer projection of the particle.

#### 31.4. CHANGE OF STATE OF THE DRYING AIR

The temperature to which the drying air may be cooled primarily depends on the corresponding water activity. Higher inlet temperature gives higher efficiency of drying. However, there is an upper limit with respect to the inlet temperature, partly because of damage to the product caused by heating. Moreover, the powder may **catch fire** in a drying chamber if it stays for a long time at a high temperature (this concerns powder deposited anywhere in the machinery). Ignition may already occur at 140  $^{0}$  C and at 220  $^{0}$  C, the time needed for spontaneous ignition is about 5 min. Therefore to control the moisture of the powder, following relationships should be followed:

- 1. If the percentage of the water in the powder has to be increased, the a w value of the outlet air should be increased; hence, outlet temperature should be lowered.
- 2. If the dry-matter content of the concentrate entering the dryer is increased or its temperature decreased, the atomization results in larger droplets, primarily by the increased viscosity. To keep the water content of the powder the same, the  $a_{\rm w}$  value of the outlet air should be lower; hence, outlet temperature should be higher.

It is common practice to control the water content of the powder, which varies due to small fluctuations in the evaporating and drying process, by regulating the concentrate supply in such a way that outlet temperature is kept constant. If outlet temperature increases, the concentrate flux is increased, and vice versa. However, the points just mentioned indicate that this is not fully correct. More sophisticated methods using computer programs have been developed to improve control of the drying process.

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# **Separation of Air and Powder**

#### 32.1. INTRODUCTION

As the product is dried, it is necessary to separate the dried product from the air. Without special design features, the product will be carried by the moist air from the drier. It is necessary to remove the particles

- (a) To get a maximum yield from the dryer by saving all powder product and
- (b) To avoid air pollution surrounding the drying plant.

#### 32.2. Location

The powder may be separated from the air primarily either

- (a) Inside the drier (internal) or
- (b) Outside the drier (external).

In both cases, it is necessary to use an additional device or component outside the drier to remove the fine or small particles which will not normally settle out in the drier. External separation devices are used on all driers.

#### 32.3. Methods

The products which are separated from the air in the drier can be removed from the drier by:

- (1) An air brush, by which air from outside the drier either at room temperature or conditioned to a lower temperature is used to direct a jet of air to move the product from the bottom of the drier,
- (2) A rake or broom which is pulled across the bottom of the drier,
- (3) A conveyor flight or auger type and
- (4) A gravity system.

Vibrators are often attached to the sides of the drier to prevent; or reduce sticking of the powder and to move the product rapidly from the drier.

#### 32.4. Variables

The variables affecting separation of powder from the air are:

- 1. Particle size
- 2. Concentration
- 3. Nature of the material, and
- 4. Quantity of the product

In a drier, where most of the powder is removed internally, the quantity of air handled by the separation system will be the same as for external separation, but the quantity of product will be much less. The product nature or characteristics such as fat, moisture, cohesion, and friction greatly affect the efficiency of separation or collection. The efficiency of collection is designated as the ratio of the output divided by input times 100% with the product at the same moisture content at the two locations. Some manufacturers rate the efficiency of collection or separation on the basis of kgs of dry matter input. If all of a product at -three per cent moisture is collected, an efficiency of 103 % would be claimed using this method of rating.

## 32.5. TYPES OF SEPARATORS

## **32.5.1.** Cyclone Separators

Cyclone or Multi Cyclone is a type of interstitial separator and is most commonly used for removing the dry product from the air. Air at a high velocity moves into a cylinder or cone which has a much larger cross-section than the entering duct. The velocity of the air is decreased in the cone, thus permitting the settling of the solids. The velocity of the air decreases near the wall of the cylinder or cone and the product falls by gravity and is removed from the bottom. Cyclones may be used for storage of product before packaging, to provide for a more efficient packaging operation.

**Centrifugal Force**: The centrifugal force acting on the particle for removing the product from the air is

$$CF = m \times a = \frac{w}{g} \times a = \frac{wV^2}{G \times r}$$

Where:

CF = force on the particle, kg.

w = weight of particle, kg.

 $g = acceleration due to gravity, 9.75 m/sec^2$ 

V = velocity of particle, m/sec.

r = radius, i.e. the radius from the center of the cyclone to the particle position or the radius of rotation in meter. The centrifugal force on the product is exerted toward the edge of the cyclone. The weight of the particle is effective vertically in the direction of the outlet.

**Arrangement**: Cyclones can be used individually or in combination, to provide multi cyclone units.

**Efficiency**: The efficiency of separation with a cyclone unit is based on product, cyclone design, and on the size of particle to be removed, but losses range from 0.5 to 3 and average one per cent. The cyclone is normally used for separation of material between 5 and  $200\mu$ . As the size of particle decreases, the efficiency of the cyclone also decreases. A properly designed cyclone will remove 99% of the solids larger than  $30\mu$ , 98% of material larger than  $20\mu$ , 90% of material larger than  $10\mu$ , but only 50% of the material smaller than  $5\mu$ .

# 32.5.2. Cloth Collectors or Bag Filters

They have been used for many years. The material may be cotton, wools or plastic. Approximately  $0.031 - 0.046 \text{ m}^2$  of bag or cloth surface per  $0.028 \text{ m}^3$  per minute of airflow is provided for external separator. Losses generally range from 0.2 to 0.5%.

## 32.5.3. Wet Scrubber or Liquid Device

They may be used where the fine particles passed through other separation devices are removed from the exhaust air and returned to the incoming product for redrying. The wet scrubber provides for a high recovery of product and heat from the exhaust air. The disadvantages are the possibility of product deterioration and contamination.

# 32.5.4. Electrostatic, Sonic or Ultrasonic, Electrical, and Packed Beds of Granular or Fibrous Materials

These types of separation now also used in the milk industry to separate fine solids from the air stream.

## 32.6. Explosion

Explosion caused by concentration of organic materials in the atmosphere depends upon the increase in temperature of the particles to the ignition point. The possibility of explosion usually is designated on the basis of concentration of the product in the air. It is generally considered that a concentration of organic dust of 178.5 g/m<sup>3</sup> or less is safe from the stand point of explosion. Moreover, the powder may catch fire in a drying chamber if it stays for a long time at a high temperature (this concerns powder deposited anywhere in the machinery). Ignition may already occur at 140 °C; at 220 °C, the time needed for spontaneous ignition is about 5 min. Hence, a dequate ventilation must be provided to maintain the concentration at low level.

## 32.7. Cooling the Powder

The dried product should be removed from the drier as quickly as possible after it is produced to minimize the effect of heat damage on the product. The product and air may be removed together from the drier and separated out side of the drier to reduce heat-effect.

Product cooling is done to prevent clumping, sticking and heat damage to the product. Prolonged heating causes staleness in nonfat dry milk, and causes fat to melt and move to the surface of whole milk powder. With more of the fat on the surface of the powder, the product will not keep as well in storage. Warm powder will hold the heat for some time in a bulk container, thus increasing the heat damage. The thermal conductivity, or k-value, is estimated at 0.03 Btu per hr sq ft  $^{0}$ F per ft., which is considerably lower than most food particles and very similar to insulation materials.

Some cooling of the product will take place in the drier when using an air brush supplied with cool air to remove the dried product from the sides and bottom of the drier. The three principles of cooling powder outside the drier involve:

- (1) **Conduction cooling** in which the product is cooled when moving through water jacketed screw conveyor.
- (2) **Convection cooling** by using room air or refrigerated air to cool to 38<sup>o</sup>C or by moving conditioned air over the product or through the conveyor handling the product.
- (3) **Radiation cooling** by placing a cold evaporator surface in view of the warm product. This method has not been exploited by the dry milk industry.

The outlet of a cyclone separator can be surrounded by a chamber through which cold-air is moved to cool the product. The material moves on to an entrainment separator for separation of solids and air. A vibrating conveyor for moving dry milk permits cooling as the product moves through the surrounding air. Oxygen can be removed more easily from warm powder than cold, when an inert gas, such as nitrogen is used for packing under vacuum. The amount of heat to be removed is given in Calorie by multiplying the specific heat by the weight, in kgs times the difference in temperature ( $^{0}$ C). The density of drum dried milk is 0.3 to 0.5 gm per ml and for spray dried is 0.5 to 0.6 gm per ml. Some drying occurs in the cooling process. About one third to one half of the heat removed in the cooling process can be considered to be used for vaporization of water.

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# **Two-Stage Drying**

#### 33.1. INTRODUCTION

Spray drying is relatively expensive with respect to energy and the capital outlay for driers is high. Hence, a better efficiency is required and in principle, it can be achieved by increasing the concentration factor of the milk before atomization as well as by applying a higher air inlet temperature. However, these measures can lead to heat damage of the product. Alternatively, the powder can be separated from the air before it is completely dry, while additional drying occurs outside the drying chamber. In this way, the outlet temperature of the air can be lower, allowing the inlet temperature of the air to be higher without increased heat damage occurring. Moreover, a larger quantity of concentrate can be dried per unit time.

#### 33.2. PROCESS

The powder may be discharged from the drying chamber after it has become so dry as to have lost its stickiness. The problem of stickiness is less than expected because of the concentration gradient formed in the powder particles. In the center of the particle, the water content is 24% for drying time of 0.8s. It is on average about 13% but only about 2% at the periphery. Presumably, these particles would still be slightly sticky because

- (1) Stickiness i.e. the tendency to stick to the machinery considerably increases with temperature; and
- (2) Upon removal from the drying air, the outside of the powder particles rapidly increases in water content due to internal exchange of water. Moreover, larger particles will be 'wetter,' hence, more sticky. But a powder with an average water content of about 8% can readily be discharged by means of cyclones.

The final drying is often achieved in a *fluid bed drier*. A layer of powder deposited on a perforated plate can in principle be fluidized by blowing air through the layer from below. In such a fluidized bed the powder layer is expanded, containing a high volume fraction of air; the mixture can flow, almost like a liquid, if the perforated plate is slightly tilted. The particles in the bed are in a constant erratic motion, which enhances drying rate. The conditions for fluidization are

- (1) The particles size is from  $\sim 20~\mu$  m to a few mm and their size distribution is not very wide; and
- (2) The air flow is evenly distributed over the bed and has a suitable velocity, e.g., about 0.3 m/s for most spray-dried powders.

Generally, the size distribution of milk powder particles is too wide: if all particles are to be fluidized, including the largest ones, the air velocity must be so high that the smallest particles are blown out of the bed. To overcome this problem, the machine is made to vibrate,

which allows fluidization at a lower air velocity. Such a fluid bed drier can then be attached to a spray drier by a flexible pipe.

In a spray drier the air inlet temperature is high; the holdup time of the powder is short, say, a few seconds. In a fluid bed drier the air inlet temperature is relatively low (e.g., 130 °C), little air is consumed, and the residence time of the powder is much longer, i.e., several minutes. Because of this, a fluid bed drier is much more suitable for the final stages of drying. For example, in a comparison between traditional and two-stage drying as shown in Table 33.1, using the same spray drier, the same skim milk concentrate with 48% dry matter, dried to the same water content of 3.5%, may yield the following:

Table-33.1. Comparison between traditional and two-stage drying parameters

No.	Number of Stages	1	2
1	Inlet air temperature (°C)	200	250
2	Outlet air temperature, chamber (°C)	94	87
3	$a_{ m w}$ outlet air, chamber	0.09	0.17
4	Total heat consumption (kJ/kg of water)	4330	3610
5	Capacity (kg of powder/h)	1300	2040

The efficiency of the heat expenditure thus is better (by 17%) and the capacity greater (by 57%); against this is the capital outlay for the fluid bed drier. The additional drying consumes only 5% of the heat. The quality of the powder (insolubility index) is certainly not poorer but, generally, better.

A fluid bed also offers additional opportunities such as,

- 1. It is quite simple to add a cooling section.
- 2. The bed can also be used for agglomerating purposes. The main incentive for agglomeration is that a fine powder poorly disperses in cold water.
- 3. Therefore, often an attempt is made to produce a coarse-grained powder. In the fluid bed the powder particles collide intensely with each other. As a result, they agglomerate if they are sufficiently sticky, i.e., have high enough water content at their periphery. Hence, agglomeration is enhanced by blowing steam into the powder (this is called rewetting, which is mostly applied when producing skim milk powder).

The air velocity in the fluid bed may be adjusted in such a way that the smallest powder particles (which have already become very dry and therefore show poor agglomeration) escape separation. The latter particles are fed back to the drying chamber, gain entrance to the atomized liquid, and become agglomerated with the drying droplets. This process is especially applied for whole milk powder.

**Fig.33.1:** Examples of Two-stage drying processes: (a) Spray drier with fluid bed (b) Fluid bed in the bottom end of a spray drier. (c) Filter mat drier.

Two-stage drying can also be effected in a modified spray drier chamber. In the bottom end of the chamber, a fluid bed is realized. It need not be vibrated as the smallest particles are blown toward the atomization region where they agglomerate with the drying drops. The air circulates in the vertical direction and is removed near the top end of the chamber. A great variety of configurations has been developed: for instance, a combination of the type depicted in (b) with a fluid bed attached as in (a) of Figure-33.1.

Another method of two-stage drying is carried out in the *filter mat drier*. The first stage is conventional spray drying. Atomization is by nozzles, and the flows of air and drop spray are cocurrent. The partly dried drops fall on a moving perforated belt. The spent air is removed through the powder bed formed on the belt and is sent to cyclones. The fines removed by the latter are added to the second section of the machine, where it agglomerates with the powder on the belt. In the third section, hot air is blown through the bed for final drying. The dry material then reaches the fourth section, where it is cooled. The powder particles become strongly aggregated and form a porous cake, which falls from the belt in large lumps. These are gingerly ground and the resulting powder is packaged.

The filter mat drier allows a greater part of the water to be removed in the second stage because the powder can hit the belt when still being sticky. The latter also makes this type of drier suitable to handle very sticky materials such as cream powders.

## 33.1. MANUFACTURE OF NONFAT DRY MILK

The manufacture of nonfat dry milks is shown as a Flow chart in Figure 33.2. For the manufacture of Non fat dry milk (NFDM)/ skim milk powder, the pasteurization can be less intense (at least phosphatase negative), according to the intended application. Homogenization is omitted, and the milk can be concentrated to somewhat higher solids content. Also no lecithinizing and gas flushing carried out. Sometimes vitamin preparations are added, especially vitamin A. This can be achieved by dry mixing afterward, or by emulsifying a concentrated solution of vitamin A in oil into a part of the skim milk.

The equipment for processing most dry milk products usually includes a separator, preheated, and/or high temperature short time pasteurizer with flow diversion valve, hot well, evaporator, preheater, filter in concentrate line, high pressure pump and other pumps, drier with milk dust collector, cooler, sifter, and packaging equipment. Many variations in equipment and methods are in use. In fact, no two plants are exactly alike in equipment and method.

In the design of equipment layout, a simple forward flow arrangement is best for quality and efficiency. The least equipment contact and the faster the forward movement of the product, the better will be the quality of the dry milk. Recycling may be detrimental to product quality and should be limited especially for low-heat nonfat dry milk. Elimination of recycling is more difficult for equipment that is used to dry several products compared to single product processing.

Fig-33.2: Flow chart for the manufacture of low-heat and high-heat non-fat-dry-milk

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# **Non Fat Dry Milk**

#### 34.1. INTRODUCTION

Low-heat nonfat dry milk manufacture requires that heating be carefully controlled to result in minimum amount of induced changes and yet accomplish proper pasteurization. Control of both time and temperature assumes much significance. High temperature short time pasteurization as part of the preheating may be by tubular or plate heaters to not less than 71.6  $^{0}$ C at least 15s. Higher temperatures or longer holds contribute directly to whey protein denaturation. This index is used as a measure of the cumulative detrimental heat effects during processing of non fat dry milk. Not more than 10% denaturation should occur.

## 34.2. Evaporation

Pasteurized skim milk is condensed continuously in an evaporator (or vacuum pan) to 40 to 48% TS. The Baum'e hydrometer is used to test the total solids during condensing. For the conversion of Baum'e reading to total solids, an in-line refractometer may be used for continuous indication of the total solids. For a low-heat product, the evaporator must be designed to operate efficiently with the product temperature below that which causes the denaturation of the proteins. Some types of double and triple effect (or more) evaporators are not suitable for production of low heat product. Therefore, necessary precaution must be taken to be sure that the design and operation of the double effect (or more) evaporator is satisfactory for low-heat product manufacture.

Once the desired total solids are obtained, the condensed skim milk is pumped continuously from the evaporator to a small balance tank. From there the condensed skim milk is pumped through a preheater to raise the temperature to 61.6 to 68.3  $^{0}$ C.

#### 34.3. Spray Drying

The high pressure pump, usually of the piston type forces the hot product through the spray nozzle into mist-like droplets in the drying chamber. The pressure of the pump ranges from about 70 to 350 kg/cm<sup>2</sup> depending on manifold conditions such as nozzle design and size, inlet and outlet air temperature, drying chamber characteristics, particle size, and moisture content desired. Other types of spray nozzles are suitable for low pressure and one utilizes high velocity hot air to achieve atomization of the liquid.

Inlet air is heated by direct flame or steam coils to 120 to 260  $^{0}$ C. Drying chamber design and subsequent equipment, as well as climatic conditions and desired moisture in the dry product influence the inlet and exit air temperatures. The exhaust air temperature is the direct guide in controlling moisture of the product. But changes are achieved through adjustment of the inlet air temperature. Low heat nonfat milk is dried to 3.0 to 4.0% moisture. Some driers have an auxiliary heater or re-drier located after the primary drying chamber. These are useful when the air is humid or for enlarging the total drying capacity of the equipment.

## 34.4. Removal, Cooling and Sifting

Most milk driers have a continuous removal system to immediately separate the dry product from the hot air stream which has been reduced to the range of approximately 80 to 99°C. The dry product should be cooled at once to approximately 32 to 43°C. A few plants still depend upon ambient conditions for cooling. Nonfat dry milk packaged to hot may become lumpy due to "heat-caking" and the development of off-flavor and off-color during storage is much more rapid. A 25 mesh screen with No. 36 wire gage is commonly used for sifting nonfat dry milk ahead of packaging.

## 34.5. Recommendations to make Low-heat powder

- (1) Apply low pasteurization (72 ° C,15 s),
- (2) Begin the evaporating process not above 70 ° C,
- (3) Evaporate the milk not too far,
- (4) Keep the concentrate temperature below 60 ° C,
- (5) Cool the concentrate if it is to be kept for a fairly long time,
- (6) Maintain the outlet temperature during spray drying at a low level, and
- (7) Mix air and droplets in the drying chamber such that no local overheating of the drying droplets can occur.

## 34.6. HIGH - HEAT NON FAT DRY MILK

## 34.6.1. Pre Heating

Except for the total heat treatment before drying, the manufacture of high heat nonfat dry milk is the same as for the low-heat product. In processing high-heat nonfat dry milk, care must be exercised to subject the skim milk to a time-temperature treatment that will impart good bread baking qualities to the nonfat dry milk. During the heat treatment, the whey (serum) protein is denatured. A test for the whey protein nitrogen is used as an index of the suitability of the dry milk for bread. The heat treatment usually is applied to skim milk directly following pasteurization. The general practice is to heat the skim milk in a hot well to 85 to 88 °C for the equivalent of 15 to 30 min by a continuous flow of skim milk in the top and later out from the bottom.

## 34.6.2. Condensing and Spray Drying

The condensing step parallels the one used for the low heat product, except the evaporator is operated for maximum efficiency without concern about serum protein denaturation. The reheating of the condensed milk ahead of the high pressure pump may be higher than for low-heat nonfat dry milk. Temperature of condensed milk may be elevated to 74 to 79 ° C or higher. A high temperature improves drying efficiency, but is limited by several conditions. The temperature must be maintained below the level where casein denaturation will cause a high solubility index.

The specific temperature at which the increased solubility index affects the grade of the dry milk is dependent upon

- 1. Heat stability of the milk,
- 2. Preheat treatment and
- 3. Time lapse necessary to allow the condensed milk to flow from the heater outlet through the high Pressure pump to the spray nozzle.

In some plants, the temperature is restricted by the built up of solids on the contact surface of the heater. Since the aim for all plants is to use the condensed milk heater for the entire day's operation (up to 22 hr) before cleaning, the maximum temperature is limited.

# 34.6.3. Moisture Control

The pump pressure ordinarily is within 105 to 350 kg/cm $^2$  with the outlet air drying temperature usually between 76 and 99  $^\circ$  C . The final moisture in the high heat nonfat dry milk is controlled to approximately 3 to 4 %.

#### 34.6.4. Orifice Size

The diameter of the orifice in the spray nozzle is selected on the basis of several factors. Drying chambers with a single spray nozzle have a large orifice. The common range in diameter is 0.272 to 0.449 cm. Orifice size for the multi-nozzle chamber usually is from 0.0635 to 0.132 cm for non fat milk drying. Other design characteristics of the drier affect the orifice size. The inlet or outlet air temperature and pump pressure will influence the orifice size selection and spray pattern. If other conditions are kept constant, a single large orifice increases the average particle size which in turn increases density and sinkability of the particles compared to drying with several nozzles.

The operator must occasionally observe the spray pattern for a malfunctioning nozzle. A particle may lodge in the nozzle and obstruct the spray pattern. An excessively worn nozzle causes an unsatisfactory spraying of the product. Both conditions can cause inadequate drying which may result in milk solids adhering to the drying chamber surface, cause lumps and higher moisture in the dry milk.

## 34.6.5. Recommendations to make High-heat powder

- (1) The milk should be intensely heated, e.g. 90  $^{\circ}$  C, 5 min or 120  $^{\circ}$  C, 1 min; often still higher temperatures are used.
- (2) A more intense preheating causes a higher viscosity of the concentrate at the same drymatter content with all of its consequences.

## 34.7. INTERMEDIATE HEAT NON FAT DRY MILK

Intermediate heat nonfat dry milk is from skim milk that has been preheated too much to qualify as low heat, but not enough to meet the high heat requirements. The whey protein nitrogen is more than 1.5 mg per g of nonfat dry milk but less than 6.0 mg. The other processing steps are the same as described for high-heat nonfat dry milk.

#### 34.8. MODIFIED DRYING PROCEDURE TO IMPROVE DISPERSIBILITY

To improve the sinkability or dispersibility of non fat dry milk as an alternative for agglomeration of nonfat dry milk, several processing steps may be modified. Each is changed to affect maximum particle size and uniformity. This involves

- (a) Concentrating the skim milk to 45 to 50% TS
- (b) A relatively low pressure for spraying
- (c) A larger nozzle orifice
- (d) The temperature of the inlet drying air is raised to obtain adequate moisture removal from the larger spray droplets
- (e) A system of separating the very small particles either by an air stream or sifting is practiced
- (f) Seeding the condensed skim milk and partial crystallization of the lactose can also be used.

Under regular conditions of drying, the separation of large particles by screening may yield 10 to 40% of the production. The product with improved dispersibility may command a small premium price. But handling of the remaining product may present a disadvantage. Furthermore, the reconstitutability of nonfat dry milk from the modified drying and screening method is generally inferior to the agglomerated product. It often is darker in color, higher in moisture and has more insoluble material (denatured protein).

## 34.9. VITAMINS FORTIFICATION OF SPRAY DRIED NON FAT DRY MILK

Nonfat dry milk contains all the vitamins of whole milk except those that are fat soluble A, D, E, and K. Of these, vitamins A and D are most frequently associated with milk, so their restoration in non fat dry milk has merit. Nonfat dry milk may be fortified with vitamin A and D by two procedures:

- 1. **Wet Process:** The vitamins in a liquid are diluted with blend edible oil (melting point ~ 37 ° C ) @ 1.5 kg of oil to the vitamins A and D concentrate sufficient for 1000 kg of milk solids. It is important that the oil and vitamins must be thoroughly homogenized to delay oxidation. Ten per cent additional IU of A and D have been recommended to assure minimum requirement after storage. The vitamin mixture may be manually added to vats of fluid skim milk or to the concentrate of known solids content. If the flow of product is reasonably constant or synchronized, the vitamin mixture may be metered into the product. Sufficient mixing must occur for uniform dispersion before drying.
- **2. Dry Process:** A dry form of vitamins A and D may be premixed with non fat dry milk and the premix dry blended into the main lot by batch or continuous procedures. The deterioration of vitamin A in storage is a little slower by the dry blending procedure; however, the vitamin cost and the expense of addition is higher.

#### 34.10. SELECTION OF THE VITAMIN CONCENTRATES FOR FORTIFICATION:

- § It should be able to have uniform distribution in the nonfat dry milk.
- § The vitamins must not adversely affect the flavor of the nonfat dry milk, either in the fresh condition or after prolonged storage.
- § The vitamin potency should remain stable for the shelf life of the fortified product.
- § The vitamins also should remain biologically available during storage.
- § The vitamin A compounds commonly added are retinyl palmitate and retinyl acetate.
- § The vitamin D compounds are rrgocalciferol and activated rehydrocholesterol.

# 34.11. MANUFACTURE OF NON FAT DRY MILK BY DRUM OR ROLLER PROCESS

The equipment and other facilities for a drum drying operation can be procured for a much lower minimum cost than for spray drying. Consequently, the drum process may be more feasible for a small volume than the spray system. In addition, some industrial users prefer a drum dried non fat and whole milk.

The skim milk should be pasteurized and the heating should be continued to approximately 85 °C for 10 min to be sure of good bake test properties. Removal of moisture by condensing to ~ 2:1 will greatly increase the capacity of the drums. If the TS is increased above 18%, the film on the roll becomes increasingly difficult to dry satisfactorily. Higher drum temperature or slower speed will cause scorching of the product despite insufficient drying. The product at 74 to 85 °C is pumped into the reservoir between the two drums. Usually the range in moisture content of drum process nonfat dry milk is 3.0 to 4.0 %. Under proper drying conditions, a uniform, thin light (in colour) sheet of dry milk is scraped from each drum. This film drops into a trough. The screw conveyor breaks up the product into small pieces, while being augured to a flaker or hammer mill. Particle size is decreased sufficiently by the mill to permit the passage through the 8-mesh screen during the sifting operation. The product is usually packaged in Kraft bag with a plastic liner reasonably impervious to moisture vapors.

#### **Precautions:**

- 1. Scorched particles content (brown and black) may be a problem.
- 2. In addition to the correct steam pressure, the proper roller conditions are important otherwise, under or over dried milk solids residues contaminate the ensuing product.

Usually quality difficulties are encountered briefly while starting the drying operation.

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# **Dry Whole Milk**

#### 35.1. INTRODUCTION

The manufacture of dry whole milk is not much different from nonfat dry milk. Since keeping quality is a restricting factor, care should be taken to obtain the maximum storage life. High quality milk with a low copper and iron content, sanitary plant practices, and a good processing procedure are important influences on keeping quality.

#### 35.2 MANUFACTURE

The manufacture of whole milk powder with all necessary stages of manufacture starting from taking milk upto the packaging and storage are shown in Figure 35.1.

Fig. 35.1: Flow diagram for the manufacture of whole milk powder

#### 35.2.1. Milk Standardization

The milk fat is standardized in ratio to the solids not fat so the dry product will meet the legal standards. Clarification may be conducted either before or after standardization to remove leucocytes and safeguard against the possibility of extraneous material in the product.

## 35.2.2. Homogenization

The concentrate is not always homogenized, especially if atomization is done by means of a nozzle, because the fat globules are effectively disrupted in the nozzle. Homogenization of highly concentrated milk considerably increases its viscosity because the transfer of large casein micelles to the fat globules gives the latter such an irregular shape as to increase the effective volume fraction of fat globules plus casein micelles. This increase leads to coarser droplets during atomization, with all drawbacks involved. Consequently, if the concentrated milk is not homogenized, evaporation can continue up to higher dry-matter content.

Homogenization of the whole milk is common if direct reconstitution of the dry whole milk is contemplated. Without homogenization the fat may churn during agitation while combining with water. Another advantage of homogenization is the improvement of keeping quality. The fat globules, although smaller, are more thoroughly recovered in the protein membrane. A pressure of 175 to 250 kg/cm² at 62 to 74 ° C provides sufficient homogenization.

#### **35.2.3. Storage**

Storage (buffering) of the concentrate before atomization is not always applied; it is done mainly to overcome differences in capacity between evaporator and drier. However, the concentrate should not be kept warm for more than a short time to prevent the growth of microorganisms. A refrigerated concentrate generally is too viscous to be atomized readily, and it is therefore heated just prior to atomization because otherwise the viscosity increases again. The heating can at the same time serve to kill bacteria that may have recontaminated the concentrate.

## 35.2.4. Preheating

### **Advantages:**

- 1. Intense pasteurization is needed to obtain resistance to autoxidation.
- 2. Low-heat treatment minimizes the cooked flavour in the product, but does not develop antioxidants for delay of oxidation, one of the principal factors in keeping quality.
- 3. Usually the primary consideration is given to prolonging the shelf life.
- 4. Preheating also destroys the enzymes. If the lipase enzymes are not destroyed, hydrolytic rancidity will occur in the dry whole milk.
- 5. Preheating must accomplish pasteurization, thus reducing the viable microorganisms.
- 6. A beneficial influence on heat stability of the product may occur from the preheat treatment.
- 7. Heating of the milk also is necessary before it enters the evaporator.

## 35.2.5. Manufacturing Process:

- § Direct steam preheating the product in the hot well has been replaced by the indirect method.
- § Tubular heaters are currently used in many plants. They eliminate dilution by the steam condensate and steam impurities that may be toxic and/or cause off-flavors.
- § Numerous optimum temperature-time conditions have been used in preheating whole milk. Commercial practice frequently employs the range of 82°C for 15 min to 93 °C for 3 min.
- § The heating, after concentration has been suggested as preferable possibly on the basis that a higher percentage of the antioxidants retained. These compounds may be removed along with the moisture during vacuum condensing.
- § When sufficient solids concentration as Baum'e degrees has been attained (35 to 45%), product is continuously removed from the evaporator.
- § Another variation in the procedure is to preheat skim milk to a high temperature necessary for formation of antioxidants and then to condense the skim milk. Homogenized, pasteurized cream is used to standardize the condensed skim milk to the desired ratio of milk fat to solids not fat. Some operators prefer to homogenize the concentrate after adding the cream.

- § The temperature of condensed milk after it is pumped from the evaporator is boosted to 62.8 to 73.9 ° C in a heat exchanger prior to the high pressure pump.
- § The condensed milk is dried with inlet air at 148 to 232 ° C and exit air at 74 to 93 ° C, depending upon drier characteristics. To reduce heat damage during dehydration, and yet obtain the desired moisture, a low exhaust air temperature is preferred.
- § The dry whole milk should be immediately removed from the hot air stream to maintain better "body characteristics", and keeping quality. The higher the temperature and the longer the time the product is above the melting point of the fat the greater the amount of free fat that results.
- § The use of refrigerated air to move the dry whole milk to a cyclone after it leaves the drying chamber is one system of decreasing the temperature. Cooling to room temperature (not below) is preferred. When ambient temperature and humidity are high, the air may be reheated just enough to avoid absorption of moisture by the product due to the dew point. However, other methods of cooling whole milk powder have proved to be satisfactory such as augering across a surface cooled by water or brine in jacketed equipment. The system must be sufficiently protected against the entrance of condensate from the ambient conditions.
- § A 12-mesh screen is used for sifting dry whole milk. The product should packaged immediately or held under vacuum for 7 to 10 days before gas packaging.

#### 35.3. FOAM SPRAY PROCESS

Milk is standardized, preheated, and homogenized similarly as for regular spray drying. During evaporation, the solids content may be increased to 50% or above. A gas, preferably nitrogen, is injected into the concentrated milk at 14 (or more) kg/cm<sup>2</sup> greater than the pump pressure. The gas is distributed into the concentrate by means of a mixing device between the high pressure pump and the nozzle. A regulator and needle valve control the flow of gas into the concentrate coming from the high pressure pump at 105 to 126 kg/cm<sup>2</sup>. The usage of gas is at the rate of 0.037 m<sup>3</sup> per liter of concentrated whole milk with 50% T.S.

Foam spray dried whole milk has improved dispersibility and approximately one-half the bulk density of the regular spray dried product. The occluded air in the particle causes poor sinkability when product is recombined with water.

## 35.4. DRUM PROCESS DRY WHOLE MILK

Only a small amount of drum dried whole milk has been processed in recent years. The poor keeping quality, unsatisfactory reconstitutability, and scorched flavor are considered to be the chief deterrents. However, for uses demanding a high percentage of free fat such as in certain confections, the drum dried product suffices.

Whole milk which has been standardized at the ratio of 3.5 (fat) to 8.75 (solids not fat) will yield a product with 28% fat if dried to 2 % moisture. The standardized milk is pasteurized and may or may not be homogenized. Unhomogenized milk will give a higher free fat in the dried form. The customary homogenization is 175 to 210 kg/cm<sup>2</sup> and the temperature is 62.5 to 76.5 <sup>0</sup>C. The preheat treatment is regulated for the intended use, but normally is desirable

#### **CONDENSED AND DRIED MILK**

only for dry milk intended for baking products. The severe heat treatment during drum drying negates any attempt to produce a low-heat product.

Concentration is not necessary, but will increase the drum drier capacity. Total solids increase should be limited to ~ 20% concentrate. The procedure and precautions for drum drying of whole milk are very similar to those presented for nonfat dry milk by this process.

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# **Technology of Dry Milks**

#### 36.1. INTRODUCTION

Milk is unique in its content of valuable nutrients. Both the chemical composition and the physical properties of the milk powder play an important role in its use with quick and complete reconstitution adding convenience to the products. Every day, millions of liters of milk are processed into dry products. By far the largest part ends up as ordinary skim milk and whole milk powders. These products are market commodities with little requirement for product functionality. Consumer demand for specific product properties, particularly in the food industry, has resulted in the development of many different dry dairy products, ranging from instant whole milk powder to speciality food ingredients. The dairy industry invests heavily in the development and production of such products.

During the last few decades there has been a growing market for powders which are instantly soluble in cold water. Ordinary non-agglomerated powders tend to lump when mixed with water, and if strong mechanical stirring is not applied, it may result in an inhomogeneous mixture which is not attractive to the consumer. The quality requirements to instant milk powders are constantly getting stricter as an increasing number of properties must be optimized simultaneously and controlled within still narrower limits in order to obtain a high quality product with the highest degree of uniformity, fulfilling the requirements set by costumers, organizations and industrial and/or legislative standards.

#### 36.2. INSTANTIZATION

The historic development started with the pioneering research of Mr. David D. Peebles in the beginning of the fifties, and instantized non-fat dry milk was marketed from 1954. Soon it replaced the regular spray dried products on the retail market. Lecithinizing during the drying in the fluid bed is not always applied; it is meant to obtain instant properties.

The principle of instantization is the agglomeration of individual spherical milk particles into clusters and the conversion of lactose from the glass into a microcrystalline form which makes the powder more wettable and less hygroscopic. In the instantization process, the surface of milk powder particles are humidified, or the particles are only partially dried during manufacturing, so that the surface is tacky and partial crystallization of lactose leading to formation of microcrystals occurs before the particles are redried. This produces a clustering of the particles in loose spongy aggregates of low density (known as agglomerates / conglomerates / granulates ) which flow freely and disperse readily in cold water, as water penetrates the spongy structure of these aggregates and allow them to sink and disperse.

The principal purpose of agglomeration, also called instantizing, is to improve the rate and completeness of the reconstitutability of dry milk products. Small single particles dissolve instantly in water. Powder consisting of small particles is, however, difficult to disperse. Big particles are easy to disperse in water, but dissolve only partially.

Agglomeration is a result of wet and/or semi-dry particle collision with size  $>125 \mu m$ . Control is achieved by returning dry fine powder to the wet spray during different stages of spray drying. Mastering the agglomeration techniques is the art of modern spray drying. Plant operation and economics are other important parameters.

## 36.3. PRODUCTS

In addition to nonfat dry milk, the instantizing process has been applied to dry whole milk and other milk fat containing dry dairy products with only limited improvement in reconstitutability resulting. The milk fat adversely affects wettability because of its hydrophobic property.

#### **36.4. MAJOR SYSTEMS**

The process may be carried out using either of these two methods:

- 1. Spray Drying Agglomeration
- 2. Rewet Agglomeration

## 36.4.1. SPRAY DRYING AGGLOMERATION

During the spray drying process, the aim is to produce particles with a big surface/mass ratio, i.e. small particles. The reconstitution in water of a powder consisting of small particles is however difficult and requires intensive mixing in order to disperse the powder, before it is totally dissolved. Bigger particles exhibit a better dispersion, but the solubility is negatively affected during the drying operation. By agglomeration both a good dispersion and a complete solution are obtained. In spray drying there are two ways of agglomeration: the spontaneous and the forced, both in a primary and secondary form as detailed in Table 36.1 and Figures 36.1 to 36.4.

Table-36.1: Types of Agglomeration process

Agglomeration	Definition	Examples	
Туре			
Spontaneous	Random, unprovoked collision of primary	All atomization	
Primary	spray particles	devices	
Forced Primary	Intended collision between primary spray	Collision of sprays	
	particles from different atomization devices	from different nozzles	
Spontaneous	Random, unprovoked collision of primary	Multi-stage Dryers	
Secondary	spray particles and fines by venturi effect		
Forced Secondary	Intended controllable collision between	Normal type when	
	primary spray particles and fines returned to	Fines Return is applied	
	the atomization zone		

Fig.36.1: Spontaneous primary agglomeration

Fig.36.2: Forced primary agglomeration

In Forced Secondary agglomeration t he small dry particles are introduced into the dryer near the atomizing device, where they will meet and collide with atomized wet droplets thus forming agglomerates consisting of many particles stuck together having a size of  $100\text{-}500~\mu$ , depending on the parameters selected.

In Forced Secondary agglomeration  ${\bf t}$  he small dry particles are introduced into the dryer near the atomizing device, where they will meet and collide with atomized wet droplets thus forming agglomerates consisting of many particles stuck together having a size of 100-500  $\mu$ , depending on the parameters selected.

Fig.36.3: Spontaneous secondary agglomeration

Fig.36.4: Forced secondary agglomeration

Due to the special air flow pattern in a Multi Stage Dryer, a considerable spontaneous, secondary agglomeration takes place. For production of high quality instant whole or skim milk powder this spontaneous agglomeration suffices, and the fines are just returned to the integrated fluid bed, from where they will get airborne again and reach the atomizing zone again. However, the agglomeration may be further enhanced by forced, primary agglomeration (collision of sprays overlapping each other from different nozzles in a multinozzle atomization unit) and/or by returning the fines to the atomization zone (forced, secondary agglomeration). Further flexibility can be gained by designing the atomization unit in a way that allows the distance between the single nozzles or between the nozzles and the fines return tube to be altered. Depending on the atomization device the fines return is designed in different ways as shown in Figures 36.5 to 36.7.

# 1. Rotary Atomization

Fig. 36.5: Fines return rotary atomizer "old type"

Fig. 36.6: Forced secondary fines return for rotary atomizer with Fines Return Air Disperser

Fig. 36.7: Forced secondary fines return for nozzle atomizer

In modern dryers fines are therefore introduced from above through the air disperser via four fines pipes situated just above the atomizer cloud. Deflector plates at the end of each fines pipe ensure a correct introduction and distribution of the fines. The unit operation of the whole process of agglomeration is illustrated in Table-36.2.

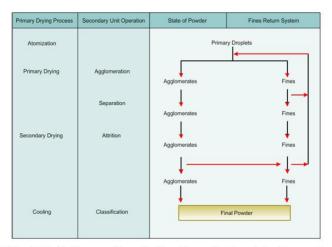


Table-36.2: Unit operations during Spray Drying / Agglomeration

#### 2. Nozzle Atomization

The fines return is an integral part of the nozzle unit with the fines duct in the centre surrounded by nozzles at the periphery. The fines are introduced tangentially into the fines distribution duct or through a center pipe. The nozzles can be welded to the nozzle rod at a certain angle, so that by turning the nozzle rod around its axis the collision point can be altered.

#### 3. Separation

It is the process of separating the part of fines which is entrained in the main drying air leaving the drying chamber. The efficiency of separation is determined by the air flow pattern and air velocities in the drying chamber.

#### 4. Attrition

It is defined as the partial break down of agglomerates in fluid beds or powder conveying systems resulting in creation of either fines, smaller agglomerates (abrasion) or of a number of smaller sized particles (fragmentation). Factors affecting the extent of attrition are the jet velocity, determined by the pressure difference across the perforated plate, the fluidization velocity and the actual design of the perforated plate.

## Classification

It is defined as the separation of fines in fluid beds. The efficiency of classification is mainly determined by the fluidization air velocity and also the fluid bed design.

After the final drying, the powder enters the cooling section where the powder is cooled by means of air at ambient temperature followed by cooled, dehumidified air. The powder is finally passed over a sifter where any oversize particles are removed. It is also possible to install a sifter with two nets thus removing any remaining particles/agglomerates of small

diameter. Together with the fines, this fraction may be returned to the atomizing device thus producing a powder with a well defined agglomerate size distribution.

The agglomeration is improved by:

- "High solids content in the concentrate
- "Bigger quantity of fines returned to the atomizing device
- "Fines introduction closer to the atomizing device
- "Shorter distance from nozzle to fluidized layer in a static fluid bed
- "Higher moisture content from the primary drying stage
- "Bigger primary particles
- "Lower pasteurization temperature of the milk prior to the evaporation

When leaving the sifter, the powder should not be exposed to strong mechanical conveying, neither by means of air nor by fast moving mechanical screws. However, today's lenient vacuum-low speed air systems are used without too much damage to the agglomerates. The best thing, however, is to install the plant so high that filling into bags or bins is possible by gravity.

## **36.2.2.2.** Rewet agglomeration

Since many powders may become instant by the mere agglomeration, many processes have been developed during the past years to agglomerate ordinary powders consisting of single spray particles usually produced in plants with pneumatic conveying system.

The major systems of agglomeration are the Peebles (Fig.36.8.), Cherry-Burrell, Blaw-Knox and Niro Agglomerator (Fig-36.9). Each varies in equipment and operation details. The general features in common are:

- 1. Wetting of surface of the particles with steam, atomized water, or a mixture of both.
- 2. Agglomeration, whereby the particles collide due to the turbulence and adhere to each other forming clusters.
- 3. Redrying with hot air.
- 4. Cooling and

Sizing to eliminate the very large agglomerates and the very small particles.

#### Fig. 36.8: The Peebles Rewet Instantizer

Fig. 36.9: Niro Multi-purpose Rewet Agglomeration plant

#### 1. Wetting

Wetting of the surface of the particles is done with humid air, steam, atomized water either pure or containing milk solids, sugar or other soluble components. The water may further contain additives such as vitamins (water soluble), minerals, colour and surface-active agents. The atomization of the moistening agent can be carried out by means of nozzles or a rotary atomizer. If a rotary atomizer with two feed pipes is used, it is possible to use a combination of steam and water or use two moistening agents, which cannot be mixed for various reasons. If the product is insoluble, an adhesive can be dissolved in the moistening agent. When doing so products otherwise impossible to agglomerate can be agglomerated with a good result.

## 2. Agglomeration

Agglomeration, whereby the moist sticky particles collide due to the turbulence and adhere to each other forming agglomerates, is essential for the rewet process. As powders with different compositions do not behave in the same way during the rewetting and agglomeration process, different equipment is needed to obtain an optimal agglomeration. In principle there are two ways of performing the agglomeration:

"Droplet agglomeration

"Surface agglomeration

# **Droplet Agglomeration**

In the droplet agglomeration process, the powder particles are wetted with droplets of liquid atomized by means of a nozzle or a rotary atomizer while suspended in air. The powder may either be introduced around the rotary atomizer or the nozzle by means of gravity or pressure air conveying, or from below by means of pressure conveying.

The droplet agglomeration process is especially used for powders containing fat such as whole milk powder and powders with a high content of sugar such as cocoa-milk-sugar mixtures. If cold-water instant whole milk powder is produced, a lecithin dosing equipment is installed between two Vibro-Fluidizers. However, the final product quality will never be as good as that produced on a drying plant equipped with fines return, fluid beds and lecithin dosing equipment.

## **Surface Agglomeration**

In the surface agglomeration, either steam or warm moist air with a high relative humidity is used as the moistening agent. The surface of the individual dry particles is wetted due to condensation of the water vapour on the colder particles, whereby the stickiness required for the agglomeration is created. The subsequent agglomeration will take place, if the particles are exposed to sufficient mechanical impact. The warm humid air is usually made by spraying steam into warm air at a given temperature to obtain a relative humidity of 100%.

The surface agglomeration is mainly used for skim milk powder when large agglomerates are aimed at. The final product properties depend to a great extent on the raw material used for the rewetting, and Table-36.3. below shows specification for a recommendable basis powder:

Table 36.3: Specifications of basis powder for agglomeration

Bulk density, tapped 1250x	0.80 g/cm <sup>3</sup>
WPNI	2-3 mg/g powder *
Insolubility index	< 0.1 ml
Particle density	1.35 g/cm <sup>3</sup>
Mean particle size	$\sim 50~\mu m$
Amount above 100 μm	max. 25%

<sup>\*</sup> corresponding to a pasteurization temperature of ≈90°C prior to evaporation.

The surface agglomeration can also be used for whole milk powder, but the agglomerates get too compact for obtaining a powder with good rehydrating properties.

## 1. Redrying

As the basis powder used for agglomeration is remoistened to obtain the desired surface characteristic for an optimal stickiness, this additional moisture has to be evaporated again in order to reach the specified moisture content. The agglomerates may break down again, if they are exposed to extensive mechanical handling, such as in a pneumatic conveying system. It is therefore necessary to perform the drying in a Vibro-Fluidizer.

## 2. Cooling

Like the redrying, the cooling is performed best in a Vibro-Fluidizer.

#### 3. Sizing

Usually, there is a well defined requirement to the agglomerate size distribution of the final powder. It is therefore necessary to sift the powder. This is done in a sieve with two different net sizes placed above each other. Thus it is possible to remove any agglomerates/lumps considered to be too big in the final product. This oversize fraction may be milled and returned to the process. Powder passing through the upper net may be further fractionated on the lower net into a main fraction and a fines fraction consisting of single particles and agglomerates being too small. This fines fraction is together with the cyclone fraction from the Vibro-Fluidizer recycled back to the process.

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# **Factors Affecting Instantizing**

#### 37.1. INTRODUCTION

Dry milk manufactured specifically for agglomeration usually gives the best results. The success of the instantizing operation depends upon adequate control of each step during manufacture.

#### 37.2. FACTORS AFFECTING

- 1. Moisture content and particle size should be as uniform as possible.
- 2. A minimum of fine particles, less than  $20\mu$  in diameter is desired with the preferred particle range of  $25\mu$  to  $50\mu$ .
- 3. Nonfat dry milk for agglomeration should be low in fat content.
- 4. Low heat (6 mg or more of WPN) or medium heat nonfat dry milk (less than 6 but more than 1.5 mg WPN) is normally used.
- 5. High-heat nonfat dry milk will agglomerate satisfactorily, but it shatters much more easily in handling after agglomerating and redrying.
- 6. The powder distribution into the wetting zone space must be uniform and at a constant rate.
- 7. Moisture condition must be uniform in all respects to avoid over or under wetting of particles.
- 8. Over wetted particles dissolve slowly and too little wetting permits excessive shattering during handling.
- 9. The air movement has to be stabilized to assure optimum particle collision. Excessive movement causes product adherence to the equipment lining.
- 10. Control of the redrying air temperature and its flow rate is necessary for adequate moisture removal without heat damage to the agglomerated product.

It is the well known fact that agglomeration lowers the density of dry milks. Usually flavor is not affected, but changes in flavour can occur as a result of agglomeration that is detrimental if the process is not carefully controlled.

#### 37.3. PROCESS PARAMETERS

1. The plant is operated such that the powder leaves the primary drying stage with 2-10% higher moisture than wanted in the final product. The cyclone fraction is returned to the

atomizing device, where the dry fine particles will collide with the primary particles thus forming agglomerates.

- 2. The powder leaving the chamber is therefore warm, moist and consists of stable agglomerates. Consequently by gentle after-drying performed in integrated fluid beds and/or a Vibro-Fluidizer the agglomerated product structures are maintained. The cooling should always be done in a fluid bed.
- 3. The powder obtained by this process can be characterized by:
- § Agglomerated product structure
- § Non-dusty
- § Lower bulk density than for powder from the pneumatic plant
- § Good flowability
- 4. The decreased drying air outlet temperature and consequently lower product temperature will result in:
- § Improved solubility because of less thermal damage
- § Low content of occluded air, because in the critical stage of the drying, with a water content of 30-10%, the blowing-up of the particles is avoided.

## 37.4. Instant Skim Milk Powder

In order to obtain a skim milk powder with good instant properties, the agglomeration plays the most important part. In order to avoid too quick wetting of the particles, the powder particles are agglomerated thus reducing the specific surface. The specific surface can also be reduced by bigger primary particles, however, with a risk of a high insolubility index. Furthermore, the instant properties, especially the wettability, are improved, if the agglomerates are so compact that the moisture absorption and dissolving process are prolonged enabling a dispersion of the powder agglomerates in the water, after which the final dissolution can take place. The powder should also have a good insolubility index.

The agglomeration is improved by reduction of the pasteurization temperature before evaporation, keeping h igher solids content in the concentrate and bigger primary particles. If recycled amount of fines are higher, introduce fines closer to the wheel or nozzle and i ncrease moisture content in the powder from the drying chamber

Typical operation conditions for the spray dryer when producing a first class instant skim milk powder, depending on type of dryer, will be having drying temperature  $200^{\circ}\text{C} \pm 20^{\circ}\text{C}$  and s olids content in the concentrate 48-50%.

## 37.5. Instant Whole Milk Powder

In whole milk powders, some of the fat is present as free fat. Free fat rejects water making it impossible to dissolve these powders properly in cold water. Hence, i n the case of whole

milk powder it is required that the water temperature is >40°C. Homogenization of the whole milk concentrate prior to drying reduces the content of free fat in the final powder. However, to be called 'instant' whole milk powder, it must be agglomerated (Figure 37.1) as well as have a surface-active agent (lecithin) applied to improve water affinity. The product then becomes instant – even in cold water.

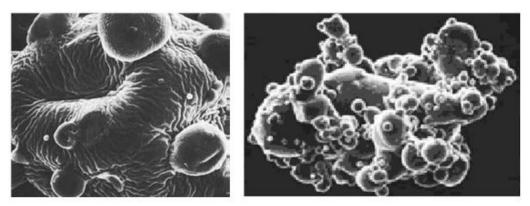


Fig.37.1: Microphoto of agglomerated whole milk powder

For this purpose lecithin (originating from soy beans) dissolved in pure butter-oil, in order to make a liquid, may be used. Lecithin is superior as to the functional performance, i.e. achieving of instant properties. The butter-oil is chosen also in order to use a natural milk component, as using a vegetable fat, even it is done in many cases, could be considered a falsification. The amount of lecithin and of the total free fat (i.e. original free fat + added butter-oil + lecithin) in the final powder may vary from 0.1-0.3% and 1-2%, respectively. However, variations within these limits result in rather big differences as to the desired properties.

A high amount of total free fat together with high amount of lecithin improves the wettability, but on the other hand it is affecting the flowability and may seriously affect the dispersibility. At lecithin levels >0.5% it is possible to detect the characteristic soy flavour. The structure of the powder and the degree of agglomeration are of importance too, as poorly agglomerated powders require higher amount of wetting agent than well agglomerated products.

## **Two-stage process:**

- 1. In the two-stage process the basic powder is collected for intermediate storage. It is important to prevent any damage to the powder by mechanical treatment. The intermediate storage is therefore preferably accomplished in bins or similar containers of 1-2 m<sup>3</sup>.
- 2. The basic powder is then transferred from the bins into the supply silo and is metered into the first Vibro-Fluidizer by means of a dosing screw. The powder is heated and at the same time any fines are blown off.
- 3. The lecithin dosing equipment consists of two vessels, dosing pump, powder trap with two-fluid nozzle, and control panel. The first vessel serves for the preparation of wetting agent, and the second one as supply vessel, from where the wetting agent is metered to the two-fluid nozzle for spraying on to the powder.

- 4. The flows, temperatures and pressures of the wetting agent and of the atomizing air are recorded. Interlocking in the control panel ensures that the flow of powder will stop automatically, if for some reason no lecithin is applied. Consequently no powder will leave the plant without a proper lecithin coating. The second Vibro-Fluidizer, also supplied with warm air, ensures a gentle but proper mixing of the powder to obtain a uniform distribution of the lecithin mix over the particle surface.
- 5. The powder leaving the lecithination unit is packed into retail packages. The filling machine is placed preferably directly below the lecithination unit with a hopper for short intermediate storage to avoid any unnecessary transport.

#### **Continuous Process:**

The powder production and lecithination can be made in one continuous process. In this case the powder trap with lecithin nozzle is placed between the integrated fluid bed and the Vibro-Fluidizer. The product quality can be compared with the one achieved by the split process operation described above.

#### **Advantages of Split Process:**

- 1. Retail packing of milk powder is never a fully continuous operation, since there is always a natural break between the powder production and the packing. During this break the powder must be stored in bulk for one to several days, preferably in bins to avoid damage.
- 2. For quality reasons it is better to store unlecithinated powder. The lecithination process therefore fits best as a part of the packing line forming one continuous operation.
- 3. The intermediate storage of the powder after production makes it possible to analyze the product to classify it and to calculate the composition and quantity of wetting agent in order to achieve the desired properties.
- 4. Fines created during storage and transport can be blown off in the first Vibro-Fluidizer of the lecithination unit.

Today, however, most instant whole milk is produced in plants equipped with a lecithin dosing equipment, placed between the integrated fluid bed and the Vibro-Fluidizer, in one processing step. The final powder is conveyed to silos by lenient low speed vacuum conveying systems - being very gentle to the agglomerated product - before packing either in retail packs or 25 kg bagging lines. The conveying lines may be equipped with pre-gassing by  $N_2/CO_2$  for prolonging the shelf life.

## 37.3. PACKAGING

## 37.3.1. Packaging of Non Fat Dry Milk

§ A suitable container for dry milk should be impervious to moisture, light, gases and insects; should be durable for handling, resistant to corrosion, of low cost; and be relatively easy to fill, seal, handle, and empty. The retail package should have a reclosable opening.

- § Nonfat dry milk for industrial use and storage may be packaged in barrels, drums, and bags or for retail purpose in metal cans, glass jars, or cartons. A polyethylene liner having 3 mm thickness inside a 6 ply Kraft paper bag is recommended. The non fat dry milk in bags having a tape over the top seal is helping to prevent insect infestation of the product after packaging.
- § Nonfat dry milk in commercial trade is commonly packaged in a 2 mil polyethylene bag inside a 4-ply Kraft paper bag. The outside layer is usually plain, but a wrinkled type is also available. Freezing, high temperature, and low humidity during storage of bags cause them to become brittle and thus damage more readily in handling.
- § Manual filling of the bags is most commonly completed by means of a simple device attached to the sifter. Automatic bagging equipment is readily available to dispense the correct weight of powder in one bag before shifting the product flow into the next bag. Bags are sewn automatically or a manually operated sewing machine suspended and counterbalanced within easy reach of the filling area. After closing, the bags are usually stacked on pallets. The bag overhang from the pallet should not be more than 5 cm.

#### 37.3.2. Retail Carton

- § Cartons of fiberboard, foil, and plastics have largely supplanted glass and metal as retail containers.
- § Because of the hygroscopic nature of dry milk, the packaging materials must provide a good vapor barrier.
- § Packaging of nonfat dry milk is quite a routine practice and the principal concerns involve keeping machine downtime to a minimum maintaining the correct net weight within narrow limits, and providing a good seal.
- § Coding of each package provides a means of identification for quality control.

## 37.3.3. Packaging Dry Whole Milk

- § Average production conditions and normal market periods for export and retail sales necessitate gas packaging of dry whole milk to delay oxidative changes.
- § The rancid flavor deterioration of dry whole milk due to oxidation necessitates inhibitory measures.
- § One of these consists of packaging the product with low oxygen content.
- § The general procedure is to immediately remove oxygen by subjecting the product to vacuum within 24 hr of drying with final packaging within a few days.
- § If gas flushing is not done, the powder may be packaged into multilayer paper bags with a polyethylene inner layer. Whole milk powder, however, is often packaged in tins or in plastic containers to minimize oxygen uptake.

## 37.3.4. Gas packaging

- § The so-called gas flushing, essentially displacing air by  $N_2$  or a mixture of  $N_2$  and  $CO_2$ , is to remove a considerable part of the oxygen and thereby to improve the stability toward autoxidation; it can be done once or twice.
- § To obtain a low level of headspace oxygen in dry whole milk, a double gassing technique is applied. The customary procedure is the collection of filled cans on trays to be conveyed into the vacuum chamber. The air is removed rapidly (60 sec.) with the gauge indicator decreasing to 73.6 cm of vacuum. After a 2 to 5 min hold, the pressure is restored with nitrogen to 0.035 to 0.07 kg/cm<sup>2</sup> above atmospheric pressure. Nitrogen may be replaced with a mixture of nitrogen and carbon dioxide, the latter being restricted to 5 to 20%.
- § After removal from the chamber, the containers are sealed by soldering the 1 to 2 mm hole in the lid or crimping on the lid. The containers are held for oxygen desorption. When an oxygen equilibrium has been attained in the headspace, usually within a week but at the most ten days, the cans are punctured and the vacuum treatment, pressure restored with nitrogen, and sealing step's are repeated.
- § Gas packaging of dry whole milk should not be delayed after drying. Otherwise quality deteriorates during the holding period. Warm powder directly from the drier tends to have a more rapid rate of oxygen desorption under vacuum. If the production is not large, dry whole milk may be placed into metal drums and air exhausted. By holding the product under partial vacuum for oxygen desorption the first gassing step in the package may be eliminated, and yet the final maximum of 2% oxygen can be attained.
- § In the recent times, commercial plants employ continuous packaging machines which completes the work of clinching the embossed bottom on the filled, inverted tins, create vacuum in the head space, flush with the inert gas and finally seaming the tins in one run. Multilayer packages are also being used for the purpose.

## a) Oxygen Limits

A good commercial operation using continuous gassing equipment can reduce the oxygen level to  $\sim 2.5$  % with a single gassing. This may be satisfactory for many storage conditions, but not 32  $^{0}$ C or above. A maximum limit is 3.0 to 3.5 % oxygen in the headspace of the can for a noticeable delay of oxidation.

#### b) Oxygen Removal by Reaction

The procedure consists of the addition of 5% hydrogen to the nitrogen used to restore atmospheric pressure after de-aeration. A packet containing a catalyst is added to the container after filling with the dry milk. The catalyst may be palladium or platinum. Each of these causes the oxygen to react with the hydrogen forming water which cannot pass back through the pouch wall into the dry product, thus effectively removing the oxygen available for oxidative reactions.

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## **Module 14**

## Physico-chemical changes taking place during manufacture of dried milks

## Lesson-38

## The Milk Powder System

#### 38.1. INTRODUCTION

The milk powder manufactured undergo several changes while conversion from liquid milk to dry product. They are:

- § The manufacturing process employed on milk ( $\sim$ 87% water,  $a_w$ =0.99) eliminates majority of water till it reaches the final stage of drying, the powder ( $\sim$ 4% moisture,  $a_w$ <0.6). The process not only eliminates the water from milk but also changes the physical structure i.e. the arrangement of the components of milk in space. In the process, the sol changes to a dust.
- § The dried milk exhibits a well pronounced dual physical structure the primary structure, which is the internal build-up of the powder particles from the milk solids and small amounts of moisture and air and the secondary structure which represents a typical powder, a system of closely packed solid particles in a gas.
- § The structural elements of dried milk particles are lactose, either amorphous or partly crystalline, casein micelles and whey protein particles, fat in globular or non-globular (free fat) forms and air as spherical cells. The fat, protein and air are presumably dispersed in continuous phase of amorphous lactose.
- § The whole milk and infant milk powders are rich in fat (20-37%) and hence need protection against oxygen and moisture, therefore, generally tinplate cans are used and the packaging is done under nitrogen cover to replace air with nitrogen in the tins. Skim milk powders have very low fat (1.5%) and as such a good moisture barrier material like multi wall paper sacks with one polythene liner is used. Recently, some skim milk powder has also been sold on polythene bags and even high density polythene bottles are being tried for retail pac ks.
- § The milk powders are concentrated mass of milk components in the form of particles with small amount of moisture and air. If sufficient care is not taken to protect them from high humidity, high temperature of storage and the entry of air, the deterioration processes are accelerated.

These changes while manufacture occurs in a sequence. They are discussed here:

## 38.2. CHANGES OF STATE OF THE DRYING DROPLETS

§ Atomizing pure water in a drying chamber in the usual way causes the water droplets to reach the wet-bulb temperature and to vaporize within 0.1s at this temperature.

- § The presence of dry matter in the droplets, however, makes an enormous difference. The diffusion coefficient of water decrease substantially with increasing dry-matter content. Accordingly, the vaporization is significantly slowed down.
- § In the drying droplet, temperature equalization occurs in less than about 10 ms. i.e. the temperature is virtually constant throughout a droplet during drying.

#### 38.3. DRYING STAGES

- § Initially, the droplet has a very high velocity relative to the drying air.
- § Therefore, there is a first stage during which circulation of liquid in the droplet occurs; this circulation greatly enhances transfer of heat and mass.
- $\S$  For a droplet of 50  $\mu$  m diameter, this stage lasts for ~2 ms. In this time, the droplet covers a distance of about 10 cm and loses a small percentage of its water.
- § Its velocity compared to the air decreases to the extent that the formed surface tension gradient of the drop surface arrests internal circulation of liquid.
- § But in the second drying stage, the difference in velocity between drop and air is still great enough to accelerate water transport.
- § The transport in the droplet occurs by diffusion but in the air by convection.
- § After about 25 ms the relative velocity of the droplet has decreased so far that the water transport has become essentially equal to that from a stationary droplet.
- § Relative to the air, the droplet then has covered a distance of a few decimeters and has lost about 30% of its original water.
- § In the third stage, lasting at least a few seconds, the droplet loses the rest of the water by diffusion.

#### 38.4. DRYING PROCESS

- § During drying the air temperature decreases and the humidity of the drying air increases. Moreover, the droplets vary in size and the smallest ones will dry fastest.
- § In practice, the mixing is always between the two extremes. For driers with a spinning disk, the situation tends to be fairly close to perfect mixing. In most driers with nozzles, it tends to be closer to cocurrent flows. In all situations, the drying time may very by a factor of, say, 100 between the smallest and the largest drops. This is of great importance for fouling of the drying chamber; the largest drops have the greatest chance of hitting the wall and of being insufficiently dry to prevent sticking to the wall.
- § Another factor that affects drying rate is the presence of vacuoles in the drying drops. It leads to significantly faster drying.

#### 38.4.1. CONCENTRATION GRADIENTS

A rapid decrease in drying rate of the droplets occurs after the water content is reduced to, say, 15%. A strong concentration gradient forms rapidly during drying. The higher the drying temperature, the stronger the effect. Therefore, stronger gradients occur for cocurrent drying. A dry outer layer, i.e., a kind of rind, is formed and because of this, the water transport is slowed down considerably. The temperature can rise significantly in the dry outer layer because a dried material assumes the air temperature, not the wet-bulb temperature. In other words, the decrease in temperature near the surface of the droplet caused by consumption of the heat of vaporization becomes far smaller because the vaporization of water is slower. Because temperature equalization happens very quickly, the whole drying droplet increases in temperature.

The concentration gradients are not of a lasting nature. The relatively dry outer layer of the droplet soon becomes firm and eventually glassy. This causes the droplet to resist further shrinkage. The droplet can react by forming vacuoles or by becoming dimpled. Especially at a low water content of the particles, so-called hair cracks may be formed.

#### 38.4.2. AROMA RETENTION

Besides water, the drying droplets lose other volatile components, including flavor compounds (aroma). In many cases, however, the loss of flavor compounds is far less than expected, in spite of their volatility because of the following conditions:

- § The effective diffusion coefficient of most flavor components in the relatively dry outer layer of the droplet decreases far stronger with decreasing water content than the diffusion coefficient of water does due to the greater molar mass.
- § Thus, the aroma retention retaining flavor compounds during drying increases with
- o droplet size because in larger droplets, the outer layer from which the flavor components get lost has a relatively smaller volume and
- o Drying temperature because, at a higher temperature a solid rind forms more rapidly.
- § Formation of vacuoles diminishes aroma retention, especially if hair cracks develop in the particles and the vacuoles come into contact with the surrounding air.
- § Dissolved milk powder often has a cooked flavor, which results from the flavor compounds formed during preheating and possibly during evaporation.
- § During drying, conditions are mostly not such that off-flavors are induced. On the contrary, a considerable part of the volatile sulfhydryl compounds is removed. A cooked flavor mainly results from methyl ketones and lactones formed by heating of the fat and from Maillard products which are almost absent in skim milk powder.

## 38.4.3. WATER ACTIVITY

If the water content of a product decreases, its water activity ( $a_w$ ) also decreases. Water activity is expressed as a fraction. In pure water  $a_w = 1$ ; in a system without water,  $a_w = 0$ .

Different dairy products have following values of a<sub>w</sub> as indicated in Table 38.1:

Table 38.1: Water activity values of different dairy products

No.	Milk Product	$\mathbf{a}_{\mathrm{w}}$
1	Milk	0.995
2	Evaporated Milk	0.986
3	Ice Cream Mix	0.97
4	Sweetened Condensed Milk	0.83
5	Skim Milk Powder, 4.5% water	0.2
6	Skim Milk Powder, 3.0% water	0.1
7	Skim Milk Powder, 1.5% water	0.02
8	Cheese	0.94-0.98

Due to the very low water activity in milk powders, it exhibits following changes:

1. The hygroscopicity increases: Usually, a (dry) product is called hygroscopic if a small increase of a w causes a considerable increase in water content. Obviously, this mainly concerns milk powder with very low water content.

Diffusion coefficients decrease: At low water content the effect is very strong. The diffusion coefficient of water decreases from approximately  $10^{-9}\,\text{m}^2$ . s  $^{-1}$  in milk to  $10^{-16}\,\text{m}^2$ . s  $^{-1}$  in skim milk powder with a small percentage of water.

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#### Lesson-39

#### **WPN Index and Heat Denaturation**

#### 39.1. INTRODUCTION

Heat treatment of the original product or the concentrate can cause denaturation of serum proteins; the conditions during spray drying are rarely such as to cause extensive heat denaturation. The extent of denaturation is an important quality mark in relation to the use of milk powder. For instance, if the powder is to be used in cheese making, practically no serum protein should have been denatured in view of the rennetability; in infant formulas, on the other hand, the rennetability should be poor.

#### 39.2. WPNI

The extent of the denaturation of serum protein can be used as a measure of the heating intensity applied. This is true also where denaturation by itself may be of no importance, but other changes associated with intense heat treatment are. An example is the flavour of a powder to be used in beverage milk, which requires a mild heat treatment. An intensive heat treatment is needed for some other uses, for instance, to acquire good stability against heat coagulation in the manufacture of recombined evaporated milk, or a high viscosity of the final product when making yogurt from reconstituted milk. It is also desired if milk powder is used in milk chocolate; presumably, Maillard products contribute to its flavor.

The *whey protein nitrogen index* (WPN index) is generally used to classify milk powders according to the intensity of the heat treatment(s) applied during manufacture. To that end, the amount of denaturable serum protein left in the reconstituted product is estimated, usually by making acid whey and determining the quantity of protein that precipitates on heating the whey. This can be done by Kjeldahl analysis of protein nitrogen or by means of a much easier turbidity test that is calibrated on the Kjeldahl method. The result is expressed as the quantity of undenatured serum protein per gram of skim milk powder. The classification is shown in Table 39.1 as follows:

Table -39.1: Whey protein nitrogen indices for heat classification of milk powders

Low-heat: WPN  $\geq$  6 mg N per gramMedium-heat: 6 > WPN > 1.5 mg N per gramHigh-heat: WPN  $\leq$ 1.5 mg N per gram

## 39.3. Damage Caused by Heating

High drying temperatures can result in undesirable changes in the dried product. Generally, it is only after the powder has been dissolved again that the changes involved are noticed. Three quite different categories of undesirable changes can be distinguished:

## 39.4.1. Heat denaturation and killing of microbes:

- a. The reaction rate is highly dependent on temperature, but the reaction is much slower and less dependent on temperature at low water content.
- b. Some results for the inactivation of phosphatase are showing that, a higher average drying temperature and the higher dry-matter content of the liquid gives more inactivation because this goes along with a higher viscosity and hence, on average, larger drops. Therefore, a longer heating time is needed at a dry-matter content in which the inactivation rate still is appreciable. However, the largest drops contain the greatest amount of material.
- c. Killing of bacteria is higher as the average drying temperature is higher. The initial increase of survival of bacteria with increasing dry-matter content is presumably due to a substantial decrease in heat sensitivity of the organism.
- d. Heat denaturation of globular proteins and consequently, inactivation of enzymes and killing of microorganisms greatly depend on water content. It may also occur that removal of water increases the concentration of a reactant or catalyst for heat inactivation; this is presumably the case for chymosin in whey, because at, say, 40% dry matter,  $a_{\rm w}$  and diffusivity are not greatly lowered.
- e. When spray-drying a starter culture, survival of bacteria is of paramount importance. Often, a relatively large proportion of an inert material, generally maltodextrin, is added to the liquid before drying, which lowers the temperature sensitivity of the bacteria. In this way, survival rates over 80% can be achieved.

## **39.4.2. Insolubility** :

- a. Part of the protein may be rendered insoluble during the drying process due to heat coagulation.
- b. The powder contains particles that do not dissolve in water, but the amount is a tiny fraction of the powder.
- Figure 39.1 shows that insolubility increases with increasing outlet temperature and increasing dry-matter content of the milk. It also greatly depends on drier design. Presumably, heat coagulation mainly occurs in some (large) drops or powder particles that recirculate in the drier and become wetted again. A cumulation of high temperatures and high dry matter content for a relatively long time then causes the problem. Modern driers tend to give very small insolubility figures.
- **Fig.39.1**: Effect of (1) The dry-matter content of the concentrate during atomization and of (2) The air outlet temperature, on the insolubility index (ADMI method) of the resulting skim milk powder. (3) Effect of outlet temperature on the percentage of the fat extracted from the resulting whole milk powder. (Walstra *et al.*, 2006)

## 39.4.3. Formation of hair cracks:

- **a.** These can form at high drying temperatures because the outer rind of a drying droplet soon reaches a glassy state; the pressure gradients developing in the particle then cause these very thin cracks to form.
- **b.** In case of whole milk powder, part of the fat can now be extracted from the powder by solvents like chloroform or light petroleum. The extractable fat is often called free fat, but that is a misleading term.

## 39.2.3. Autoxidation of lipids :

Autoxidation of lipids follows quite a different pattern (Figure 39.3, curve 4). The reaction rate is high for low  $a_{\rm w}$ . Possible causes are that water lowers the lifetime of free radicals, slows down the decomposition of hydroperoxides, and lowers the catalytic activity of metal ions, such as  ${\rm Cu}^{2+}$ . The rate of many reactions greatly depends on the water content; an example is given in Figure 39.2.

**FIGURE 39.2:** Rate of Maillard reactions (—) and of protein becoming insoluble (---) in concentrated skim milk at high temperature ( $^{\sim}80~^{\circ}$  C) as a function of water content. (Walstra *et al.*, 2006)

Because of an increase in the concentration of reactants, the rate of bimolecular reactions at first increases due to removal of water. On further increase of  $Q^*$ , the reaction rate often decreases again; this decrease would be caused by reduced diffusivity. A good example is the Maillard reaction (Figure 39.3, curve 5). The irreversible loss of solubility of milk protein in milk powders, and the rate of gelation of concentrated milks show a trend similar to curve 5. On removal of water from milk, it thus is advisable to pass the level of approximately 10% water in the product as quickly as possible.

**FIGURE 39.3:** Relative reaction rate (Kr) of various reactions plotted against the water activity ( $a_w$ ) of (concentrated) skim milk (powder). The upper abscissa scale gives the water content (% w/w). (1) Growth of *Staphylococcus aureus*; (2) oxidative degradation of ascorbic acid; (3) enzyme action (e.g., lipase); (4) lipid autoxidation; (5) Maillard reaction (non-ezymatic browning). (Walstra *et al.*, 2006)

Thus, the influence of some process variables on product properties is inevitably complex as the relationship between two parameters may depend on other variables and also it is mostly not possible to vary only one process variable. However, it is observed that a two-stage drying provides increased possibilities to make powder with various desirable characteristics. The major process variables and properties of the dried product affected are listed hereunder in Table 39.2.

Table 39.2: Major process variables and properties of the dried products influenced

Duadust nuonauties influenced
Product properties influenced
Moisture content
Particle size
Vacuole volume
Packed volume
WPN Index
Insolubility

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## **Module 15**

## Physical properties of dried milks

## Lesson-40

#### **40.1. INTRODUCTION**

Good-quality dried milk should flow readily, be free from lumps or caking and be uniformly white or light cream in colour. **Related Documents:** Several properties of powdered products affect the quality and the suitability of the powder in specific applications. The physical properties of the powder may confer different properties upon powders of identical chemical composition.

A milk powder particle generally consists of a continuous mass of amorphous lactose and other low-molar-mass components in which fat globules, casein micelles, and serum protein molecules are embedded. The lactose generally is in a glassy state, the time available for its crystallization being too short. If, however, precrystallization has taken place, large lactose crystals may be present. When precrystallized whey powder is examined microscopically, most of the particles look more like lactose crystals of the tomahawk shape to which other material adheres. If lactose has been allowed to crystallize afterward due to water absorption, its crystals are generally small (about 1  $\mu$  m).

#### **40.2. ROLLER-DRIED MILK POWDER**

It consists of solid, irregular masses containing little enclosed air. The fat globule structure is broken by contact of the milk with the very hot rollers with the pressure of the scraper knives, so that free fat becomes scattered throughout the powder - some of it on the surface and is readily extracted by solvents. Roller-dried milk looks completely different from spray powder in the microscope. It consists of fair-sized flakes. Due to the intense heat treatment during the drying it has a brownish color, a strong cooked flavor, and the availability of lysine has been considerably reduced, by 20 to 50%.

#### **40.3. FREEZE-DRIED MILK**

It consists of coarse, irregularly shaped, and very voluminous powder particles, which dissolve readily and completely. However, the fat globules show considerable coalescence, unless intense homogenization has been applied. In most cases, damage due to heat treatment is minimal.

#### **40.4. SPRAY DRIED MILK POWDER**

#### 40.4.1. Particles Shape:

Spray dried milk are found to be round with different surface structures, as revealed by electron microscopy. Some particle may be smooth, but most of them are severely wrinkled with deep surface folds and having "apple-like structure" caused by an implosion during the

last stage of drying process or during the cooling of particles. The deep surface folds are formed due to the presence of casein in the spray dried material.

The air may be present as a single cell or as a number of small bubbles, and usually occupies 15% to 25% of the volume of the particle. In addition, there are always some solid particles, the proportion varying considerably (10 to 70 % of the total) between different atomizers. The air cell volume may influence the keeping quality of the powder.

The body of the particle of whole milk powder is usually porous, whereas in case of skim milk powder the body in most particles is compact. Particles prepared from unhomogenized concentrated whole milk are more porous than particles made from homogenized concentrated milk. Similarly, small particles are more porous than large particles. High porosity is usually associated with the occurrence of cracks and capillaries in the particles.

Structure of agglomerated skim milk powder ranges from smooth comprising of fused primary globular particles with very small rosettes like lactose crystals on the surface to having rough surfaces covered with relatively large lactose crystals. Interior of these particles is found to be hollow and the crust is compact. The surface of the instant milk particles is very fragile and needs careful handling to avoid shattering and dust formation.

#### 40.4.2. Particle size distribution

Particle size is of importance for the reconstitution of powder, its flowability and appearance. Particle size distributions of milk powder are usually between 20 and 60  $\mu$  m, and the distribution is relatively wide. The particles in agglomerated powder are much larger, up to 1 mm, and are irregular in shape. Such a powder usually contains very few separate particles smaller than 10  $\mu$  m. Within one sample of nonagglomerated powder the larger-sized particles have on average a higher vacuole volume, partly because a drying droplet shrinks more strongly if it contains no vacuoles. The particle size distribution of a milk powder depends on a number of factors in the production process. These are:

- (a) Speed of rotation or pressure applied
- (b) Feed rate of the concentrate
- (c) Velocity of concentrate through the orifice (in case of nozzle type atomizer)
- (d) Feed concentration and its viscosity.
- (e) Temperature difference between the drying droplet and the hot air in the drier
- **40.4.3. Effect of Atomizers**: With a given liquid, pressure-jet atomizers tend to produce the lowest air-cell volume and the highest proportion of solid particles. On the other hand, air cell volume is also closely related to the concentration of the liquid being sprayed, and thus preconcentration of the milk to a high solids content give less trapped air in the powder. In general, highly concentrated milks are sprayed more readily with centrifugal atomizers, and this factor tends to be the important one. After drying, further air diffuses into the particle for about 24 h owing to cooling and contraction.

**40.4.4. Status of Lactose**: Lactose is the major constituent of the particle itself and comprises about 38 % of full cream powder and 50% of separated milk powder. It forms an amorphous glass-like envelop entrained in the particle and is also in the continuous phase within it. The outer envelope is only and slightly permeable and retains enclosed air and gases and seals in the fat which is only partially extractable by solvents.

**40.4.5. Free-Fat**: The proportion of free fat is very variable and it is stated to range from 3% to 10% in spray dried milk as compared with 43% to 75% in freeze dried milk and about 90% in roller-dried milk.

Lactose is, however very hygroscopic and readily absorbs moisture from air (over 50% RH) or from within the powder if its moisture content exceeds 5%. The amorphous lactose then crystallizes as  $\acute{\alpha}$ - lactose mono-hydrate and forms a crystal lattice which renders the particle permeable. The fat is than freed, and at critical moisture content between 8.6 to 9.2% is almost complete extractable by solvents. The powder itself also gradually cakes into hard lumps. In spray powder the fat globule structure is largely retained although pressure-jet atomizers may produce some degree of homogenization to globule of smaller size.

Milk powder properties are divided into two main groups:

- 1. General properties: Includes properties mentioned under International Standards ensuing the required bacteriological quality, freedom from defect, and composition.
- 2. Properties specifically related to milk powders: Includes such properties as are directly influenced by the special technology and processes applied in the milk drying industry. These properties can be further divided into-
- a. Physical properties which define the structure of the powdered product.
- b. Functional properties which define the consumer's requirements that ensure that the product is suitable for a given purpose.
- c. Product faults which indicate some und esi rable though often unavoid able deterioration caused by processing.

The distinction between the se groups is often not sharp. For example, high free fat is a defect of instant whole milk powder for household use, but is an important functional property of whole milk powder for the chocolate industry. Bulk density which is listed among the physical properties is also an important functional property.

In order to achieve the desired characteristics and functional properties, it is essential to know the influence of the operating parameters on the individual properties. The properties of the powder are affected by the milk quality, the design of the evaporator and dryer, and by the process conditions. Some properties are governed by conditions outside the evaporator and dryer. This is the case when whey is dried, as the desired non-hygroscopic nature of the powder is chiefly governed by crystallization of the lactose between evaporator and dryer. Heat classification of the powder is determined outside the evaporator and dryer, as it is a function of the heat treatment prior to evaporation.

The properties of dried milk of importance are shown in Figure 40.1 below.

## Fig.40.1: Properties of milk powders

#### **40.4.1.** Moisture

- § The moisture content of a milk powder is defined as that part of the water contained by the solid which is in a form capable of taking part in deterioration of the powder. Thus water which is bound in the lactose crystal is not normally considered to be part of the moisture content of milk powder.
- § Residual moisture is one of the most important properties of milk powder both from a quality and an economic point of view. The quality specifications lay down the maximum permissible moisture to achieve the desired shelf life. The economic aspect demands that maximum moisture content be approached as closely as possible, while at the same time ensuring that no portion of the product will exceed this moisture level.
- § High moisture content has a major influence upon the development of most storage defects except fat oxidation. Normal roller-dried milk contains 1.5 to 2.5% and spray-dried milk 2.0 to 3.5% moisture. In the manufacture of powder of minimum moisture content, the nature of the packing is extremely important owing to the hygroscopic nature of dried milk solids. There appears to be critical level around 5.0% moisture, and every effort should be made to maintain a level not exceeding 4.0%.
- § The moisture content will have an influence on the keeping quality of the powder. High moisture content (high water activity  $a_w$ ) will thus decrease the keeping quality, as the proteins will denature and the lactose, which is found in an amorphous stage, will crystallize causing the free fat to increase in whole milk powders, and oxidation of the fat will be the result. The Maillard reaction, which is a reaction between the NH<sub>2</sub> group in the amino acid lysine, and lactose, becomes more pronounced, and the powder may even become brown and lumpy. The Maillard reaction is directly proportional to the storage time, temperature and residual moisture content. The moisture can be controlled by the outlet temperature of the dryer or by applying more heat to the Vibro-Fluidizer. Moisture absorption should be avoided, and dehumidification of the cooling air is recommended in humid areas.
- § In two-stage drying, it is also important to control the intermediate moisture, i.e. the moisture of the powder at exit from the drying chamber, because it influences many other properties including solubility index, particle density, bulk density, agglomeration, etc.
- § It is also important to check occasionally how moisture content changes through the fluid bed system, to ensure that extensive overdrying followed by re-humidification to the specified final moisture content in the cooling section is not taking place.
- § In addition to the grading standards, numerous customer specifications call for even lower moisture contents than one indicated above.
- § Because there is some gain in moisture content during pneumatic conveying and blending and to a lesser extent during storage, it is normal to produce powder from the drier at a lower moisture content than that called for by the specification so that the final powder remains within specification.

§ The packing material should be of such a quality that very little vapour will penetrate the bag or container. As there will always be some vapour diffusion it is recommended to store the powder in a dry, cool place, where the water vapour pressure will be low.

The influence of various factors on powder moisture content ex-chamber is shown in Figure 40.2 below . For some variables the magnitude of i nfluence is known reasonably accurately, whilst for others only the trend is known. The degree of influence is presented as linearly proportional. It should be o bvious, however, that this is not the case, though they may be considered to be such within the relatively narrow range under consideration. In regard to the feed total solids content, it is valid up to about 48-50% solids, i.e. in the range where the logarithm of viscosity is linearly proportional to the solids content. Above this solids level, the viscosity increases more rapidly, which in turn influences the residual moisture content.

## Fig. 40.2: Effect of technological variable on powder moisture

## The moisture content of a powder is estimated by

- (a) Oven drying at  $102^{\circ}\text{C} \pm 2^{\circ}\text{C}$  to constant weight.
- (b) Toluene distillation method
- (c) Karl Fischer titration method.
- § Because of the importance of powder moisture content, many efforts have been made to control this parameter automatically. Automatic moisture control based on infrared absorption is in operation on many plants, sometimes achieving standard deviation less than 0.1%. Even better results have been achieved when combining the infrared control with feedforward loops in which the set point of outlet air temperature is adjusted according to variations of the inlet pa rameters.
- § For process control, a modified quick oven-drying method and laboratory infrared apparatus are used.
- (a) The outlet temperature is usually used as the parameter by which the final moisture of the product is controlled, thus compensating for the unavoidable (and often unknown) variations of other factors.
- § The changes of the outlet temperature on a given dryer for a given product can be expressed by following linear equation, which is valid within the range of normal running conditions.
- $\S$  It is observed that each 1 % rise in powder moisture makes it possible to decrease the outlet temperature by 5 ° C or to increase the inlet temperature by 50 ° C at a constant outlet temperature, while at the same time, keeping the particle temperature almost unchanged.

#### **40.4. BULK DENSITY**

The *density* of a powder may be defined in various ways. The density of the particle material, i.e., excluding the vacuoles, is called the *true density*. The weight of powder which can be

packed into a given volume - known as the bulk, apparent, or packing density can vary considerably.

- § Bulk density is important economically, commercially and functionally because it affects the size of containers, storage space and transport space.
- § When transporting powders over long distances, the producers are interested in high bulk density to reduce the volume. Also, high bulk density saves in packaging material. In some instances producers may be interested in low bulk density to supply optically larger amounts of powder on the retail market than that of their competitors. Low bulk density, as influenced by agglomeration, is also an important part of powder properties, influencing the instant characteristics.
- § The bulk density of milk powders is a very complex property being the result of several other properties, and being influenced by a number of factors as shown in Figure 40.3 below. The primary factors determining the bulk density are:
- (a) The particle density, given by:
- a. Product mass density
- b. The content of occluded air inside the particles.
- (b) The content of interstitial air, i.e. the air between the particles, given by
- a. Particle size distribution
- b. Agglomeration
- (c) Bulk density is defined as the weight of a given volume of powder and is expressed in g/ml, g/100 ml, or g/l. The reciprocal value is the bulk volume which is expressed in ml/100 g or ml/g. The bulk volume is usually used when a graduated cylinder glass is used for the determination. The volume of 100 g of powder is then measured in the cylinder. The value may either be expressed as tapped 0 times (loose), tapped 10 times (poured), 100 times (Tapped), or 1250 times (Tapped-to-extreme).

#### Fig. 40.3: Influence of various factors on bulk density of milk powders

## **Spray Dried Powder**

Powder particles are dispersed in air and spray particles also contain air with in them. The density of air free milk solids is 1.32 g/ml. for whole milk and 1.46 g/ml for separated milk, but the bulk density of normal spray powder may very between 0.5 and 0.8 g/ml.

## The bulk density of spray powder depends

(1) Partly upon particle size because small spheres pack more closely together than large ones, although a mixture of particles can also give a heavy powder owing to packing of small particles between large ones. In this respect jet atomizers, particularly pneumatic types, have

the advantage, whereas centrifugal atomizers tend to produce large particles and lighter powders.

- (2) On the other hand, bulk density is also influenced by the air content of the particles and consequently there is a direct relationship between bulk density and the degree of preconcentration which is advisable when a heavy powder is required. This factor is probably the most important one and favours the centrifugal atomizer.
- (3) It is also possible to increase bulk density by de-aeration of the liquid before drying.

## The bulk density of roller powder depends upon

- (1) The fineness of grinding and is therefore variable. In general, it is lighter than spray powder and varies between 0.3 and 0.5 g/ml.
- (2) Although roller powder contains little entrapped air, there are considerable voids between the irregular particles, which pack less closely than regular spheres.

The bulk density of milk powders is a very complex property, as it is a result of several other properties. However, the primary factors determining the bulk density are discussed as follows: **Related Documents:** 

#### 40.4. PARTICLE DENSITY/OCCLUDED AIR

- § The particle density depends on many factors. Composition of the solids plays an important part, first of all because it defines the product mass density (for instance the density of whole milk solids is less than that of non-fat milk solids). High protein content tends to reduce particle density as it increases the tendency of the feed to foam. This foaming can be suppressed somewhat by high heat pre-treatment (denaturing the whey proteins) and also by high concentration combined with heating the feed.
- § To achieve high particle density, it is important to avoid any treatment which may incorporate air into the feed, such as excessive agitation, etc. Rotary atomizers tend to incorporate air into the droplets, and pressure nozzles produce much higher particle density than rotary wheels. However, special vane-shaped rotary wheels, sealed disc atomizer, steamflushing of the air space in a disc atomizer are now available with less tendency to entrain air in product droplets.
- § The presence of air in the atomized droplets causes occluded air in dried particles. Depending on drying conditions, or to be precise on the particle temperature during the drying process, those air bubbles initially present may expand and further reduce the particle density.
- § The droplet formed on atomization of a concentrate with high total solids contains more solids and hence less water than droplets of the same size produced from a concentrate with lower solids . The void size and amount of vapour available to expand is therefore less in droplets from high solids concentrate .
- § A high inlet air temperature causes rapid formation of a solid surface/crust on the particle, thus increasing the void size and the quantity of vapour, and tends to cause ballooning of the

particle. The effect of high inlet air temperature can be counteracted by decreasing the outlet air temperature and by the use of secondary drying. Therefore, if all other conditions are the same, the two stage drying process provides higher particle density than single-stage drying. The resultant increase in bulk density is due to the more rapid cooling of the inlet air thus delaying the formation of a solid surface on the particles.

§ The influence of inlet air temperature, outlet air temperature, atomizer speed on bulk density of milk powder is depicted in the Tables 40.1, 40.2 and 40.3 respectively.

Table 40.4 gives some idea of the densities likely to be encountered in particle - with spray dried full cream powder manufactured in different ways.

		Dry matte Viscosity Homogeni	content of the co	t of the concentrate	e t in conc		: 47.8 % : 77 cP : 23 %	6		
		Out let ten Peripheral	velocity	of the a	air itomizer		: 90°C : 135 m	/s_		
temp. of	%	y Index	content		Density	penetration 100 g	particle	volume	a	-
Inlet te air, <sup>0</sup> C	Moisture, 9	ADMI Solubility I	Freefal		Bulk De	Ns per cm3/100	Mean density	Vacuole volume		Viscosity
155	2.54	< 0.05	18.	57	0.68	3.40	1.10	14.0	5	880
165 175	2.52	< 0.05	17.		0.66 0.65	2.84	1.08	15.2 16.0		900
185	2.77	< 0.05	12.		0.65	2.06	1.07	16.4		910
195	2.79	0.05	11.	70	0.63	2.08	1.06	17.2	4	845
205	3.01	0.16	11.	70 (	0.63	2.30	1.06	16.3		925
215 225	3.16	0.25	7.8 7.5	50 0	0.62 0.61	2.22	1.06	16.8 17.3		890 880
		Dry matter Viscosity of Homogeniz Inlet tempe Peripheral	content of the co- cation leverature o	of the concentrate vel of fat	entrate oncentra e in conc	ite :	51.5 % 85 cP 22 % 195°C 135 m/	•	ailk pe	owde
	-	Peripherai	velocity	or the a	tomizer					
p. of	<b>%</b>	Index	content		4	penetration 100 g	particle	<u>m</u>		÷
temp.	25	E .	8		Density	<b>₹</b> %		2 0	0	<u>S</u>
<i>©</i> ≇	Moisture, 9	ADMI Solubility I	Free-fat		Bulk D		Mean density	Vacuole volume cm <sup>3</sup> /100 g		Viscosity of
					- 三 D. 66	≥ 5		> 5		
70 75	5.96 5.12	0.10 0.10	7.4 9.8		0.64	1.02	1.14	5.25 9.89		1325
80	4.21	0.10	6.1		0.64	1.83	1.11	11.68		1400
85	3.3	0.10	31.2	21 C	0.64	1.90	1.09	13.42	2	1490
90	3.14	0.15	16.5	53 C	0.62	2.95	1.07	15.21	7	1590
95	2.41	0.50	19.0 25.5	00 0	0.61	3.77 4.62	1.04	18.68		1745
105	1.66	2.20	29.4	14 0	0.54	5.16	0.98	24.43		950
		ience of th tured from					e atomiz	zer on	whole	milk
		Dry matte Viscosity Homogen Inlet temp	of the co	oncentra evel of fa	te at in con		: 51.19 : 120 c : 25 % : 195°C : 90°C	P		
	of		Index							
ے <u>پ</u>	Peripheral velocity the atomizer, m/s		Solubility Ir	=		_	density	<u>ಕ</u>	%,	
Revolution of the atomizer (RPM)	i ke	*	- E	Fat Content	-	penetration, 1/100g	n particle d	Vacuole volume, an/100g	Degree of nomogenization,9	-
ar (F	Peripheral vel the atomizer, 1	25	200	2	Bulk Density	etral oe	· · · · · · · · · · · · · · · · · · ·	90	Degree of homogeniz	SCOSITY, (
evolutio omizer (	: 출 - 흥	Moisture,	ADMI	क रिव	<u>=</u>	penetr: 100g	Meanp	cuole v	36 SO	80
		Me	7		屋	2.72	Me			
19600	106	3.66	0.70	10.80	0.63		1.08	13.81	45	140
25000	120		0.20	9.88	0.63	3.00	1.09	13.52	58 68	185
28300	153	2.46	0.05	7.43	0.61	2.39	1.06	16.16	79	340
31300	169	2.63 <	0.05	9.39	0.61	2.64	1.06	16.53	84	410
Fable-40 powders		ct of differe	nt proce	essing co	ndition	s on the c	lensity of	whole	milk	
	Pı	ocessing Co	ndition	s		Dens		Specific		ne,
Conventi 20°C	onal spr	ay dried at 1	1% solid	ls, outlet	temp.	g/n 0.3	9	2	.54	
	onal spr	ay dried at 3	4% solid	ls, outlet	temp.	0.5	0.55			
	onal spr	ay dried at 4	7% solid	ls, outlet	temp.	0.6	0.66 1.51			
Conventi	onal spr	ay dried at 4	7% solid	ls, outlet	temp.	0.4	6	2	.19	
		Straight through instantized Re-wet instantized								
l 13°C Straight t Re-wet in	hrough i	nstantized d				0.6			.60	

§ The ways in which particles pack together also influence the bulk density. It depends on the particle size range. The wider the range of sizes, the more likely it is that small particles will pack in the voids left between large particles and the higher will be the bulk density. As smaller particles contain proportionately less occluded air than large particles, the removal of fine material, for instance in the manufacture of instant whole milk powder, affects the bulk density both by decreasing the average particle density and by decreasing the number of small particles available to fill the voids between the larger particles. If a powder is subjected to severe mechanical action, there may be a breakdown of individual particles, forming more fine material and decreasing the number of internal voids, thus increasing the bulk density.

§ The poro sity is defined as the percentage, by volume, of the powder mass occupied by air surrounding the individual particles. Table 40. 5 shows the effect of different processing conditions on the porosity of whole milk powder.

Table 40.5: Effect of different processing conditions on the porosity of whole milk powders

<b>Processing Conditions</b>	Powder Density, g/ml	Specific volume, ml/g	Particle density, g/ml	Porosity,
Spray dried at 11% solids, outlet temp. 90°C	0.39	2.54	0.802	51
Spray dried at 34% solids, outlet temp. 90°C	0.55	1.81	1.017	46
Spray dried at 47% solids, outlet temp. 90°C	0.66	1.51	1.150	42
Spray dried at 47% solids, outlet temp. 113°C	0.46	2.19	0.913	50
Straight through instantized	0.63	1.60	1.100	43
Re-wet instantized	0.33	3.00	1.100	70

§ The particle density is given by the density of the powder solids and the occluded air in the particles. The powder solids density expresses the density of solids without any air and is given by the composition of the powder. The solids densities of various typical components in milk powders are shown in Table 40.6 as follows:

Table: 40.6: Solids densities of various typical components in milk powders

Solids, air and moisture free:	Density, g/ml at 20°C
Milk fat	0.94
Non-fat milk solids	1.52
Calcium caseinate phosphate complex	1.39
Amorphous lactose	1.52
Beta-lactose	1.59
Alpha-lactose monohydrate	1.545

§ Powder solids density cannot be changed without changing the composition and is thus for a given product constant.

The particle density for the reciprocal value of the occluded air content is influenced by many factors. They are summarized here:

- 1. Pasteurization temperature of the milk prior to evaporation: The pasteurization temperature of the milk prior to the evaporation changes the denaturation degree of the whey proteins and thereby their physical stage and behaviour during drying. High pasteurization temperature results in many denatured whey proteins being very compact and different from undenatured whey proteins which is sponge like. A high degree of denaturation will give low occluded air content (high particle and bulk density) and vice-versa.
- 2. **Amount of air in the concentrate:** The amount of air in the feed naturally gives a high content of occluded air, especially if the surrounding air temperature during the critical stage of the drying is high causing case-hardening.
- 3. *Foaming ability of the concentrate:* The foaming ability of the feed is determining how much of the air whipped into the concentrate will remain there and in the created droplets.
- 4. *Type of wheel used or size of nozzle:* Besides the foaming ability of the concentrate, the type of wheel and nozzle is decisive as to the amount of air that will be whipped into the concentrate.
- 5. *Solids content in the concentrate:* Feed concentration plays an important role and high concentration gives less occluded air content.
- 6. *Drying conditions (one-stage or two-stage):* The drying conditions and temperature of the particle during the drying are one of the main factors. Gentle drying, i.e. low surrounding temperatures as in two-stage drying results in low occluded air.

#### **40.4. INTERSTITIAL AIR**

This is a very complex property, too. The less interstitial air the higher the bulk density.

- § The amount of interstitial air is determined by the particle size distribution and the degree of agglomeration.
- § A powder with particles of the same diameter would be ideal from a drying point of view, but undesirable from a bulk density point of view, as the air space between the particles will be very large thus resulting in low bulk density.
- § The ideal is a wide particle size distribution with enough small particles to fill out the space between the medium and large particles thus resulting in a powder with high bulk density. There is, however, a limit as to how small particles are wanted from a recovery point of view, plus the fact that a powder with many small particles will be dusty. Furthermore, they will affect the flowability negatively.

#### **CONDENSED AND DRIED MILK**

A wider particle size distribution, but in the bigger particle size spectrum is therefore wanted. This can be obtained by using high solids content and/or viscosity, reducing the velocity of the wheel or pressure of the pressure nozzles, or using bigger nozzle size. Powders with extremely high bulk density can be achieved in two-stage dryers.

\*\*\*\*\*\*

#### Lesson-41

## **Physical Properties of Dried Milks-II**

#### 41.1. INTRODUCTION

Dry milks exhibit so many other physical properties also. They are discussed here.

#### 41.2. LUMP FORMATION

Spray powder may contain small hard lumps when some of the milk droplets are only partially dried under following different conditions:

- (1) If the milk feed to the atomizer is increased whilst the volume and temperature of the hot air supply remains unchanged.
- (2) When the larger particles remain moist and develop lumps at points where they are distributed in the powder mass.
- (3) Similar effect may result if the speed of a centrifugal atomizer is reduced (e.g. by a slipping belt drive) as a reduced rate of rotation increases particle size
- (4) A falling air temperature may also cause incomplete drying of the large particles. Changes in feed rate are usually indicated by a fluctuating outlet air temperature.

Control instruments should be placed conveniently to permit continuous observation (and when possible recording) of atomizer speed, air flow rate, inlet temperature, and outlet temperature, whilst the milk feed should be calibrated to permit very small known alterations of setting to be made.

#### 41.3. FLOWABILITY

The flowability of a powder refers to the ease with which the powder particles move with respect to one another. This is an important but complex property. The main factors controlling flowability are:

- § Two free-flowing powders mixed together will not necessarily be free-flowing.
- § A good flowability is obtained from large particles or agglomerates without small particles this will, however, tend to decrease the bulk density.
- § Also the particle surface plays an important role and especially the content of free fat.
- § A powder with a good flowability will increase especially the poured and loose bulk density.

- § Milk fat is an important factor in cohesion. Increasing fat content up to 20% increases the resistance to flow. It is suggested that once a fat content of 20% is reached, the amount of surface fat on the particles is sufficiently high to cause maximum resistance to flow and therefore further increase in fat content apparently does not cause increase in cohesion and thus decrease in flowability.
- § Pressure nozzle powders are superior in this respect to rotary atomizer powders.
- § Two Stage drying gives better results than Single-Stage drying.
- § Particle size distribution cohesion or resistance to flow, of spray dried milk powder increases with decreasing particle size.
- § Other factors improving flowability are:
- (a) Agglomeration
- (b) Low percentage of f ines
- (c) Addition of f ree flowing agent (e.g. SiO<sub>2</sub>, Na-Al-silicate, or Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>) in milk powder for coffee machines.
- (d) Addition of components by dry mixing (e.g. sugar, whey powder)
- (e) Low relative humidity of atmospheric air

## 41.4. RECONSTITUTION / INSTANT PROPERTIES

The milk powder is said to have good reconstitution properties if the reconstituted liquid milk differs little or not at all from homogenized whole milk prepared from sweet fresh milk. The reconstitution process is extremely complex, involving many different factors and phenomena. The factors affecting reconstitution can be divided into four main categories as shown in Table 41.1.

Table -41.1: Grouping of factors affecting the reconstitution characteristics of milk powders

No.	Factors affecting reconstitution characteristics
1	Physical properties of the powder and powder mass structure e.g. particle density
	specific volume, etc.
2	Physico-chemical properties of the powders e.g. free fat, protein structure, etc.
3	Reconstitution conditions used e.g. water temperature, speed of mixing, etc.
4	Age of powder and storage conditions

The process of reconstitution takes place through different stages as shown in Figure 41.1.

#### Figure 41.1: Reconstitution of milk powder

The phenomena involved in reconstitution process and corresponding powder-properties are shown below in Table 41.2.

Table-41.2: Phenomena involved in reconstitution process and corresponding powderproperties

Reconstitution phenomena	Milk powder property
Wetting	Wettability
Sinking / Penetration	Sinkability/ Penetrability
Dispersing	Dispersibility
Dissolving	Solubility

§ Splitting the reconstitution process into the above steps is helpful for understanding the process, for instance to be able to find the reasons for lack of instant property. On the other hand there is no sharp borderline between these individual reconstitution steps, i.e. they do not p roceed successively but simultaneously, at least two at a time and the individual properties influence each other. Therefore, it is difficult to determine the individual properties independently of the others.

The factors influencing instant properties are shown in Figure 41.2 . Friability, which is the opposite of mechanical stability, is included, because high friability will result in the breaking down of the agglomerates into fines thus adversely affecting the instant properties.

## Fig-41.2: The factors influencing instant properties of milk powders

## 41.1.1. Wettability

- § The wettability is a measure for the ability of a powder to be wetted with water at a given temperature. This is only used when producing instant powders. The wettability depends on the surfaces of the agglomerates or single particles are they water repellent or will they absorb water too quickly thus forming a film through which the water cannot penetrate.
- § The fine powder produced by the spray process is difficult to wet and, tend to produce lumps which are slow to disperse in water. It appears that very small particles under  $\sim 50\mu$  swells within initial contact with water and blocks the interstices and hinders the access of more water. A large particles size is preferable, and a figure of  $100-150\mu$  is generally considered to be an ideal size.
- § Generally speaking, wetting is a process in which the gaseous phase at the surface of the solid phase is replaced by a liquid phase, all three phases coexisting for some time, so that a certain amount of intermixtures and solutions is not only possible but usually unavoidable.

§ The factor deciding if there will be any wetting at all is the interfacial tension between the particle surface and the water. Skim milk powder particles will usually be wetted easily (provided < 0.03% fat on the surface), as the powder material is mainly lactose being in an amorphous phase and protein, both absorbing water readily. However, whole milk powder particles are always covered by a layer of fat, making them water repellent. The amount of this surface free fat varies between 0.5 and 3% of the powder.

§ This water repellence of the particles caused by their fat coating may be overcome, and an interfacial tension facilitating the wetting may be achieved by adding a surface active agent to the surface free fat. It has been known for years that phospholipids such as lecithin are well suited for this purpose.

## 41.1.2. Sinkability

- § When the particles have been wetted, the individual components of the milk powder start dissolving and dispersing, thus forming a concentrated solution of milk around the particles. At the same time the particles start sinking to the bottom, but in order to make the particles sink, the density of the particles has to be greater than that of the water.
- § The density of a particle depends on its composition and amount of occluded air. During the first stages of reconstitution the density of the particles decreases, mainly because the lactose and the minerals, which are the heaviest milk components, start dissolving faster than the other components. At the same time, the density of the solution being formed is increased because of the dissolving lactose, so that the difference between the densities of the particles and of the surrounding liquid is reduced. The particle density may even become the same or lower than that of the liquid, so that, after the initial sinking, the particles start to rise again. Thus to prevent this, the particle density should be high, i.e. the content of occluded air should be low.
- § The reconstitution of a mass of powder is more complicated. Powder is a composite surface with a greatly ramified system of capillaries of various dimensions and a complicated geometrical pattern thus having different capillary attraction effects.
- § Under these conditions there will be wetting not only on the surface of the water, but also of particles lying above the surface, as the water is drawn toward them by capillary attraction. This replacement of interstitial air by water through capillary penetration is very often incomplete, as the amount of penetrating water is insufficient, thus leaving air bubbles between the wetted particles. In this way we have all three phases going on simultaneously, resulting in the coexistence of their products of varying concentrations. This coexistence is very dangerous, because after a short time the space between the particles will be filled with milk of different concentrations. This results in a sticky jelly with islands of unwetted powder and residual air. Furthermore, lumps, that are wet and swollen outside and dry inside, are created. As these are impervious to water, their complete reconstitution is extremely difficult even with strong agitation.
- § Skim milk powder should be wetted within 15 sec. to be termed instant. For whole milk powder there is no requirement, but many producers of instant whole milk powder manufacture the powder to the same standard as valid for the skim milk powder. However, for the subsequent dispersing process, especially for whole milk powder, it is advantageous

that the wettability is about 30-60 sec., as it eases the subsequent dispersion of the powder into the water.

## 41.1.3. Dispersibility

Another important property of instant powders is the ability to disperse in water by gentle stirring. This means that the powder should disintegrate into agglomerates which again should disintegrate into the single primary particles.

- § To obtain a good dispersibility of a powder it is necessary that the powder is wettable and that the agglomeration is optimal, i.e. no fine particles should be present.
- § The analytical method is very difficult to define and perform and the reproducibility is very poor. There are numerous methods, and the results cannot be compared.
- § The powder is considered instant by IDF, if the dispersibility is at least 85% (whole milk) or 90% (skim milk). However, plants with new drying technology easily produce powders with a dispersibility of 95%.

## **41.1.4. Solubility**

To obtain fully reconstituted milk in a reasonably short time and with minimum effort, capillary penetration of water into the powder must therefore be avoided. The capillary effect depends on the structure of the powder, i.e. the size of the agglomerates, the size and the amount of non-agglomerated particles, the amount of interstitial air and the specific surface area of the powder. Penetration of water into the powder is easily avoided/delayed - to allow dispersion before dissolution - when the powder consists of large agglomerates.

Milk powder has to be soluble in water. However, not all of the components in the powders are soluble when reconstituted in water. In powders produced in modern dryers, this amount is very small and approaching 100% solubility.

The solubility (more strictly, reconstitutability) of dried milk is a very important commercial property. Loss of solubility is due to denaturation of protein by heat treatment during the manufacturing process.

- § The severe heat treatment used in atmospheric roller- drying reduces the solubility of the powder to 80 to 85% by damage to the fat globule. Structure also releases some drops of oil, and the reconstituted milk has a less attractive appearance. Some makers employ preliminary homogenization of the milk to secure improvement.
- § Spray dried milk (once it has been wetted) should be highly soluble up to 98 to 99% and the fat globules should be largely undamaged. The solubility of the powder is determined by the heat treatments used at all stages of the manufacturing process.
- § The use of temperatures above 74°C for pre- heating the milk reduces the solubility of the powder as the temperature rises. At 88°C as used for "high-heat" powder, the reduction is definite but of little real importance to most purposes.

- § Usually the evaporation process is of less significance, but some reduction of solubility may result if a high-temperature effect is used or the milk is over concentrated.
- § Powder solubility is also affected by the temperature of the hot air in the drying chamber and the time of exposure of the powder to it. The maximum desirable air inlet temperature varies with the degree of air turbulence and the speed of removal of the powder. It should not exceed 170°C. Rapid removal of the powder is essential, and recovery in cyclones is preferable to bag collectors.
- § The final solubility tends to be the cumulative result of relatively small effects at each succeeding stage of the whole process. Protein insolubility due to heating of the liquid milk probably differs from that induced by exposure of the powder to hot air. The former is an irreversible change, while the latter is at least partly reversible if the powder is reconstituted in water at 50°C instead of 20°C.
- § A spray powder of poor solubility tends to form three layers when reconstituted. The top layer consists, mainly of fat globules which have carried some insoluble protein to the surface. A sediment at the base consists of settled insoluble protein which has also entangled some fat globules, whilst the middle layer of soluble protein also permeates both top and bottom layers.
- § During storage, loss of solubility may occur, depending mainly upon the moisture content of the powder and the storage temperature. Large-scale reconstitution of spray dried milk for consumption as fluid milk or for manufacturing purposes requires considerable care and even dispersal of the milk solids. The complete absorption of water by the powder takes some time; hence it is desirable for the reconstituted milk to stand undisturbed for 3 to 4 hrs after preparation. If the product is to be consumed as fluid milk, preliminary cold storage for 24 hrs is needed fully to regain the flavour and characteristics of fluid milk. In addition, homogenization is often employed.
- § The reasons for high Insolubility Index (i.e. bad solubility) in a powder may be many. It is usually denatured caseins or very complex combinations of casein-whey protein and lactose, the chemistry of which is not fully understood. The main contributing factors are:
- 1. Bad quality milk with a high development of lactic acid.
- 2. High temperatures of the concentrate during the evaporation will cause a pronounced agethickening resulting in viscosity increase and bad atomization, i.e. high temperatures during the drying.
- 3. Generally it may be said that the higher the temperatures and viscosities during the processing, the higher Insolubility Index may be expected. Powders with a high lactose content such as baby food will practically never get a high Insolubility Index, as lactose protects the proteins from denaturation.
- 4. Powders dried according to the one-stage drying principle will more easily get a high Insolubility Index than from the two-stage drying principle.

## 41.2. AGGLOMERATE STRUCTURE AND POWDER PROPERTIES

Depending on the design and adjustment of the fines return system - particularly the location of the introduction of the fines in relation to the atomization device - different agglomerate structures result (Figure 41.3), which influences certain powder properties, such as bulk density, mechanical stability, dispersibility and slowly dispersible particles as shown in Figure 41.4.

"If the fines are introduced close to the atomizing devise, the moisture content of the primary spray particles is high and thereby their plasticity and stickiness, and the fines particles may penetrate primary particles or be completely covered by concentrate. Such agglomerates have been termed 'Onion'-structured (Figure 41.5). 'Onion'-structured agglomerates are characterized by a high mechanical stability and a high bulk density, but they will often appear as slowly dispersible particles after reconstitution.

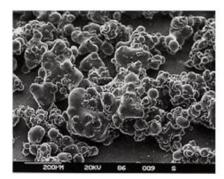
When collision takes place at a progressively longer distance, away from the atomizing device, less compact agglomerate structures are obtained. Such structures have been termed 'Raspberry'- and 'Grape'-structures (Figure 41.6) in decreasing order of compactness.

## Fig.41.3: Types of agglomerates

properties Agglomerate & Powder relationship Fig.41.4: structure

"With progressively looser agglomerate structures, the bulk density and mechanical stability decrease gradually, and the overall instant properties improve. However, if a 'Loose Grape'structure is eventually obtained, the mechanical stability may be so low that the powder becomes very susceptible to attrition resulting in deteriorated instant properties.

A 'Compact Grape'-structure is regarded as the ideal compromise where the powder has simultaneously good instant properties and sufficient mechanical strength to enable necessary transport and packaging



'Onion'-structured agglomerate

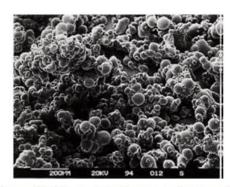


Figure 41.5: Scanning electron micrograph of Figure 41.6: Scanning electron micrograph of 'Grape' -structured agglomerate

#### 41.1. SCORCHED PARTICLES

Scorched particles are generally accepted to be a measure for any deposits in the drying chamber having been exposed to high temperatures thus getting scorched, discoloured and at the same time insoluble.

- § It is not only the dryer that contributes to the scorched particles, as even the raw milk may contain some dirt or sediment, and if not clarified in a separator these will be found in the powder.
- § Also from the evaporator, brown, insoluble, jelly lumps may contribute to the scorched particles, if deposits have been formed in the tubes due to insufficient coverage of the tubes, or insufficient cleaning.
- § If the scorched particles originate from the dryer, the reason is very often deposits in the wheel or around the nozzles or in the air disperser. How to solve the problem may differ from case to case, but adjustment of the air disperser will usually help in most cases.

The test for determining scorched particles is simple and rapid which involves taking of 25 g skim milk powder, 32.5 g whole milk powder or 15 g whey powder (or equivalent amount of concentrate depending on total solids) and mixing with 250 ml of water of 18-28°C in 60 sec. in the same kind of mixer as used for insolubility index. The milk solution is then filtered and the filter pad is compared with a standard for classification. The scorched particles are expressed as A, B, C, or D depending on the intensity and colour of the particles left on the filter as shown in Figure 41.7 below

## Figure 41.7: ADMI chart for Scorched Particles Standards for dry Milk

If scorched particles cannot be traced to the evaporator, or the spray dryer, they may originate from milk powder used in "compounded" products like baby food.

#### 41.1. Total Fat

- $\S$  Most fat globules are less than 2  $\mu$  m, but a small proportion of the fat (e.g., 2% of it) is to be found as a thin layer on parts of the surface of the powder particles. The vacuole volume of most powders varies from 50 to 400 ml . kg  $^{-1}$  .
- § The total fat in the whole milk powder is a question of standardizing the raw milk prior to the processing and has got nothing to do with the drying process.
- § As the fat content in the raw milk in practically all cases is too high when producing whole milk powder, skim milk powder is sometimes used for standardizing. As the solids content will increase by adding skim milk powder, the evaporator should be designed accordingly.

#### 41.2. Surface Free Fat

In whole milk powder, the fat is present as fine globules covered with a membrane substance and distributed evenly in the particles. However, not all the fat is protected by a membrane, especially on the surface of the particle, but it is also found inside the particles. This type of fat is termed Free Fat, and it will have a direct influence on the shelf-life of the powder and is directly responsible for the non-wettable surface when the powder is mixed with cold water.

- § Free fat is the portion of the fat content of a milk powder which can be extracted by organic solvents (e.g. carbon tetrachloride).
- § Free f at does not exist in globules protected by the amorphous lactose, but occurs as "pools" on the external and internal surfaces (craters) of powder particles.
- § The free fat content is expressed as a percentage either of the total fat content or of the total mass of powder. A free fat content of 10% is excessive if it refers to a powder basis but may be acceptable if it refers to a total fat basis.
- § Free fat content of milk powders affect both reconstitution and powder stability.

## Free fat in the whole milk powder cannot be avoided but reduced considerably by:

- (a) Direct pasteurization, especially at low temperature, results in low viscosity of the concentrate and a fine atomization with a big surface to mass ratio leading to increased free fat content.
- (b) The f ormation of free fat is reduced by a high content of amorphous lactose, and is increased by high protein content.
- (c) Low melting fats tend to produce higher levels of free fat.
- (d) As total fat content increases above ~ 28%, the free fat rises progressively
- (e) The free fat is most efficiently reduced by homogenization of the concentrate, preferably in a two-stage homogenizer.
- (f) Avoiding excessive pumping and agitation of the raw uncondensed milk. Recirculation in the evaporator should be avoided by all means.
- (g) Gentle drying conditions: Effects of drying variable such as inlet air temperature, outlet air temperature, atomizer speed etc. are shown in Tables 39.1, 39.2 and 39.3 respectively. It is a general rule that nozzles produce a powder with a lower free fat content than with the wheel, mainly due to the homogenization effect of the nozzle.
- In plants with integrated fluid beds the free fat will increase, if the bed temperature is too low signifying too high moisture content in the powder, which results in lactose crystallization.
- (i) Gentle powder treatment:

- a. Any strong mechanical handling of the powder should be avoided.
- b. The two-stage drying gives a powder with a lower free fat content than the one-stage drying.
- c. Avoiding pneumatic transport
- d. Low pressure drop in cyclones
- e. Effective cooling of powder by fluid bed.
- (j) The powder moisture should not be too low.
- (k) Avoid lactose crystallization
- (l) Addit ion of emulsifier

## 41.3. Whey Powder

When producing whey powder, especially the non-caking type, the following properties, and some of those mentioned earlier for Milk Powder, should be considered:

#### 41.3.1. Total Moisture and Free Moisture

During the manufacture of non-caking whey powder, the lactose is crystallized during which process the lactose picks up one molecule of water.

#### 41.3.2. Hygroscopicity and Caking Properties

The hygroscopicity, i.e. the ability to absorb moisture, of the whey powders is determined by the degree of crystallization of the lactose. But also the salts and even the proteins absorb water, however, only in a limited amount compared with the lactose.

The caking properties of whey powder are even more important, as they are a measure for the tendency to form hard lumps in bags during storage. There is no direct relation between the hygroscopicity and cakeness, as a rich crystallization in itself is not enough to prevent caking. Thus it is of importance that the crystals are small, and that they are evenly distributed in all the powder particles. The sticky powder formed during the moisture increase will during the crystallization become cemented in hard lumps. Non-caking whey powders should have a degree of caking of <10%.

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#### Module 16

#### **Defects in dried milk**

#### Lesson-42

# **Defects in Dried Milk during Manufacture and Storage, Their Causes and Prevention-I**

#### **42.1. INTRODUCTION**

Shelf-life of milk powder is dependent on quality of milk used, method of manufacture, composition of final product, efficiency of packaging and temperature of storage. Hygienic quality of the final product is very important from consumer's safety point of view. Powder must be free from all the pathogens including those which can cause food poisoning. The physico-chemical changes that occur on storage of dried milks determine the storage stability and the nutritional characteristics and are of paramount importance from economical, commercial and functional view points.

Micro-organisms do not proliferate in dried milk; storage defects are purely chemical in nature. These tend to develop slowly, and normally the product may be stored without deterioration for many months. The useful storage life is much influenced by the temperature of storage and the difficulties are more pronounced in hot climates, where the product is potentially of greatest value.

## 42.2. THE PHYSICO-CHEMICAL CHANGES DURING STORAGE OF MILK POWDERS

It has been observed that the life of roller dried whole milk powder from cow's milk is about 10 months; the spray dried product however cannot be stored more than 6 months at 17<sup>o</sup>C and half of this time at 27<sup>o</sup>C.

Although the milk powders are not sterile products, the low water activity does not permit microbial growth and microbial activity. The same is true of enzymic activity. The changes occurring in milk powders on storage are of physical, chemical and physicochemical in nature. Both the lipids and the non-lipid constituents of milk undergo such changes and normally the rates of such reactions are accelerated with the elevation of storage temperature and levels of moisture and oxygen in the product.

The quality of milk and the processing parameters adopted while manufacture of milk powders are also the major factors determining their storage stability.

#### 42.2.1. Water Activity $(a_w)$

The most important variable determining the rate of undesirable changes in milk powder is the water content. When comparing different types of powder, it is probably easiest to consider water activity ( $a_w$ ). The relationship depends on the composition of the product.

- § The higher  $a_w$  of whole milk powder as compared to skim milk powder of the same water content is caused by the fat not affecting  $a_w$ .
- $\S$  Whey powder has  $a_w$  slightly different from that of skim milk powder because in a dry product the soluble constituents (especially sugar and salts) decrease  $a_w$  somewhat less than casein. This is only true, however, as long as all lactose is amorphous, which often does not apply to whey powder.
- § The  $a_{\rm w}$  is considerably reduced if lactose crystallizes without absorption of water by the powder, at least if  $a_{\rm w}$  is less than about 0.5.
- § Crystalline lactose binds water very strongly, and that is also why the usual oven-drying methods to estimate the water content do not include the bulk of the water of crystallization. If the water content excluding the water of crystallization is taken as a basis, then  $a_{\rm w}$  is even higher for the powder with crystallized lactose.
- $\S$  It is thus advisable to make milk powder sufficiently dry and to keep it in that condition. If it is not hermetically sealed from the outside air, it will attract water in most climates. The higher the temperature, the higher the water activity. Because several reactions are faster at a higher  $a_{\rm w}$ , this implies that a temperature increase may well cause an extra acceleration of deterioration.
- § The deterioration effect may be especially strong if the powder loses its glassy state. A lactose—water mixture will be at most ambient temperatures in the glassy state if its water activity is below 0.3. Because lactose is the dominant component of the amorphous material in a powder particle, about the same relation is supposed to hold for the powder. This means that most dairy powders are in the glassy state (i.e., the nonfat part of the material), except if the water content is high and the temperature is also high. A change in conditions leading to a glass—liquid transition will strongly accelerate most reactions and physical changes occurring in a powder.

## 42.2.2. Colour, flavor and off flavor characteristics

The user of dried milk is concerned mainly with the flavour defects which arise during storage. Following aspects are important which affect the colour and flavour of dry milks:

- § The use of poor quality milk is not desirable for manufacture of good quality dry milk.
- § Some feed taints may be removed during the course of vacuum evaporation or drying, but not always.
- § Acidity already developed in the milk will be retained in the powder, which acquires an unpleasant flavour.
- § Neutralization of acid milk has been practiced, but the flavour of the resulting powder remains unsatisfactory and the colour may darken. Very acid milk cannot be dried by the roller process. First quality powder should not exceed an acidity of 0.15 (as lactic acid), and no powder should exceed 0.17%.

§ Assuming that good quality milk has been used, the most likely defect of freshly made powder is some degree of burnt flavour. A distinct burnt flavour is only likely to occur in spray powder following considerable overheating in the drying chamber, but may arise much more readily in roller powder owing to overheating on the rollers or faulty adjustment of the scraper knives. In any case, roller powders always possess a distinct cooked flavour. The flavour of spray powder is influenced considerably by the milk preheating temperature used. Thus "low-heat" powder or powder from "ultra-high temperature" processed milk gives reconstituted milk having a flavour close to that of pasteurized milk. "High-heat" powder when reconstituted possesses a more cooked flavour, which however, is not unpleasant and indeed is preferred by some consumers. In practice, storage changes produce various flavour defects which mask other factors and after 2-3 months the "low-heat" powder is commonly inferior if special precautions have not been taken.

The colour and flavor changes will progress during prolonged storage if conditions like elevated temperature in combination with increased moisture content prevail. Although much research remains to be done, two major types of deterioration are well recognized, affecting fat in one case and protein plus lactose in the other. Ultimately these affect the flavour, solubility, colour and nutritional value of the powder. Two major categories of reactions are:

- (a) The oxidation of lipids and
- (b) The non enzymatic browning commonly known as the Millard reaction.

The former is associated with the tallowiness whereas presumably both these reactions contribute to stale and allied flavours. With the advancement of storage, the discolouration will also progress and fluorescence substances are also produced.

## 42.2.3. Fat Decomposition

Oxidation of the fat leading to the production of flat, and finally marked tallowy off-flavours is a major storage defect of full cream powder and some times occurs to a lesser extent in the residual fat content of separated milk powder. Some of the observations made by different workers are:

- § The chemical changes result from the addition of oxygen to the double bonds of unsaturated glycerides, giving peroxides which can be estimated by chemical means.
- § In the oxidation of linoleic acid, the C-11 methylene group is preferential point of attack. Many other volatile aliphatic compounds such as alcohols, lactones, esters, acids and hydrocarbons arise from oxidation of unsaturated fatty acids.
- § At first, the peroxides accumulate but later they decompose to various aldehydes, ketones and acids which impart the unpleasant flavours.
- § Aldehydes are detectable with colour reagents but the sense of taste is much more sensitive than chemical tests.
- § The reaction commences with a slow linear induction phase, followed by a period of rapid exponential change and combination with oxygen, large quantity of O<sub>2</sub> can be absorbed. The

break point at which the oxidation changes from first to second phase is generally found to be at 37 weeks in whole milk powder.

- § Peroxides are formed when the container contains an excess of free  $O_2$ , but later decomposition of peroxides exceeds the rate of their formation.
- § The keeping quality of individual "low heat" powders varies considerably from 4-12 months in temperate climates and perhaps half this time in hot climates.
- § Though the development of poor flavour is parallel to  $O_2$  absorption, the ratio is not constant and the most stable powders require the most  $O_2$  in order to deteriorate to a given flavour value.
- § The odour and flavour profiles of dried milk with addition of antioxidants (BHA/BHT & Maillard reaction mixtures from histidine and glucose) and stored under air and nitrogen are as follows:
- · The Maillard reaction products are as effective as BHA/BHT in retarding the development of oxidative off flavours and off odours.
- $\cdot$  S amples to which antioxidants are added or samples stored under nitrogen retain a higher acceptance for storage times as long as 84 weeks.
- · The 2, 3 butadione has a sweet and buttery odour and flavour at low concentrations and the branched chain aldehydes, the Strecker aldehydes are generally described as having fruity odours and flavours at low concentrations.
- · The sulphur compounds are known to impart cooked odours and flavours.
- $\cdot$  Some compounds, 1-octen-3-one and 1-octene-3-ol that are known to give rise to metallic and mushroom odour and flavour respectively in oxidatively deteriorated milk products.
- § Factors affecting oxidative stability of lipids in dried milks include oxygen, moisture, temperature, light and metals like copper and iron. These influence the system depending upon their fat content, presence or absence of antioxidant and packaging under the nitrogen atmosphere.
- § The use of antioxidants instead of nitrogen gas packaging, derived from various materials showing promise as practical antioxidants are: wheat germ oil, gum guaiac, rice bran concentrate mixed tocopherols, oat flour and special preparations made from ethyl gallate, ascorbic acid, nordihydroguaiaretic acid (NDGA), thiourea, hydroquinone monobenzyl ether, flavones, querectin, ascorbyl, palmitate, histide, tryptophan, phosphoric acid either alone or in combination of a few.
- § For addition of any antioxidant legal acceptance is must and for the present in India, no antioxidant other than lecithin, ascorbic acid and tocopherol is permitted accepting BHA not more than 0.01% by weight in milk powder. In infant milk no antioxidant is permitted.

§ Inspite of some fairly promising indications that antioxidants retard tallowiness in dry milks, packaging in inert gas is the most efficient method at present available for preventing fat deterioration.

# 42.2.4. Fat Hydrolysis

True rancidity results from the hydrolysis of fat to free fatty acids such as butyric acid. The lipolytic enzymes involved can be derived from the milk itself or they may be of microbiological origin. This defect has become unusual since strict attention is now given to both milk production and plant hygiene and higher preheating temperatures which inactivate lipases.

# 42.2.5. Other Forms of Fat Deterioration and their prevention

The other flavours are described as "stale" or most characteristically as like "Coconut". A 'toffee" flavour may also be a form of the same defect. They are produced only from milk fat and do not occur in dried separated milk. The defect is accelerated by high-temperature storage and by high moisture content, but is independent of  $O_2$  and occurs in gas packed powder. This form of deterioration seems to occur at a fairly early stage during storage, it develops before the fat oxidation defect from which it also differs in flavour. There is strong evidence that the change is caused by a rearrangement of monoethenoid fatty acids in milk fat to form lactones and that  $\delta$ -decalactone in minute quantities is the substance responsible for the coconut flavour. The origin of this lactone is thought to be 9-deconoic acid. These rearrangements involve no  $O_2$  and the empirical formulae remain the same. No definite method of prevention has been suggested except low-temperature storage.

In the absence of special precautions, oxidation occurs rather more rapidly in spray than in roller powders, but in recent years much progress has been made in extending the life of spray powders. With full-cream powder the main cause of deterioration is overcome if fat oxidation is prevented, other possible defects being within the control of manufacturer. Hence, to keep the autoxidation within reasonable limits for a long time, following measures should be taken:

- 1. **High Temperature Preheating:** The milk should be intensely heated to form antioxidants. The problem is, of course, that the heat treatment also causes a distinct cooked flavor. Compared with pre-heating the milk at 73.8 °C, preheating at 82.2 °C improves the keeping quality of the powder, whilst at 87.8 °C, the increase is 3-5 times. The mechanism is not fully understood, but in general it is associated with the formation of trace of protein decomposition products containing a sulfhydryl group which function as antioxidants and prolong the induction period. Natural milk proteins contain no free -SH groups, but in the denatured protein formed in heated milk, such groups become unmasked and reactive. The first action may be the formation of cysteine followed by further reaction at the exposed sulfur ends of the chains to split off H<sub>2</sub>S or methyl mercaptan. Traces of H<sub>2</sub>S are detectable in the heated milk. The action of -SH group is uncertain, but may be due in part to prevention of the catalytic action of heavy metals by removing them as sulfides.
- 2. High temperature preheating may also prevent changes produced by enzymes derived from the raw milk or from bacteria, thus improves the bacteriological quality of the powder and preserves the vitamin content during storage.

- 3. The rate of autoxidation strongly increases with decreasing  $a_{\rm w}$ ; however, to prevent other types of deterioration (especially Maillard reactions)  $a_{\rm w}$  should be as low as possible. The effective Q 10 of the autoxidation reaction in milk powder is relatively low (about 1.5) because a higher temperature also causes higher  $a_{\rm w}$ . The most suitable water content is generally 2.5 to 3%.
- 4. **Packing in Inert Gas:** This depends upon prevention of the chemical reaction by removing the  $O_2$  from the container and replacing it with  $N_2$  or  $CO_2$  usually the former. It is the only method which completely prevents oxidation for practical purposes and can extend storage life up to 7-10 years even at high temperature. Oxygen should be removed as effectively as possible by gas flushing. A problem is that the vacuoles in the powder particles contain some air, hence,  $O_2$ . Either the powder should contain hardly any vacuoles or the gas flushing should be repeated after a few days. Equilibrating the gas inside and outside the vacuoles by diffusion takes several days in whole milk powder (several weeks in skim milk powder). Equilibration is faster if the powder particles have a greater number of cracks.
- 5. Addition of Antioxidants: Of the more successful substances "Ascorbic acid" added @0.03% in the liquid milk prolongs powder keeping quality by several months but disappears during storage. The most effective antioxidants appear to be esters of gallic acid particularly Ethyl or Propyl gallate. The incorporation of as little as 0.07% of ethyl gallate in the powder prolongs the keeping quality to about two years when only low temperature preheating has been used. Ethyl gallate is non-toxic and has no effect on flavour; it does not disappear during storage and appears to remain unchanged. Keeping quality may be extended still further by combining high-temperature preheating with the addition of ethyl gallate. In this way, it is possible to produce powders having a keeping quality of from 3-4 years in temperate climates. Nordihydroguaiaretic acid has also been found effective when added @ 0.04% of the weight of the fat.
- 6. The rate of deterioration increases rapidly with rise of storage temperature. The keeping quality of an unstable powder may fall as low as 6 weeks in a tropical climate.
- 7. Oxidation is more rapid in Powder of high acidity.
- 8. The powder should be packaged in such a way that air and light are kept out. Generally, this implies packaging in cans.
- 9. Rigorous measures should be employed against contamination of the milk with heavy metals such as copper and iron which act as catalysts and shorten the induction phase. Stainless steel equipment should be used.
- 10. Other less certain factors are the possible presence of oxidizing enzymes of microbiological origin in the milk and seasonal variations in the glycerides-composition of the fat.
- 11. Intensive homogenization of the concentrate should be carried out.

#### 42.2.6. Protein - Lactose / Maillard Reactions

Millard reactions between protein and lactose produce a series of storage defects which often occur together in a progressive sequence of stale flavours, increasing loss of solubility,

unpleasant "glue" flavour and darkening or brown discoloration. These changes may occur in all types of powder but are associated predominantly with separated dried milk and tend to be most pronounced in roller dried powders. They are greatly accelerated by high-storage temperatures and in addition, the moisture content of the powder is a major factor. Critical moisture levels appear to be about 5% for spray powder and 4% for roller powder. Above these figures, the defects are accentuated.

This reaction in addition to accounting for the major part of browning, the protein-carbohydrate complex or its decomposition products also result in production of reducing substances, fluorescent compounds and disagreeable flavour materials. About 80 different compounds have been isolated and identified which include furanics, lactones, pyrazines, pyridines, acetyl pyrrole, amides, pyrolidinone, succinate, glutarimides, carboxylic acids, acetone, 2-heptanone, maltol etc.

Some of the findings in this regard are:

- § Maillard reactions increase considerably with water content and with temperature. They lead to browning and to an off flavor. The 'gluey' flavor that always develops during storage of dry milk products with too-high water content is usually ascribed to Maillard reactions; the main component appears to be *o* -aminoacetophenone.
- § If extensive Maillard reactions occur, they are always accompanied by insolubilization of the protein. Accordingly, the insolubility index increases when milk powder is stored for long at a high water content and temperature.
- § One of the first reaction products detected in Maillard reaction is hydroxymethyl furfural (HMF), the concentration of which increases with storage time, temperature and moisture content. Many of the products of Maillard reaction are aroma substances (aldehydes, reductones, other furfurals etc.) which have an appetizing odour, but high levels of these compounds may cause considerable odours and off flavours. The ε-amino groups of lysine are mainly involved and because the compounds formed during the reaction such as fructoselysine, furosine etc. are resistant to enzymes, the content of available lysine is reduced, but usually only to a small extent. Another result of the reaction is the production of substances which lead to colour changes, but only in a more advanced stage of the reaction.
- § Browning reactions of the Maillard type occur if the storage of powdered dairy products at ambient temperature and having the moisture content  $\geq$  5%. The HMF content is further increased in milk powder with added iron or added Vitamin A.
- § With the progress of Maillard reaction, the decrease of amino-N is closely related to increase in combined sugar, increase in oxygen absorption and carbon dioxide production, increase in colour and reducing power and decrease in solubility.
- § A number of compounds have been shown to inhibit the browning reaction. In milk products, active -SH groups serve as natural inhibitors in retarding the heat induced browning, but the mechanism is not understood. Sodium bisulphite, sulphur dioxide and fo rmaldehyde also inhibit browning in milk system as well as in simpler amino acid-sugar solutions.

#### 42.2.7. Deterioration of a high moisture powder proceeds in a series of stages:

- § First the lactose absorbs moisture and changes to crystalline L -lactose monohydrate containing 5% water of crystallization.
- § In powders of low moisture content, the lactose is present in the amorphous  $\alpha$  and  $\beta$ -forms (1: 1.5) with only 0.5 to 5.0% of lactose hydrate. If the moisture content and the temperature remain low, the lactose preserves its amorphous form and no deterioration occurs. If the moisture content is high, as much as 40-60% of the lactose may be hydrated in a fresh powder and at levels of 6.5 to 7.0% for full-cream powder and 7.5 to 8.0% for separated milk powder crystallization becomes rapid and complete (dried milk can also absorb moisture very readily from the air). Such powders may show an apparent decrease in their moisture content. There is often a loss of free-flowing properties and possibly some definite "Caking" of the powder. The impervious lactose envelope around spray powder particles probably changes to a crystalline lattice.
- § The free lactose content of the powder then falls and an insoluble protein-lactose compound is formed which contains lactose equivalent to the quantity of the free soluble lactose which has disappeared. The reaction is presumed to occur between the aldehyde group of the lactose and a protein-amino group. The amino group mainly involved is that of lysine, and about 40% of the original lysine disappears, which considerably reduces the nutritional value of the powder. As the protein lactose reaction proceeds, the remaining protein also becomes progressively insoluble and eventually the loss of solubility may be pronounced, particularly of roller -dried milk.
- § The final stage involves a decomposition of the protein lactose compound to form products which include substances having an unpleasant glue-like flavour and a brown colour. The reaction involves absorption of  $O_2$ , and the evolution of  $O_2$ , but the exact nature of the breakdown and the identification of the decomposition products are uncertain. In general, the  $O_2$  reacts preferentially with the fat in full-cream powders, and tallowy flavours predominates, whilst the stale and gluey flavours are more characteristic of separated milk powder, but both forms of deterioration can proceed simultaneously.
- $\S$  If the powder is gas packed, the protein lactose reaction and loss of solubility may still occur, but the final changes are much less serious. There may be some development of brown colour and a slight caramalize flavour, but unpleasant flavours are unusual. It would appear that development of the gluey flavour requires a supply of  $O_2$ . Gas packing is therefore partially effective but is not a real solution.

The manufacturer can avoid these defects of browning more simply and with greater certainty by producing powders by limiting heat treatments and time and of low moisture content and using moisture-proof packaging combined with low storage temperatures.

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## Lesson-43

# Defects in Dried Milk during Manufacture and Storage, Their Causes and Prevention-II

#### 43.1. INTRODUCTION

Apart from normal defects observed in dried milks, the defects related to reconstitution and nutritional properties of dried milks are also important.

#### 43.2. DISCOLOURATION

#### 43.2.1. Roller Dried Powder

Small errors of operation during roller-drying can readily result in dried milk having a darker shade of colour. More serious failures or acid milk may produce a distinctly brown product. Discolouration often associated with a heated flavour, results from overheating due to excessive time of contact between the milk and the hot roller surfaces, too high a steam temperature / pressure or prolonged heating of partly concentrated milk between the upper surfaces of the rollers.

A more common defect is the presence of brown scorched particles, sometimes sufficiently numerous to impart a visible speckled appearance. Even high quality roller powder usually contains a few specks of scorched material. The major cause is incomplete removal of the dried milk film from points on the rollers. Any milk solids which pass the scraper knives remain on the hot roller surface and eventually become burnt. Frequent grinding and sharpening of the knives, accurate alignment across the rollers, and careful adjustment of knife pressures at all points is one of the most essential and skilled operation in the production of roller powder.

# 43.2.2. Spray Dried Powder

A general brown colour and burnt flavour is not associated with spray powder unless serious overheating has occurred in the drier. Particles of foreign matter may, however, be present and although they are not usually noticeable in the powder, they may be clearly apparent as sediment in the reconstituted milk. Such sediment may be of several kinds, and is best collected by filtering the reconstituted milk through a cotton pad; the specks may then be examined with a lens. Inadequate filtration or clarification of the milk supply may result in the presence of typical foreign matter from the raw milk.

The air supply to the drier should be filtered to prevent the admission of out side dust and particular care should be taken to avoid the entry of minute particles of coal or ashes when coal fired boilers are used. The air supply is some times drawn from inside the building, because of its higher temperature, but this involves the risk of introducing powder dust which may become charred when heated.

However, scorched particles arise mainly from powder deposits which have been held for long periods in ducts or at other points in the drier or from drying chamber due to inadequate mixing and directional control of the milk and air streams. The moist powder sticks to the hot metal surfaces and eventually becomes brown or even black. Subsequently large pieces of discoloured material may become detached and mix with the powder bulk. Some drying chambers are provided with vibrators or internal rotating mechanical devices to prevent such accumulations.

### **43.2.3.** Caking

Dried milk absorbs moisture very readily from atmosphere, and if packed in materials which are not impermeable to moisture such as plain paper sacks, cardboard cartons, etc., it may develop large hard lumps or severe caking during storage. High quality powder may be ruined by poor packaging. Low initial moisture content is also highly desirable. With modern properly managed plant, lumpy powder is more likely to arise from storage defects rather than from faulty manufacturing processes.

Crystallization of lactose is responsible for caking, as it causes the powder particles, largely consisting of lactose, to grow together (to sinter). Because water is needed for crystallization of  $\alpha$ -lactose, caking does not occur at low  $a_{\rm w}$ , say, below 0.4. At a higher temperature, crystallization can occur far more readily,  $a_{\rm w}$  being higher; moreover, the viscosity of the highly concentrated lactose solution (essentially the continuous phase of the powder particles) is lower, causing nucleation, hence crystallization, to be faster. Lactose crystallization also leads to protein insolubility, de-emulsification of fat and accelerated flavour deterioration.

The susceptibility to caking, especially high in whey powder, is considerably reduced if most of the lactose is crystallized before the drying (in the concentrate). Such precrystallized powder is usually called 'nonhygroscopic,' which may be a misnomer because the powder concerned does not attract less water (this is determined by its  $a_{\rm w}$  in relation to that of the air), but the consequences are less noticeable.

To avoid caking, it is necessary that suitable packaging materials providing barrier to passage of moisture are selected and the product is stored at low humidity levels.

# 43.2.4. Bulk Density

It has been observed that on storage, density of milk powders increases suggesting that on storage, the proportion of larger particles increase in the product.

# 43.3. PROPERTIES RELATED TO RECONSTITUTION OF DRY MILKS

The major defects in liquid milks prepared from milk powder are lumps, insoluble sediment, floating foam, charred particles, extraneous dirt and churned fat.

# 43.3.1. Insolubility Index and White Flecks:

The insolubles in milk powders are a lesser problem than poor reconstitution, although in practice the two terms are sometimes taken as synonymous. The term "insolubility index" instead of "solubility index" was introduced when the IDF modification of the original ADMI

method was issued. Insolubility index is often considered as one of the most important quality criteria. There are two types of insolubility:

§ The one caused by heating powder in the dry state, which decreases with increasing water temperature, as shown in Figure 43.1 and other caused by heating in the liquid (or concentrate) state, which does not improve with increasing water temperature.

It is a measure of the extent of denaturation of the proteins in milk powders.

# Fig. 43.1: Effect of water temperature on the solubility of whole milk powder

- § It is determined as the volume of the insoluble sediment after dissolving and centrifuging according to a prescribed procedure. The analysis of insolubles shows these to be a protein/fat/mineral complex, the fat probably being held by relatively weak chemical bonds. The protein itself is casein and/or denatured whey proteins.
- § The rate of insolubilization is dependent on both the moisture content and the storage temperature of the milk powders .
- § The insolubilization can be inhibited for all practical conditions of storage by maintaining the moisture level below 3%.
- § Normally beyond certain period of storage at elevated temperature viz. 40<sup>o</sup>C, the solubility index increases and such effect is much pronounced in high-heat powders.

The occurrence of insoluble sediment is undesirable, but to some extent unavoidable. The factors affecting solubility of milk powders are shown in Table 43.1. The main factor causing solubility defects is the particle temperature during the water removal process or the particle temperature history.

Table 43.1: Factors influencing the solubility of milk powders

#### Quality of milk:

- a. pH of the milk
- b. Free lactic acid
- c. Mineral balance
- d. Level and type of protein

### Processing and drying conditions:

- a. Conditions of preheating
- b. Excessive burn-on in the evaporator, caused by high acidity of milk or incorrect operation
- c. Excessive holding times for milk concentrates especially at elevated temperatures
- d. Subjecting partly-dried powder particles to high temperature air
- e. High inlet and outlet temperatures
- f. Failure to cool the powder before silo storage or packing

§ At a given temperature, the rate or denaturation of protein in concentrated milk doubles for every 5% increase in total solids upto about 92%. Most of the denaturation during the drying process occurs when the concentration of total solids is greater than 50%. The effect of concentrate total solids on insolubility is given in Table 43.2.

The occurrence of white flecks is of similar origin to insoluble sediment. Unlike insolubility, this defect can be detected visually but is difficult to determine quantitatively

Table 43.2: Percentage insolubility in water at 20°C of whole milk powder prepared under different spray drying conditions

Outlet temperature,	Concentra	te solids, % w/w	
<sup>0</sup> C	35	42	47
114	14	15	22
105	4	8	15
100	1	6	13
95	1	2	10
90	1	1	5
85	1	1	1

NB: Practical acceptable level is < 2%

# 43.1.1. pH / Acidity on reconstitution / recombination

This has influence on the chemical and functional characteristics of milk powders such as heat stability, viscosity and rennetability. The decrease in pH and increase in acidity could be attributed to the tying of amino groups of the protein in the Maillard reaction during storage of dried milks. Keeping aside the type of dried milk, higher the temperature of storage, higher the decline in pH of milk powders.

# 43.1.2. Viscosity on reconstitution

Storage conditions obviously exert an influence on the viscosity. Pre-heat treatment and the temperature of storage affect the viscosity. Low heat powders show faster and higher rise of viscosity at higher temperature of storage due to decrease in pH of the milk powders during storage.

#### 43.1.3. Free fat

The amount of free fat in dried milks varies appreciably depending upon the manufacturing method and storage conditions. It is lowest in spray dried powders and highest in roller dried ones. The figures for free fat as % of the total fat have been reported in the literature ranging from 1-20% in spray dried powder and in roller dried powders 91.6 - 95.8%.

Storage conditions may be important for the free fat content of milk powders. If lactose crystallizes, due to moisture absorption from surroundings, the free fat content increases

sharply. Higher temperature of storage has also the similar effect. A steep increase in the free fat of spray dried whole milk at an average moisture content of 5.3% is observed.

The presence of larger quantities of free fat tend to increase the susceptibility of fat oxidation and thus to decrease in keeping quality of dried milk. During storage of spray dried whole milk, the free fat on the particles surface is oxidized first, while the interior fat is protected by the dense mass of SNF. The spray dried whole milk, varying considerably in free fat content, is found to have no effect on the development of oxidation flavour during storage at 30  $^{\circ}$ C in the presence of air. The degree of oxidation of free fat is 5 to 8 times greater than that of the remaining fat in the dried milks containing high levels of polyunsaturated fatty acids. Contradictory reports are available regarding the faster oxidative deterioration of the product containing higher free fat.

# 43.1.4. Reducing capacity

On storage, the acid ferricyanide reducing capacity of milk powders increases and this results from interaction of lactose with protein (Maillard reaction) and probably also from decomposition of lactose catalysed by the buffer salts of milk. The degree of preheat treatment of milk prior to evaporation and drying has an influence on the ferricyanide reducing values of final powder. On storage the capacity of dried milk powders to reduce acid ferricyanide increases due to enhanced interaction between lactose and proteins.

#### 43.1.5. Production of water and carbon dioxide

On storage of dried milks, both carbon dioxide and water are produced as the Maillard reaction products. Carbon dioxide is produced from non lipid constituents of milk at a rate which depend upon the moisture and temperature of storage. Oxygen accelerates the rate of production of carbon dioxide. Moisture of powders increases during storage under conditions which permit that reaction to occur.

# 43.1.6. Heat stability on reconstitution

There is a general decline in heat stability and such decrease is higher in powders held at higher temperatures of storage and time. This could be attributed to decrease in pH of the powders during storage and associated marked increase in solubility index, either or both of which may contribute to a reduction in heat stability. It is known that a change of 0.05 of a pH unit may alter heat stability times substantially.

#### 43.1.7. Rennet coagulation time and curd tension on reconstitution

Reconstituted low-heat powders has an initial lower coagulation time as compared with medium-heat powder but the reconstituted high-heat powder do not coagulate when held for an extended period. The coagulation time of low-heat sample increase on storage period and by higher storage temperatures.

The curd tension of the reconstituted low-heat powder falls within medium firm range. The medium heat powders give a softer curd, the curd tension of such powders decrease with storage time being adversely influenced by higher storage temperature.

Both rennet coagulation time and curd tension are influenced by decrease in pH and affected by change in calcium sensitivity during storage of milk powders particularly at higher temperatures. It is known that the calcium and magnesium ion concentrations of reconstituted spray dried milk powders are somewhat lower than the levels found in raw milk and it is also established that there is decrease in the sensitivity of casein to calcium ions in case of milk powders stored in excess of 4  $^{0}$  C.

# 43.2. LOSS OF NUTRITIONAL VALUE

Loss of nutritive value during storage primarily concerns loss of available lysine due to Maillard reactions. Storage at 20 ° C at normal water content does not cause an appreciable loss; at 30 ° C, a loss of 12% after storing for 3 years has been reported. Extensive Maillard reactions cause a decrease in protein digestibility and formation of weak mutagens.

Extensive autoxidation results in formation of reaction products between hydroperoxides and amino acid residues and between carbonyl compounds and  $\epsilon$ -amino groups; this may cause the biological value of the protein to decrease slightly. Of greater concern is the loss of vitamin A in vitamin-fortified skim milk powder, due to its oxidation. This especially occurs if the vitamin preparation is dissolved in oil and then emulsified into the skim milk before atomization. Usually, dry added preparations are more stable. It is, however, very difficult to homogeneously distribute a minute amount of a powder throughout a bulk mass. Some of the important nutritional characteristics of dried milks as affected on storage are:

# 43.2.1. Quality of milk proteins

Non fat dry milk (3.3% moisture) when stored at 25 and 37  $^{\circ}$  C for one year it is observed that nitrogen digestibility, protein efficiency ratio (PER) and net protein utilization (NPU) are not affected. The proteolytic digestibility of proteins is significantly reduced in skim milk powders stored at 40  $^{\circ}$  C and 0.80  $a_w$  for one month.

#### 43.2.2. Available Lysine

- $\S$  Nonfat dry milk powders stored at 25 and 37  $^{\circ}$  C for one year when incorporated into diets limiting in lysine indicate that relative nutritive value decreases by 8% in samples at 25  $^{\circ}$  C and by 8, 9 and 8% in samples stored for 3, 6 and 12 months at 37  $^{\circ}$  C respectively. Chemically available lysine also shows similar small decreases in samples stored at 37  $^{\circ}$  C .
- § The major cause of deterioration in milk powder is the reaction between the free amino groups and the reducing sugars. Powders with less than 5% moisture show no decrease in biological value after storage at 28.5 ° C for 27 months suggesting that moisture content plays an important role in this reaction.
- $\S$  Storage of skim milk powder samples at 40 ° C and at low water activity or under dry conditions show no loss of available lysine. Storage at 40 ° C at 0.57 and 0.86  $a_{ww}$  for one month; the loss of tryptophan and arginine as being about 10%, but others such as proline and tyrosine decrease only slightly. considerably reduce the amount of methionine by 6 and 19% respectively. Losses of other essential amino acids are observed only after storage at 40 ° C and 0.80 a

#### 43.2.3. Vitamin Content

The effect on vitamins stability at various storage conditions reported in literature are as under:

- § Instantized nonfat dry milk stored for 20 years showed vitamins decrease to varying degree with thiamine showing the greatest loss (6%), riboflavin decreased by 50% whereas only a 2% loss occurred for niacin.
- § In one case a 33% reduction in the content of vitamin B6 was detected in skim milk powder after storage for 40 months.
- § In another study losses of B<sub>1</sub> and C were 10% after 2 years of storage.
- § The amount of vitamin C loss depends upon the oxygen and water vapour permeability of the packaging material. It is suggested that milk powders should also be protected against light to keep losses of the light sensitive vitamins, in particular riboflavin, as low as possible.
- $\S$  The vitamin A stability in skim milk powder was measured over a period of 16 weeks at 21, 26 and 32  $^{\circ}$  C . The samples kept in dark lost 20% of the vitamin activity at 21  $^{0}$  C, 27% at 26  $^{0}$  C and 38% at 32  $^{\circ}$  C .
- § Vitamin fortified milk powder stored at ambient temperature for 2 years showed no loss of thiamine, riboflavin, niacinamide, pyridoxine, ascorbic acid and L-tocopherol acetate. So it was concluded that these vitamins will also survive when milk powder is stored at higher temperature for short period i.e. for 6 months. Only vitamin A was substantially degraded.

On storage of milk powders, the digestibility of protein and the available lysine content have been found to decrease. Such decreases are dependent on the moisture content, storage period and storage temperature. Higher moisture and longer periods of storage at elevated temperatures have been shown to accelerate such deteriorations. The same has been found to be applicable to various vitamins in the product. It is suggested that to protect light sensitive vitamins, exposure of powders to such conditions should be avoided.

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## Module 17

# Definition and legal standards for dried milks

# Lesson-44

#### 44.1. INTRODUCTION

The dried milks along with infant milk foods, cereal based weaning foods are covered under FSSA and BIS. They are described here.

# 44.2 FOOD SAFETY AND STANDARDS (FOOD PRODUCTS STANDARDS AND FOOD ADDITIVES) REGULATIONS

**a. CREAM POWDER** - means the product obtained by partial removal of water from cream obtained from milk of cow and / or buffalo. The fat and / or protein content of the cream may be adjusted by addition and/ or withdrawal of milk constituents in such a way as not to alter the whey protein to casein ratio of the milk being adjusted. It shall be of uniform colour and shall have pleasant taste and flavour free from off flavour and rancidity. It shall also be free from vegetable oil/ fat, mineral oil, added flavour and any substance foreign to milk.

The product may contain food additives permitted in these regulations including Appendix A. It shall conform to the microbiological requirements prescribed in Appendix B. It shall conform to the following requirements:—

- (i) Moisture Not more than 5.0 percent
- (ii) Milk fat Not less than 42.0 percent
- (iii) Milk protein in Milk solid not fat Not less than 34.0 percent
- **b. MILK POWDER** means the product obtained by partial removal of water from milk of Cow and / or Buffalo. The fat and / or protein content of the milk may be adjusted by addition and/ or withdrawal of milk constituents in such a way as not to alter the whey protein to casein ratio of the milk being adjusted. It shall be of uniform colour and shall have pleasant taste and flavour free from off flavour and rancidity. It shall also be free from vegetable oil/fat, mineral oil, thickening agents, added flavour and sweetening agent. It may contain food additives permitted in these regulations including Appendix A. It shall conform to the microbiological requirements prescribed in Appendix B. It shall conform to the following requirements:—

Table-44.1: FSSA standards for milk powder

No.	Parameter	Whole Milk	Partly Skim Milk	Skim Milk
		Powder	Powder	Powder
1	Moisture	Not more than 4	Not more than 5 percent	Not more than
		percent m/m	m/m	5 percent m/m
2	Milk Fat	Not less than	Not less than 1.5 percent	Not more than
		26.0 percent	m/m and Not more than	1.5 percent
		m/m	26.0 percent m/m	m/m
3	Milk Protein in Milk	Not less than	Not less than 34.0	Not less than
	Solids Not Fat	34.0 percent	percent m/m	34.0 percent
		m/m		m/m
4	Titratable acidity (ml 0.1	Not more than	Not more than 18.0	Not more than
	N NaOH / 10g Solids	18.0		18.0
	Not Fat)			
5	Insolubility Index	Not more than	Not more than 2.0 ml	Not more than
		2.0 ml		2.0 ml
6	Total Ash on dry weight	Not more than	Not more than 8.2 per	Not more than
	basis	7.3 per cent	cent	8.2 per cent

#### c. FOODS FOR INFANT NUTRITION

1. **INFANT MILK FOOD** - means the product prepared by spray drying of the milk of cow or buffalo or a mixture thereof. The milk may be modified by the partial removal/substitution of different milk solids; carbohydrates, such as sucrose, dextrose and dextrins/maltodextrin, maltose and lactose; salts like phosphates and citrates; vitamins A, D, E, B Group, Vitamin C and other vitamins; and minerals like iron, copper, zinc and iodine.

The product shall be free of lumps and shall be uniform in appearance. It shall be free from starch and added antioxidants. It shall also be free from dirt, extraneous matter, preservatives and added colour and flavour and from any material which is harmful to human health. It shall not have rancid taste or musty odour. It shall not contain food additives. It shall conform to the following requirements, namely:—

Table-44.2: FSSA standards for infant milk food

_	Table-44.2: FSSA standards for infant milk food	
1	Moisture, per cent by weight (not more than)	4.5
2	Total milk protein, per cent by weight (not less than)	12.0
3	Milk fat, per cent by weight (not less than)	18.0
4	Total ash, per cent by weight (not more than)	8.5
5	Ash insoluble in dilute Hydrochloric acid, per cent by weight (not more than)	0.1
6	Solubility:	
	Solubility Index maximum	2.0 ml
	Solubility per cent by weight (not less than)	98.5
7	Vitamin A (as retinol) μg. per 100 g. (not less than)	350 μg
8	Added Vitamin D (expressed as Cholecalciferol or Ergocalciferol) µg per 100g.	4.5 μg
	(not less than)	
9	Vitamin C, mg per 100 g. (not less than)	35 μg
10	Thiamine, μg per 100 g. (not less than)	185 μg
11	Riboflavin, µg per 100 g. (not less than)	275 μg
12	Niacin, μg per 100 g. (not less than)	1160
		μg
13	Pyridoxine μg per 100 g. (not less than)	160 μg
14	Folic acid, μg per 100 g. (not less than)	20 μg
15	Pantothenic acid, mg per 100 g. (not less than)	1.4 mg
16	Vitamin B12, μg per 100 g. (not less than)	0.7 μg
17	Choline, mg per 100 g. (not less than)	32 μg
18	Vitamin K μg per 100 g. (not less than)	18 μg
19	Biotin, μg per 100 g. (not less than)	7.0 μg
20	Sodium mg per 100 g. (not less than)	90 μg
21	Potassium, mg per 100 g. (not less than)	370 mg
22	Chloride, mg per 100 g. (not less than)	250 mg
23	Calcium, mg per 100 g. (not less than)	230 mg
24	Phosphorous, mg per 100 g. (not less than)	115 mg
25	Magnesium, mg per 100 g. (not less than)	22 mg
26	Iron, mg per 100 g. (not less than)	5.0 mg
27	Iodine, μg per 100 g. (not less than)	20 μg
28	Copper, μg per 100 g. (not less than)	280 μg
29	Zinc, mg per 100 g. (not less than) and	2.5 mg
	not more than	5.0 mg
30	Manganese, μg per 100g. (not less than)	20 μg
31	Selenium, μg per 100 g. (not less than)	14 μg
32		10,000
33	Coliform count absent in	0.1 g
34	Yeast and mould count absent in	0.1 g
35	Salmonella and Shigella absent in	25 g
36	E. coli absent in	0.1 g
37	Staphylococcus aureas absent in	0.1 g

It shall be packed in hermetically sealed, clean and sound containers or in flexible pack made from film or combination or any of the substrate made of Board paper, polyethylene, polyester metallised film or in such a way to protect from deterioration. It may be packed in nitrogen or a mixture of nitrogen and carbon dioxide.

1. **INFANT FORMULA** - means the product prepared by spray drying of the milk of cow or buffalo or mixture thereof. The milk may be modified by partial removal/substitution of milk fat with vegetable oils rich in polyunsaturated fatty acids and/or by different milk solids; carbohydrates such as sucrose, dextrose and dextrins/ maltodextrin, maltose and lactose; salts such as phosphates and citrates; vitamins A, D, E, B and C group and other vitamins; minerals such as iron, copper, zinc and iodine and others. Vegetables oils rich in

polyunsaturated fatty acids shall be added to partially substitute milk fat to an extent that the product shall contain a minimum of 12 per cent by weight of milk fat and a minimum of linoleate content of 1.398 g per 100 g. of the product.

# It shall conform to

Table-44.3: FSSA standards for infant formula

1	Moisture, per cent by weight (not more than)	4.5
2	Total milk protein, per cent by weight (not less than) and	10.0
-	not more than	16.0
3	Total fat, percent by weight (not less than)	18.0
	Milk Fat, percent by weight (not less than)	12.0
	Linoleate per 100 gram (not less than)	1.398g
4	Total ash, per cent by weight (not more than)	8.5
5	Ash insoluble in dilute Hydrochloric acid, per cent by weight (not more than)	0.1
6	Solubility:	
	(a) Solubility Index maximum	2.0 ml
	(b) Solubility per cent by weight (not less than)	98.5
7	Vitamin A (as retinol) µg. per 100 g. (not less than)	350 μg
8	Added Vitamin D (expressed as Cholecalciferol or Ergocalciferol) µg. per 100g.	4.5 μg
	(not less than)	
9	Vitamin C, mg per 100 g. (not less than)	35 μg
10	Thiamine, µg per 100 g. (not less than)	185 μg
11	Riboflavin, μg per 100 g. (not less than)	275 μg
12	Niacin, µg per 100 g. (not less than)	1160
	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	μg
13	Pyridoxine μg per 100 g. (not less than)	160 μg
	Folic acid, µg per 100 g. (not less than)	20 μg
	Pantothenic acid, mg per 100 g. (not less than)	1.4 mg
	Vitamin B12, μg per 100 g. (not less than)	0.7 μg
17	Choline, mg per 100 g. (not less than)	32 mg
18	Vitamin K μg per 100 g. (not less than)	18 μg
19	Biotin, μg per 100 g. (not less than)	7.0 μg
20	Vitamin E (as a-tocopherol compounds) IU per 100g. (not less than)	3.15 IU
21	Sodium mg per 100 g. (not less than)	90 mg
22	Potassium, mg per 100 g. (not less than)	370 mg
23	Chloride, mg per 100 g. (not less than)	250 mg
24	Calcium, mg per 100 g. (not less than)	230 mg
25	Phosphorous, mg per 100 g. (not less than)	115 mg
26	Magnesium, mg per 100 g. (not less than)	22 mg
27	Iron, mg per 100 g. (not less than)	5.0 mg
28	Iodine, μg per 100 g. (not less than)	20 μg
	Copper, µg per 100 g. (not less than)	280 μg
30	Zinc, mg per 100 g. (not less than) and	2.5 mg
	not more than	5.0 mg
31	Manganese, μg per 100g. (not less than)	20 μg
32	Selenium, μg per 100 g. (not less than)	14 μg
33	Bacterial count, per g. (not more than)	10,000
34	Coliform count absent in	0.1 g
35	Yeast and mould count absent in	0.1 g
36	Salmonella and Shigella absent in	25 g
37	E. coli absent in	0.1 g
38	Staphylococcus aureas absent in	0.1 g

#### d. MALTED AND MALT BASED FOODS

1. MALTED MILK FOOD - means the product obtained by mixing whole milk, partly skimmed milk or milk powder with the wort separately from a mash of ground barley malt, any other malted cereal grain and wheat flour or any other cereal flour or malt extract with or without addition of flavouring agents and spices, emulsifying agents, eggs, protein isolates, edible common salt, sodium or potassium bicarbonate, minerals and vitamins and without added sugar in such a manner as to secure complete hydrolysis of starchy material and prepared in a powder or granule or flake form by roller drying, spray drying, vacuum drying or by any other process. It may contain cocoa powder. It shall be free from dirt and other extraneous matter. It shall not contain any added starch (except starch natural to cocoa powder) and added non-milk fat. It shall not contain any preservative or added colour. Malted milk food containing cocoa powder may contain added sugar. Malted milk food shall also conform to the following standards, namely:—

Table-44.4: FSSA standards for malted milk foods

Sr.	Parameter	Malted milk food	Malted milkfood with
No.		without Cocoa powder	cocoa powder
(a)	Moisture	Not more than 5 per cent	Not more than 5 per
(90%)		by weight.	cent by weight
(b)	Total protein	Not less than 12.5 per	Not less than 11.25 per
	(N x 6.25) (on dry basis)	cent by weight.	cent by weight.
(c)	Total fat (on Dry basis)	Not less than 7.5% by	Not less than 6% by
		weight	weight
(d)	Total ash (on dry basis	Not more than 5% by	Not more than 5% by
		weight	weight.
(e)	Acid insoluble ash (on dry basis)	Not more than 0.1 per	Not more than 0.1 per
	(in dilute HCl)	cent by weight	cent by weight
(f)	Solubility	Not less than 85% by	Not less than 80% by
		weight.	weight.
(g)	Cocoa powder (on dry basis)	-	Not less than 5.0% by
			weight.
(h)	Test for starch	Negative	-
(i)	Bacterial count	Not more than 50,000 per	Not more than 50,000
		gram.	per gram.
(j)	Coliform count	Not more than 10 per	Not more than 10 per
		gram	gram.
(k)	Yeast and mould count		absent in 0.1 gm
(1)	Salmonella and Shigella		absent in 0.1 gm
(m)	E.Coli		absent in 0.1 gm
(n)	Vibrio cholera and		absent in 0.1 gm
1000000	V.Paraheamolyticus		
(0)	Faecal streptococci and		absent in 0.1 gm
	Staphylococcus aureas		

2. MALT BASED FOODS (MALT FOOD) - means the product obtained by mixing malt (wort or flour or malt extract) of any kind obtained by controlled germination of seeds (cereals and/or grain legumes), involving mainly steeping germination and kiln drying processes with other cereal and legume flour with or without whole milk or milk powder, flavouring agents, spices, emulsifying agents, eggs, egg powder, protein isolates, protein hydrolysates, edible common salt, liquid glucose, sodium or potassium bicarbonate minerals, amino acids and vitamins. It may contain added sugar and/or cocoa powder and processed in such a manner to secure partial or complete hydrolysis of starchy material in the form of powder or granules or flakes by drying or by dry mixing of the ingredients. The grains, legumes and their products used in preparation of malt shall be sound, uninfested and free from insect fragments, rat excreta, fungal infested grains or any other type of insect or fungal damage.

It shall also conform to the following standards, namely:—

Moisture Not more than 5 per cent, by weight Not less than 7.0 per cent, Total Protein (N x 6.25) (on dry basis) by weight Total ash (on dry basis) Not more than 5 per cent, by weight Acid insoluble ash (in dilute HCl) Not more than 0.1 per cent, by weight Not more than 50,000 per Total plate count gram Coliform count Not more than 10 per gram. Yeast and Mould Count Not more than 100 per gram. Absent in 10 gram. Salmonella and Shigella Absent in 25 gram Alcoholic Acidity (expressed as H2SO4) with 90 per cent Not more than 0.30 per alcohol (on dry weight basis) cent. Vibrio cholera and V.Paraheamolyticus absent in 0.1 gm Faecal streptococci and Staphylococcus aureas absent in 0.1 gm

Table-44.5: FSSA standards for malt based foods

The details of these and other following legal standards as per The Food Safety and Standards Regulations, 2011 for dried milk products can be referred on web site http://www.fssai.gov.in/

- (i) PREMATURE/LOW BIRTH WEIGHT INFANT MILK SUBSTITUTES
- a. Lactose free infant milk substitute
- b. Lactose and sucrose free infant milk substitute
- c. Sucrose free infant milk substitute
- d. Hypoallergenic infant milk substitutes

- (ii) PROCESSED CEREAL BASED COMPLEMENTARY FOOD COMMONLY CALLED AS WEANING FOOD OR SUPPLEMENTARY FOOD
- (iii) FOLLOW-UP FORMULA-COMPLEMENTARY FOOD

# 44.3. BIS REQUIREMENTS FOR MILK POWDERS

**44.3.1. BIS SPECIFICATION FOR MILK POWDER:** (IS: 1165-1975, 1992, 2002 (Fourth revision))

The bulk of the milk powder at present available in the market is prepared by two well-known commercially established processes, known as the "ROLLER DRYING PROCESS" and the "SPRAY DRYING PROCESS."

In the ROLLER DRYING process, milk, either in its original state or after condensation under vacuum, is allowed to run in a thin film on to steam-heated metal rollers, which are kept revolving at a low speed. The rollers are heated internally by means of superheated steam. The milk becomes dry before a revolution is completed, when the dried film is removed by means of a metal scraper blade placed at an angle to the surface of the roller. The dried product is afterwards brocken-up and sieved.

In the SPRAY DRYING process, milk usually precondensed, is sprayed into a large drying chamber, heated air being drawn into the chamber at the same time. The milk being in a finely divided state, dries almost immediately and falls as a dry powder on to the floor of the chamber.

The method of drying has a fundamental influence on the solubility of the milk powder. Due to the severe heat treatment to which the milk is subjected in the roller drying process, the solubility of the material prepared by this process is reduced by the greater denaturation of the proteins. Separate limits have, therefore, been fixed in this standard for roller dried and spray dried milk powders.

A major defect in the case of milk powder (whole) is the development on storage of a flat flavour, which later becomes tallowy and unpleasant. This is caused by oxidation of the fat present, which commences slowly but rapidly accelerates thereafter. Milk powder (whole) manufactured by the spray drying process is particularly prone to oxidation, through air entrapped inside the particles. Accordingly, with the trade practices prevailing in the country, this standard specifies that spray dried milk powder (whole) shall be packed in nitrogen or a mixture of nitrogen and carbon dioxide.

# 44.3.2. REQUIREMENTS OF MILK POWDERS

The milk powder shall be white, or white with greenish tinge or light cream in colour. It shall be free from lumps except those that break up readily under slight pressure and shall be reasonably free from scorched particles. It shall be also free from dirt and other extraneous matter.

The product shall be processed and packed in premises maintained under hygienic conditions. It shall also be stored and distributed under hygienic conditions.

The material shall be packed in hermatically sealed, clean, sound tin containers, unless otherwise agreed to between the purchaser and the supplier, in such a way as to protect it from deterioration except that WMP, when manufactured by spray drying process, shall be packed in nitrogen or CO<sub>2</sub>.

Partly Skim Milk Powder and Skim Milk Powder may be packed in bags of food grade polyethylene of minimum thickness 0.05 mm. The polyethylene bags should subsequently be encased in multi-walled kraft paper, such as crepe kraft paper bags of 80 GSM ( $g/m^2$ ) grade lined with hessian cloth having a mass of 270  $g/m^2$  and having two inner layers of plain kraft paper of 80 GSM ( $g/m^2$ ) grade.

The following particulars shall be marked or labelled on each container:

- 1. Name and type of material
- 2. Name and address of the manufacturer
- 3. Batch or code number
- 4. Month and year of manufacture
- 5. Net mass
- 6. Process of manufacture (Spray dried or Roller dried)
- 7. Expiry date
- 8. Any other requirements under the Standards of weight & Measure (Packaged Commodities) Rules and PFA Rules

The container may also be marked with the ISI certification mark.

44.3.3. BIS Requirements for Milk Powders Quality

Table-44.6: BIS Requirements for Milk Powders Quality

Sr.	Characteristics	Requirements for Milk Powders			
No.		Whole	Partly Skim	Skim	Extra Grade
1	Flavour and Odour	Clean	Clean	Clean	-
2	Moisture, % by mass, Max.	4.0	4.0	4.0	3.5
3	Total Solids, (Milk solids and	96.0	96.0	96.0	96.5
	added salts), % by mass, Min.				
4	Insolubility Index, Max.				
	(a) Roller Dried	15.0 ml	15.0 ml	15.0 ml	-
	(b) Spray Dried	2.0 ml	2.0 ml	2.0 ml	0.5 ml
5	Total Ash, (on dry basis), % by	7.3	8.8	8.2	7.3
	mass, Max.				
6	Fat, % by mass	NLT	NLT 1.6 &	NMT 1.5	NMT 1.25
		26.0	NMT 24.0		
7	Titratable Acidity (Lactic	1.2	1.4	1.5	19.5 ml of 0.1
	acidity), % by mass, Max.				N NaOH
8	Lactate content, mg/g., Max.	-	-	-	1.5
9	Scorched particles, Max.	-	-	-	15 mg
					equivalent to
					Disc B
10	Bacterial Count / g., Max.	50,000	50,000	50,000	-
11	Coliform counts, per g, Max.	90	90	90	-

NLT- Not Less Than, NMT- Not More Than

#### 44.3.4. Specifications for Malted Milk Food (IS: 1806-1975, Reaffirmed 2000)

Table 44.7: Specifications for Malted Milk Food

Sr.	Characteristics	Grade I	Grade II
No.		(skimmed)	(Partly skimmed)
1	Moisture, % by wt., Max.	4.0	4.0
2	Total Protein, (Nx6.25) (on dry basis) % by mass, Min.	13.0	11.5
3	Fat on dry basis, % by mass, Min.	7.5	6.0
4	Total Ash (on dry basis) % by mass, Max.	5.0	5.0
5	Acid Insoluble Ash (on dry basis), % by mass, Max.	0.1	0.1
6	Solubility, % by mass, Min.	85	85
7	Cocoa powder (on dry basis) % by mass, Min.	-	5.0
8	Test for starch	Negative	Negative
9	Bacterial Count, per g., Max.	50,000	50,000
10	Coliform Count, per g., Max.	10	10

# 44.3.5. Specifications for Processed Cereal Weaning Food (IS: 1656-1985, 1997)

Table 44.8: Specifications for Processed Cereal Weaning Food

Sr.	Characteristics	Requirement
No.		
1	Moisture, % by wt., Max.	5.0
2	Total Protein, (Nx6.25) (on dry basis) % by wt., Min.	12.0
3	Fat, % by wt., Max.	7.5
4	Total carbohydrates, % by wt., Min.	55.0
5	Total Ash, % by wt., Max.	5.0
6	Ash insoluble in HCl, % by wt., Max.	0.1
7	Crude fibre (on dry basis), % by wt., Max.	1.0
8	Vit. A (as retinol, µg/100 g, min.	35.0
9	Vit. C, mg/100 g., Min.	25.0
10	Added Vit. D, µg/100 m.	5.0
11	Thiamine (as hydrochloride) mg/100 g, Min.	0.5
12	Riboflavin, mg/100 g, Min.	0.6
13	Nicotinic acid, mg/100 g, Min.	5.0
14	L-ascorbyl palmitate, mg/kg of fat, Max.	5.0
15	Iron, mg/100g, Min.	5.0
16	Bacterial Count, per g, Max.	40,000
17	Coliform Count	Absent in 0.1 gn

#### INTERNATIONAL STANDARDS

 $The international standards\ prescribed\ by\ American\ Dairy\ products\ Institute\ (ADPI)\ and\ Codex\ Alimentarius\ can\ be\ referred\ on\ the\ web\ sites:$ 

- 1. http://www.adpi.org
- $2. \quad \textbf{http://www.codexalimentarius.org}$

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## **Module 18**

# Manufacture of infant foods, malted and formulated dried products

# Lesson-45

# Human milk and cow milk

#### **45.1. INTRODUCTION**

Milk is nature's ideal food f or infants and growing children, except in rare cases of lactose intolerance. After arrival on this earth and during the first phase of growth, a human baby thrives on milk. In fact, mother's milk is not only desirable but essential from the time the child is born and hence the baby is recommended to be breast-fed until it is normal. It has always been an accepted fact that the best food for babies younger than six months is mother's milk due to the composition and content of various vitamins and essential amino acids. Human milk not only provides the infant with essential nutrients required during a critical growth period but also protect the new born from disease .

The requirements for an infant for proper growth include carbohydrate, fat, protein, vitamins, minerals and water. The concentration of these nutrients varies according to the needs of the off-spring, which depends upon maturity of birth, rate of growth, digestive system and environment it is born in to. In each species, the milk meets the specific needs of the off-spring. The milk of one animal can not be a real substitute for that of another. Hence, the most ideal situation for infant feeding would be to feed the child with human milk during the early stage of his growth. However, situation may arise when a human baby may be required to be given milk other than from his mother.

Many babies are fed with ordinary cow's milk to which some water and sugar are added. The widespread availability and commercial promotion of prepared infant formulas has led to a progressive decline in breast feeding throughout the world. For this reason, the WHO not only has sought to encourage breast feeding in the developing countries, but also has passed an International Code of Marketing of Breast Milk Substitutes.

**Infant formula** as per CODEX standards is a breast-milk substitute specially manufactured to satisfy, by itself, the nutritional requirements of infants during the first months of life up to the introduction of appropriate complementary feeding. The product is so processed by physical means only and so packaged as to prevent spoilage and contamination under all normal conditions of handling, storage and distribution in the country where the product is sold.

# 45.2. HUMAN MILK

Chemical composition of human milk differs from that of cow or buffalo milk as shown in **Table 45.1 & 45.2** below:

Table-45.1: Chemical composition of human, cow and buffalo milks

No.	Components, g/100 ml	Human	Cow	Buffalo
1	Proteins	1.2	3.3	4.2
2	Fat	3.8	4.1	7.0
3	Carbohydrate	7.0	4.5	5.1
4	Ash	0.21	0.72	0.82

(Ganguli, 1977)

Table-45.2: Major differences in the make up of different constituents of human, cow and buffalo milks

No.	Components	Human	Cow	Buffalo
1	Proteins			
	<ul> <li>Caseins, % of total protein</li> </ul>	40	82.3	75.6
	<ul> <li>Whey proteins, % of total protein</li> </ul>	60	17.7	24.4
	■ Immunoglobulins (g/100 ml)	1.0-1.5	0.5	NA
	<ul> <li>Lactoferrin (mg/ml)</li> </ul>	1.39	0.09	0.32
	<ul> <li>Lysozyme (mg/100 ml)</li> </ul>	40	0.013	NA
2	Lipids			
	<ul> <li>Oleic acid (mg/lit)</li> </ul>	14.9	10.3	9.5
	<ul> <li>Linoleic acid (mg/lit)</li> </ul>	3.4	0.8	0.5
	<ul> <li>Linolenic acid (mg/lit)</li> </ul>	Traces	0.2	0.3
	<ul> <li>Arachidonic acid (mg/lit)</li> </ul>	0.33	Traces	NA
	<ul> <li>Cholesterol (mg/lit)</li> </ul>	0.2	0.14	NA
3	Lactose	6.9	4.8	5.1
4	Minerals			
	■ Total (%)	0.21	0.68	0.82
	■ Ca (mg/100 ml)	31	128	210
	■ P (mg/100 ml)	15	87	128
	■ Fe (mg/100 ml)	0.15	0.14	6.2
5	Vitamins & Growth factors			7
	■ Vit A (mg/100 ml)	0.05	0.03	0.05
	• Vit E (mg/100 ml)	0.54	0.07	NA
	<ul> <li>Nicotinic acid (mg/100 ml)</li> </ul>	0.24	0.08	0.1
	<ul> <li>Bifidus factor (units)</li> </ul>	40	1	NA

NA – Data not available

(Mathur & Pahwa, 1980)

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# 1. Carbohydrates

Human milk contains higher carbohydrates (lactose) than cow or buffalo milk and hence 40% of the total energy of human milk is supplied by this constituent of milk which is only 29% in case of cow milk. Moreover, presence of non-lactose oligosaccharides such as bifidus stimulating factors in human milk enables the growth of healthy microflora in the intenstines.

# 2. Fats/lipids

Fat present in human milk provides essential fatty acids (required for nervous system development and prostaglandin synthesis), fat soluble vitamins and sterols. A unique feature of human milk is that increase in level of fat during feeding to infant serves as an appetite control mechanism for the infant. Almost an equal amount of fat is present in cow and human milks; however, qualitatively they differ widely in their fatty acid make-up as shown in Table 45.3.

Table 45.3: Fatty acid composition (% wt.) of human and cow milks

No.	Fatty acid	Human milk	Cow milk
1	4:0 Butyric	-	3.3
2	6:0 Caproic	Trace	1.6
3	8:0 Caprylic	Trace	1.3
4	10:0 Caprolic	1.3	3.0
5	12:0 Lauric	3.1	3.1
6	14:0 Myristic	5.1	14.2
7	16:0 Palmitic	20.2	42.9
8	16:1 Palmitoleic	5.7	3.7
9	18:0 Stearic	5.0	5.7
10	18:1 Oleic	46.4	16.7
11	18:2 Linoleic	13.0	1.6
12	18:3 Linoleneic	1.5	1.8
13	Unsaturated / Saturated Ratio	1.92	0.32

(Friend et al., 1983)

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<sup>§</sup> Human milk fat is rich in polyunsaturated fatty acids (PUFA) (notably linoleic acid- an essential fatty acid- about 7 to 8 times higher) which play an important role in brain development.

<sup>§</sup> Moreover, two important saturated fatty acids - myristic and palmitic - along with stearic acid are preferentially esterified at the beta position in human milk fat.

<sup>§</sup> The presence of comparatively high content of palmitic acid in position Sn-2 increases the digestibility of human milk fat. As a consequence of this, lipids from human milk are more

easily absorbed and excretion of fat by infants fed with breast milk is only about half to that of bottle-fed infants.

- § In addition, human milk fat contains easily digestible oleic acid in higher proportion while cow milk fat contains excess of butyric acid which may give rise to fatty diarrhea (steatorrhea).
- § Higher content of 18:1 in human milk also enables improved retention of calcium in breastfed infants.
- § Absence of orotic acid in human milk makes it less prone to occurrence of atherosclerotic plaques in breast-fed infants.
- § Over and above these differences, human milk has been shown to have higher content of unsaponifiable matter, higher ratio of phosphatidyl ethanolamine to sphingomylein and predominancy of phosphatidyl inositol.

Because of the differences in fatty acid make up of cow or buffalo milk from that of human milk, it has been proposed that all infant foods should contain PUFA rich vegetable oils so as to provide a minimum of 300 mg/k cal of linoleic acid in the formulas.

#### 2. Proteins

The characteristics of the human milk as compared to cow milk are shown in Table 45.4:

No.	Cow milk	Human milk
1	Caseins $(\alpha, \beta, \gamma, \kappa)$ : 80%	Caseins $(\alpha, \beta, \kappa)$ : 25%
2	Whey proteins ( $\alpha$ -lactal burnin, $\beta$ -	Whey proteins ( $\alpha$ -lact albumin, lact of errin
	lactoglobulin & others: 20 %	& others): 75 %

Table 45.4: Protein components of cow and human milk

- $\S$  The whey proteins are richer in essential amino acid content (PER = 3.11) compared to caseins (PER=2.50), hence nutritional requirements of infant could be met from relatively lower amount of good quality proteins, typical of human milk .
- $\S$  Human milk has a higher proportion of  $\alpha$ -lactalbumin but no  $\beta$  lactoglobulin.  $\alpha$ -lactalbumin, the most abundant protein of human milk, is of major importance to the infant from a nutritional view as it serves as a source of essential amino acids for the infant as well as acting as a crucial component of an enzyme system that biosynthesizes lactose.
- § The antimicrobial proteins of human milk (lactoferrin, lysozyme and IgA) account for 75% of the protein in human colostrum, versus 39% in mature human milk and less than 0.1% in cow milk.

- § The amino acid profile of human milk has unique ratio (0.76) between cystine and methionine as against 3.1 in case of cow milk.
- § The addition of demineralized whey to adjust the casein to whey protein ratio of cow milk to that of human milk changes the ratio to 1.4.
- § Human milk contains 13 g free amino acids of which 4 g are essential amino acids for the human infant. Cow milk contains 33 g free amino acids of which 16 g are essential.
- i. Glutamic acid accounts for ~ 40% of the free amino acid pool in human milk.
- ii. Taurine is the next major constituent of the free amino acid pool in human milk, but is only in trace amounts in cow milk.
- iii. Taurine has been postulated to play a major role in fat utilization and brain development in the infant as well as in stimulating "in vitro" activity of pancreatic lipases.
- $\S$  The proteins most concerned with the nutritional activities are the caseins,  $\alpha$ -lactalbumin and the folate and vitamin B12- binding proteins. There are also fat globule membrane proteins,  $\beta$  microglobulin and corticosteroid binding proteins.
- § In several brands of infant foods presently available, the protein content is adjusted to around 1.6% (similar to human milk) but still the casein content is 75% of the total protein. Use of demineralized whey is helpful, however, in adjustment of casein to whey protein ratio (40:60) similar to that in human milk. However, yet there is an ample scope for making the infant foods similar to human milk with respect to various metabolic responses.

# 2. Minerals:

Human milk possesses a mineral content which is less than one third than in cow/buffalo milk and calcium to phosphorus ratio of 2.1 as compared to about 1.5 in cow and 1.6 in buffalo milk. The low mineral content results in a low renal solute load.

#### 45.3. STRIKING FEATURES OF HUMAN MILK

The protein and non-protein nitrogen fractions of human and cow milk are shown in Table 45.5. The striking features of human milk when fed to infants are as under:

- 1. Digestion of milk by a baby is speedy due to formation of soft curd.
- 2. Quite less load on the kidney due to low calcium content.
- 3. Easy digestion and faster absorption of casein-due to smaller size of casein micelles.
- 4. Proper development of brain constituents due to higher lactose content and presence of relatively more amount of PUFA.

Bactericidal and anti - infective properties - due to presence of lysozyme, secretory IgA, bifidus factor and lactoferrin

Table 45.5: Protein and Non-protein N components in Human and Cow milk

No.	Component,	Human Milk, g/l	Cow, g/l
1	Casein	2	24
2	α	0.18	16-21
3	αs	-	12-17
4	β	1.28	8-10
5	κ	0.54	3
6	γ	-	1
7	Whey proteins	6	7
8	α-lactalbumin	3	1
9	β-lactoglobulin	0	4
10	Lactoferrin	1.7	Trace
11	Lysozyme	400	0.13
12	Serum albumin	0.5	0.4
	■ Ig A	1.0	0.03
	■ Ig G	0.03	0.6
	■ Ig M	0.02	0.03
13	Amino acids	13	33
	<ul><li>Essential</li></ul>	4	16
	■ Taurine, mg	42.1	1.2
14	Non-Protein Nitrogen	0.5	0.3

(Friend et al., 1983)

# 45.5.1. Host – resistant factors in human milk

- 1. Immunoglobulins (Ig) (Milk antibodies): The most abundant Ig of human milk is IgA versus Ig G in cow milk. It provides the infant with protection against enteric infection and serves as an "intestinal paint" to prevent the passage of various foreign proteins and bacteria from the intestinal tract into circulation. Milk antibodies function as specific host resistance factors and provide crucial immunological protection until the new born infant's defense system can be established.
- 2. **Lactoferrin** (**LF**): Human milk contains from 3 to 100 times as much iron binding protein LF as cow milk. LF is active "in vitro" against enteropathogenic *E. coli*, *Vibrio cholerae*, *St. mutans* and *Candida albicans*, presumably by chelating iron and making it unavailable for microbial growth.
- 3. **Lactoperoxidase** (**LP**): In cow milk, LP serves as a major antimicrobial agent; it catalyzes the oxidation of thiocyanate by  $H_2O_2$  to hypothiocyanate. It is active against several microorganisms. Although human milk LP survives gastric digestion "in vitro" its low concentration in milk may preclude any significant antibacterial role in infant intestine.

- 4. **Lysozyme:** It cleaves the cell wall peptoglycan of a number of Gram positive and Gram negative microorganisms and appears to act with complement to potentiate the activity of IgA against *E. coli*, and with peroxide and ascorbate to lyse *E. coli* and salmonella. It also plays a role in protecting against various viruses. Human milk contains ~ 3000 times as much lysozyme as cow milk.
- 5. **Bifidobacteria:** Protect the infant against disease by producing volatile acids which inhibit the proliferation of pathogenic microorganisms in the gut. Within 3-4 days after birth, the intestinal tract of breast fed infant contains up to 99% *Bifidobacterium bifidum* Type IV whereas the formula -fed infants do not contain Type IV but rather 30-40% *B. bifidum* Type II. A number of substances in human milk have been reported to stimulate the proliferation of the bifidobacteria:
- a. **Buffering capacity:** Because of low buffering capacity of human milk, the pH of intestine drops, making growth conditions favourable for bifidobacteria and unfavourable for others.
- b. Lactulose: Not present in human milk or cow milk, but when milk is sterilized (as in case of sterilized formulas)  $\sim 1.5\%$  of the total lactose is converted to lactulose during heating. It encourages the growth of bifidobacteria.
- c. **Lactoferrin:** Indirectly promotes the growth of bifidobacteria by inhibiting the growth of competing *E. coli*.
- d. **Pantothenic acid**: Derivatives of this acid stimulate one strain of *B. infantis*.
- e. **Oligosaccharides and glycoproteins:** Stimulate the growth of *B. bifidum* var: *Pennsylvanicus*. Human milk contains ~ 30 to 100 fold more stimulatory activity than cow milk.
- f. **Others:** Human milk contains fatty acids, monoglycerides, triglycerides, free fatty acids, copper, orosomucoid, influenze virus hemagglutination inhibitor, etc. which help an infant to get protection from several pathogenic organisms and viruses.

Thus the presence of bifidus factor in human milk is important as it helps in establishment of bifidobacteria in the intestine and in maintaining lower pH of the stool, better absorption of minerals, synthesis of certain B-complex vitamins, resistance to enteropathogenic bacteria, detoxification in chronic liver diseases, etc.

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# Lesson-46

# **HUMAN MILK SUBSTITUTES**

# **46.1. INTRODUCTION**

While human milk is superior for the new born infant, human milk substitutes (HMS) play a necessary role in infant nutrition when breast feeding is not possible, desirable o r sufficient.

#### **46.2. HUMAN MILK SUBSTITUTE**

Since the advent of first commercial formula developed by scientists of Nestlé's research group in 1866, a wide range of infant formulas have been developed by the manufacturers as shown in **Table 46.1.** 

Table 46.1: Range of infant formulas available in the market

No.	Type of Formula		Description		
1	Standard Formula	•	Use of cow/buffalo milk to replace / supplement human milk.		
			Adjustments made for protein : fat : carbohydrate ratio.		
			Vitamins and iron fortified.		
2 Soy-based milk-free • Use of soy flour		٠	Use of soy flour or water soluble soy protein isolate.		
	(SBMS) Formula		Useful to infants allergic to milk proteins, having lactose		
			deficiency and who show signs of galactosemia.		
3	Specialized /	•	Meant for infants having a variety of nutritional problems		
	Predigested		due to digestive malfunctions, physiological		
	Formula		underdevelopment or gastro intestinal infections.		
			Usually nitrogen is supplied through hydrolyzed proteins, $$		
			energy through easy to assimilate simple carbohydrates,		
			with minimal load on digestive enzyme.		
4	Humanized /	•	Development of nutritionally superior formulas having		
	Materialized		varying degree of chemical and biochemical similarity of		
	Formula		$\alpha s\text{-case}\textsc{in}$ and $\beta\text{-lg}$ from cow/buffalo milk.		
	•		Adjustment of casein: whey protein ratio, PUFA and mineral contents.		

However, there are many limitations of these formulas as far as their use for infant feeding is concerned. These include the following:

- (1). High casein low whey protein character of standard formula results in lower nitrogen retention, lower weight gains in premature infants, low blood serum albumin levels, higher urea level and increased renal osmolar loads.
- (2). Absorption of dietary fat is lower and makeup of adipose tissue fat is different when qualitatively fat is not similar to human milk fat.
- (3). Lower absorption of calcium and iron.
- (4). Overgrowth of *E. coli* in intestine and hence alkaline pH of stools, lower resistance of infant to enteropathogenic bacteria, deconjugation of bile salts leading to reduced fat absorption and higher urinary excretion of tryptophan.
- (5). Lack of in-built self regulating mechanism available in breast-fed infants.
- (6). More common occurrence of atherosclerotic plaques in bottle-fed infants due to high levels of cholesterol as orotic acid is absent in human milk .
- (7). Loose and malodorous stools when soy flour is used in SBMF formula.
- (8). Hemolytic form of anemia due to Vitamin E deficiency.
- (9). Inhibition of trypsin, enlargement of pancreas and goitogenic effects due to presence of residual activity of antinutritional factors in inadequately processed soy beans.

To overcome these limitations, a new generation of nutritionally superior formulas has been developed, having varying degree of chemical and biochemical similarity with human milk. There are two types of approaches tried for this:

- (1). Removal/reduction from cow/buffalo milk substances like  $\alpha$ s-casein and b-lg through enzyme treatments. However,  $\alpha$ s-casein is hydrolyzed in the digestive tract of the infant and thus formula containing hydrolyzed casein may not serve any useful purpose nutritionally.
- (2). Modifications justifiable from nutritional angle-for example adjustment of casein: whey protein ratio in cow / buffalo milk so as to simulate human milk . Infants fed this have shown comparable levels of blood serum albumin, blood urea, nitrogen retention and urinary osmolarity as breast fed infants.

From nutritional point of vie w, following suggestions are important for humanized milk formula:

- (1). Modification of proteins of cow / buffalo milk to make them similar to human milk (whey protein: casein=60:40). This will help in enhancing nitrogen utilization and in minimizing renal osmolar load.
- (2). Modification of triglyceride makeup of cow / buffalo milk fats so as to simulate ratios of short, medium and long chain triglycerides as well as that of saturated to unsaturated fatty acids to that of human milk fat. This will enhance linoleic acid and vitamin E contents.

- (3). Adjustment of proteins: fat: carbohydrate ratio. This will reduce the protein content and increase the carbohydrate content of cow / buffalo milk and make it similar to that of human milk.
- (4). Modification of calcium: phosphorous ratio as well as reduction in total ash content. This will enhance mineral utilization and minimize renal osmolar loads.
- (5). Enrichment of cow / buffalo milk with factor to support growth of *B. bifidum* in intestines.
- (6). Enrichment of cow / buffalo milk with lactoferrins, transferrins and immunoglobulins to promote resistance towards enteropathogenic bacteria and to enhance iron absorption.
- (7). Partial substitution of cow / buffalo milk f at with oils rich in PUFA, as well as use of carbohydrate sources such as maltodextrins may help in reducing the cost.

# 46.3. ACHIEVEMENTS SO FAR FOR HUMANIZATION OF BUFFALO MILK:

- (1). αs-casein reduced to human milk level by renneting.
- (2). Total protein concentration reduced to human milk level by proteolysis.
- (3). Calcium level reduced by electrodialysis
- (4). Curd tension reduced by ion-exchange
- (5). Level of unsaturated f atty acids improved by corn oil fortification with milk f at.

#### 46.4. RESEARCH ON FORMULATING HUMAN MILK SUBSTITUTES

Modification of cow/buffalo milk properties: Allergic reactions to cow milk are mainly due to b -lactoglobulin in addition to caseins, α-lactalbumin, serum albumin, immunoglobulins and digests of these proteins. To overcome these, b -lg is removed by gel filtration and the remainder of the proteins blended; caseins may be hydrolyzed followed by gel filtration to prepare a non-antigenic fraction. Ganguli and Kuchroo (1979) prepared humanized infant formula from buffalo milk containing low levels of antigenic αs–casein (**Figure 46.1**).

Fig- 46.1: Preparation of humanized buffalo milk powder (Ganguly and Kuchroo, 1979)

(1). Adjustment of fatty acid composition: Ratio of unsaturated fatty acids (UFA) and saturated fatty acids (SFA) adjusted to 1.92 (similar to human milk) by incorporation of soybean, oleo and safflower oils (rich in UFA) and coconut and milk fat (rich in SFA). Corn oil may be added to increase oleic acid. Blending of milk fat and vegetable fats enables in improving digestibility of infant formulas. Use of sunflower oil as well as one third milk fat replacement by maize oil has been reported.

- (2). Use of goat milk: Major components of goat milk (3% fat, 3% protein 4.5% lactose, 0.8% ash) resemble cow milk in composition, however, levels of potassium, chloride and biotin are higher than in cow milk and folic acid content is less (lack of folic acid may lead to anemia in infants). Goat milk lacks in  $\alpha$ s-casein but  $\alpha$ -lactalbumin, b-lactoglobulin,  $\kappa$ -casein, b-casein and  $\alpha$ s2 casein are similar to that of cow milk. Goat milk is used in treatments of infants sensitive to cow milk protein; fat in goat milk is said to be more digestible (than fat in cow milk) due to higher proportion of short chain fatty acids, smaller size fat globules and lack of fat globule clustering euglobulin. However, no scientific evidence in support of this is available.
- (3). **Human milk based formula**: Lucas *et al.* (1980) blended whole human milk, human cream, salt and lactose-free human milk protein powder into a high protein, high fat and high energy infant formula for low birth weight infants. The increased concentration of antimicrobial proteins allowed subsequent pasteurization of the formula with minimal loss in antimicrobial activity.
- (4). **Preparation of immunoglobulin concentrates**: The colostrum obtained from hyperimmunized pregnant cows against enteropathogenic *E. coli* is partially purified, sterilized and freeze-dried to yield a colostral whey protein preparation containing 30-45% immunoglobulins. The product has potent anti *E. coli* activity. Infants suffering from *E. coli* mediated diarrhoea have shown better recovery. However, practicality of this procedure is doubtful and even if it is practical, the resultant product may be classified as a pharmaceutical.
- (5). **Addition of lysozyme:** The biological value of human milk is proportional to its lysozyme content. Egg white lysozyme (EWL) has a similar composition, though different antigenic specificity, than human milk lysozyme. Humanized infant formulas containing EWL have been patented in Japan and Russia. Even though increased intestinal levels of bifidobacteria have been reported in infants given lysozyme treated formula, growth of bifidobacteria is not stimulated in cow milk due to lysozyme.
- (6). **Addition of bifidobacteria**: Infant formula fortified with spray dried concentrate of bifidobacteria (final concentration 5.73 log CFU/ml) has been reported (Table 46.2). Feeding trials have shown:
- a. Decrease in pH and coliform counts of feces and increase in bifidobacteria to the level obtained with human milk .
- b. Significant increase in retention of calcium, phosphorous and iron level.
- c. Improvement in the proportion of bifidus organisms have been reported through the application of neuraminidase in infant formulas.
- d. In a process involving cultivation of *B. bifidum* in concentrated milk (20-25% TS), the product is spray dried after development of certain acidity. The survival of organisms during spray drying was found to be satisfactory.
- e. In an yet another process, direct incorporation of *B. bifidum* cells into the liquid concentrate prior to spray drying has been reported. The product was shown to contain 15,000 viable cells of *B. bifidum* /g and it had bifidus activity 170% higher than commercial

formulas and about 80% bifidus activity of human milk . On storage of the product for 6 months at room temperature and at 37°C, survival of *B. bifidus* was 48.4% and 60.4% respectively whereas retention of bifidus activity was 84.6% 77.9% respectively.

Lactulose (prepared by heating lactose) has been incorporated (either alone or with bifidobacteria) into formulas to increase the population of bifidobacteria in infants which greatly reduced intestinal pathogen *E. coli*.

Table 46.2: Differences between infants fed human milk, conventional formula and bifidus concentrate formula

No.	Parameter	Human Milk	Conventional	Bifidus
			formula	concentrate
1	Ca retention, %	60	26.7	56.9
2	P retention, %	98.6	84.4	93.6
3	Fe retention, %	36.4	9.8	32.5
4	Fecal pH	5.3	6.83	5.89
5	Fecal Bifidus (log CFU)	8.97	0.0	8.87
6	Fecal Coliform (log CFU)	6.6	7.5	7.09

(Friend et al., 1983)

- (1). **Use of hydrolyzed lactose:** Use of hydrolyzed lactose products, particularly for sufferers of lactose intolerance, has been also advocated. However, it is necessary, to examine the absorption patterns of infants given such hydrolyzed milks and also the technological effects of processing prior to use of such products.
- (2). Addition of other lactobacilli: A number of other closely related lactobacilli, which inhibit the human intestinal tract, synthesize organic acids (acetic, benzoic, lactic),  $H_2O_2$ , B vitamins and enzymes, may provide nutritional and/or therapeutic benefits. The lactobacilli also produce natural antibodies (**Table 46.3**) with broad spectrum activity against pathogenic and non pathogenic organisms. Significant improvement in weight gain in newborn infants given formula containing viable *L. acidophilus* have been reported.

Table 46.3: Natural antibodies produced by various lactobacilli

No.	Type of lactobacilli	Antibody produced
1	L. acidophilus	Acidophilin, Acidolin, Lactocidin
2	L. bulgaricus	Bulgarican
3	L. brevis	Lactobacillin (H <sub>2</sub> 0 <sub>2</sub> )
4	L. plantarum	Lactolin

(Friend et al., 1983)

#### **46.5. BABY FOOD POWDERS**

For storage stability, volume and convenience, powdered baby food of different compositions, to be dissolved in water before use has been developed by the multinational companies in their attempt to resemble mother's milk. They have gained increasing acceptance including formulations made for hyper allergic babies. In modern production of baby food powder each component in whole milk from cows is standardized to be as close to the composition of human milk as possible. For some formulations milk is not even used. Powdered baby food available on the market comprises a large group of products with different compositions, which can be classified in the following groups:

- 1. Ordinary whole milk powder
- 2. Whole milk powder with added carbohydrates
- 3. Fermented milk
- 4. Humanized milk
- 5. Products with starch

# 1. Ordinary Whole Milk Powder

The simplest type of baby food is whole milk powder with 25 to 28% fat in the solids, or half-cream milk powder with about 14% fat. Usually some vitamins are added, e.g. A,  $B_1$  and  $D_2$ , in order to equalize the seasonal variations in the vitamin content of natural milk, and to keep it, throughout the year, at the optimum standard level required for babies. The vitamins are added either to the liquid milk or dosed continuously into the concentrate by means of the dosing pump. To ensure uniform distribution of the vitamins, especially the fat-soluble ones, homogenization of the concentrate is required.

# 2. Whole Milk Powder with added Carbohydrates

This group of baby foods is based on full-cream or half-cream milk with added sucrose and maltodextrin. The sucrose is added to the milk prior to the evaporation. This allows for pasteurization of the sucrose as well, and a final product with an acceptable plate count is produced. Today industrial ready-made maltodextrin is available in most places. The maltodextrin is then mixed with partially concentrated milk. Very often vitamins are added. The whole mixture must be homogenized before drying. If the preparation of the feed is a batch process, there will be some storage and delay between the evaporator and the dryer.

# 3. Fermented Milk

The fermented milk product, usually half-cream is made by means of special culture starter strains. The pre-concentrated milk - usually not concentrated to more than 22-25% TS - is inoculated by a starter (which is normally a mixture of streptococcus lactic and lactobacillus) and fermented at a slightly elevated temperature for 6 to 10 hours.

After the required acidity has been achieved the fermented concentrate is homogenized, cooled and spray dried.

#### 4. Humanized Milk

The biggest group of baby foods is the so-called humanized milk. Human milk has a very different composition from cow's milk, as it has a higher content of lactose, a lower content of proteins with different protein composition (mainly an albumin milk while cow's milk is a casein milk) and a different fat composition (as it has a considerably higher content of unsaturated fat acids) and a lower content of minerals. There are different steps of humanization.

§ The simplest one is merely to increase the content of lactose and add some vegetable fat to increase the amount of unsaturated fat acids. Other additives, such as vitamins, ferric ions or lactulose (galactosido-fructose) are used as well, with the aim of converting cow's milk into a composition as close as possible to human milk.

§ Cow's milk contains about twice as much protein as human milk, it contains less carbohydrate and about the same amount of fat. The pre-dominant type of protein is not the same in the two kinds of milk. Thus the curd formed by human milk is soft and flocculent in the baby's stomach, whereas that of cow's milk is more tenacious and elastic. Furthermore, cow's milk contains more mineral salts (especially Ca<sup>++</sup>) and less vitamins.

Two main groups of humanized baby food dominate the market as shown in Table 46.4.

Table- 46.4: Main groups of humanized baby food dominating the market

Baby Food Composition		Age of Baby		
		0-6 months	6-12 months	
Protein	:			
	- Casein	6%	17%	
	- Whey protein	10%	4%	
Fat:				
	- Animal	6%	5%	
	- Vegetable	20%	16%	
Carboh	ydrate:			
	- Lactose	56%	40%	
	- Maltodextrine	-	14%	
Ash		2%	4%	

§ A dilution of cow's milk with carbohydrates and fat until the protein content is 15 g/l is not enough, as the composition of the protein differs to a great extent in the two kinds of milk. WPC is used extensively in baby food manufacture to adjust the protein content.

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- $\S$  Cheese whey, the protein content of which is practically only lactalbumin and  $\beta$ -globulin, is often used for standardizing. It is, however, not recommended to use it directly, as the mineral content would become too large in the final product.
- § Removal of part of the salts, by membrane filtration or demineralization either by ion-exchange or electrodialysis, is therefore nowadays used more and more.
- § Regarding the fat content, human milk contains more than cow's milk, and furthermore it contains more of the polyunsaturated fat acids, such as linoleic and arachidonic acids being necessary for a better utilization of the supplied energy. It is therefore necessary to add fat after the protein standardization.
- § Vegetable fat (often corn, palm or sunflower oil) is therefore added, as it contains above mentioned polyunsaturated fat acids. If the animal fat is replaced completely, the end product would then be short of oleic acid being present in the animal fat.
- § The mineral content of cow's milk is about four times that of human milk. During the standardization with especially whey proteins (provided the whey is demineralized) but also with fat and possibly lactose, the mineral content is reduced to an acceptable level. However, in most cases the iron content has now become too low and iron lactate, iron saccharate or iron sulphate are often added.
- § The vitamin concentration in cow's milk is usually smaller than in human milk. Furthermore it varies with the season, so vitamins are always added to the baby food, also because the processing destroys part of the vitamins.
- § Special products where the soy proteins are replacing the milk proteins are produced for allergic babies. Due to the soy proteins, the concentrate will have a high viscosity, and it is thus necessary to lower the solids content to 40-45%.

#### 1. Products with Starch

Another big group of baby foods, for babies >9 months, contains starch as shown in Table 46.5 typically with the following approximate composition:

Table 46.5: Approximate composition of baby foods for babies >9 months of age

40-50%	Whole milk concentrate
35-45%	Starch
10-15%	Saccharose
2.5%	Vitamins + Minerals

The starch can be added to the process either by dry dosing or in liquid form together with the remaining components:

• **Dry dosing** - whole milk and sucrose are mixed and pasteurized, after which it is evaporated to about 45% total solids and spray dried using an inlet temperature of 180°C. The powdered starch is dosed via the fines return system into the spray dryer

and is agglomerated with the concentrate particles. The agglomeration of the starch is very important to avoid separation during storage. As powdered starch often contains bacteria, a gamma radiation can be necessary to ensure an acceptable end product.

• **Liquid mixing** and pasteurizing offer the advantage that the starch can be pasteurized. If the pasteurization temperature is kept below 60°C, the starch will not be precooked, and the mixture is dried from a feed with 45% solids and a drying temperature of 180°C. If the pasteurizing temperature exceeds 72°C, the starch will be precooked and the viscosity will increase. Due to this the solids content must be reduced to 20-25%. Drying temperatures will be 180°C. Bag filters on the exhaust air must be used, as the powder loss will otherwise be too high.

#### **Related Documents:**

Thus, human milk does not only contain nutrients but also has a complete immune system consisting of leucocytes, antibodies, complements, lysozyme, lactoferrin, lactoperoxidase, xanthine oxidase, etc. Artificial milks are, and will continue to be, used in those cases where a mother cannot or will not feed her child. But the model for these artificial milks should be breast milk. The immunological adjustment is essential as protective proteins in bovine milk get usually destroyed during manufacture of infant milk f oods due to heat sensitive nature. Preservation of these proteins or their replacement through isolation from raw milk is essential. However, before any infant milk food formula is introduced in the market with a claim of close resemblance to the breast milk, much more needs to be learnt about the physiological response of the infant.

#### 46.6. MALTED MILK

A British pharmacist James Horlick developed ideas for an improved, wheat and malt-based nutritional supplement for infants. In 1873, James and his brother William formed a company to manufacture their brand of infant food. The company originally marketed its new product as "Diastoid," but trademarked the name "malted milk" in 1887. Despite its origins as a health food for infants and invalids, malted milk found unexpected markets due to its nutritive value, convenience, digestibility and palatability. It is also appreciated for its lightweight, non-perishable and high-calorie qualities worldwide.

**Malted milk** is a powdered food product made from a mixture of malted barley, wheat flour, and whole milk, which is evaporated until it forms a powder. Malt powder comes in two forms: diastatic and non-diastatic. Diastatic malt contains enzymes that break down starch into sugar; this is the form bakers add to bread dough to help the dough rise and create a good crust. Non-diastatic malt has no active enzymes and is used primarily for flavor, mostly in beverages. It sometimes contains sugar, coloring agents, and other additives.

## 46.6.1.Malt

**Malting** is a process applied to cereal grains, in which the grains are made to germinate by soaking in water and are then quickly halted from germinating further by drying/heating with hot air. Thus, malting is a combination of two processes: the sprouting process and the kilndrying process. The term "malt" refers to several products of the process:

• The grains to which this process has been applied, for example malted barley;

- The sugar, heavy in maltose, derived from such grains, such as the baker's malt used in various cereals; or
- A product based on malted milk, similar to a malted milkshake (i.e., "malts").

Malting grains develops the enzymes that are required to modify the grain's starches into sugars, including monosaccharides (glucose, fructose, etc.) and disaccharides (sucrose, etc.). It also develops other enzymes, such as proteases, which break down the proteins in the grain into forms which can be utilized by yeast. Barley is the most commonly malted grain in part because of its high diastatic power or enzyme content.

# 46.6.2. Drying of Malted Milk

The malted milk food is generally prepared by tray-drying method under vacuum which is essentially a batch operation and hence adds to the cost. Spray drying method reduce the cost of product owing to high volume of continuous and automated operation. Thus the production of good quality, low cost malted milk food with improved physical characteristics appears to be better proposition than continuing with the traditional tray-drying method. The method employing spray process is shown in the following flow chart Figure 46.2

Fig. 46.2: The method employing spray process for Malted milk product

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#### Module 19

# Microbiological quality of various dried milks including Infant Foods

#### Lesson-47

# Microbiological Quality of Various Dried Milks Including Infant Foods

# **47.1. INTRODUCTION**

Microbial and enzymic deterioration are rare in milk powder. However some changes related to these might be observed in dried milks.

#### 47.2. EFFECT OF WATER ACTIVITY

Growth of microorganisms can strongly depend on water content of a food. For microbial deterioration to occur,  $a_w$  should increase to over 0.6 (and for the majority of microorganisms much higher); such a high  $a_w$  can only be reached if the powder is exposed to fairly moist air. Deterioration then is often caused by molds. Enzymatic hydrolysis of fat has been observed at  $a_w \ge 0.1$ , although extremely slow. Accordingly, whole milk powder must be free of lipase. Milk lipase will always be inactivated by the intense pasteurization of the milk as applied in the manufacture of whole milk powder. This is by no means ensured, however, for bacterial lipases. Hence, not too many lipase-forming bacteria should occur in the raw milk. Proteolysis in milk powder appears highly improbable and has not been reported.

However, enzymic deterioration of liquid products made from the milk powder can occur if enzymes are present before the drying, as drying usually does not cause substantial inactivation of enzymes. Several authors relate the possibilities of growth of microorganisms to the water activity, and the lowest values at which organisms can grow are shown in Table 47.1.

Table 47.1: Lowest values of water activity at which organisms grows

No.	Organisms	$\mathbf{a}_{\mathrm{w}}$	No.	Organisms	$\mathbf{a}_{\mathrm{w}}$
1	Bacteria	0.98-0.90	4	Halophilic bacteria	down to $\sim 0.75$
2	Yeasts	$\sim 0.9$	5	Osmopholic  ye asts	$down \ to \sim 0.6$
3	Molds	0.92-0.80	6	Xerophilic molds	$down \ to \sim 0.6$

A simple explanation is that a low value of  $a_{\rm w}$  implies a high value of the osmotic pressure. The organism is unable to tolerate high osmotic pressure as it will cause water to be drawn from the cell, damaging its metabolic system, generally because the internal concentration of harmful substances becomes high. However, microorganisms have acquired various mechanisms to neutralize the effect of a high osmotic pressure and the effectivity of these vary greatly among organisms:

- 1. A fairly small difference in osmotic pressure between *t* he cell and the environment can often be tolerated. Bacterial spores can tolerate a considerable difference in osmotic pressure.
- 2. Some solutes, e.g., most alcohols, can pass the cell membrane unhindered; hence, these do not cause an osmotic pressure difference. If such a solute is, moreover, compatible, which means that it is not harmful to the cell at moderate concentration, the organism can survive and possibly grow.
- 3. The organisms may produce and accumulate low-molar-mass compatible substances that keep the internal osmotic pressure high; it often concerns specific amino acids.
- 4. Specific harmful substances, e.g., lactic acid, may be to some extent removed from the cell.

Consequently, it often makes much difference what solute is involved; in other words, the lowest value of a w tolerated strongly depends on the composition of the medium.

#### 47. 3. EFFECT OF HYGIENIC ASPECTS

The requirements for the bacteriological quality of milk powder partly depend on its intended use and, in connection with this, also on the manufacturing process. For example, whether the powder is meant for direct consumption or whether it is subjected to a heat treatment after reconstitution (e.g., for recombined milk) is important. The heat treatment during the manufacture of (skim) milk powder, classified as 'low heat', usually is not more intense than the heat treatment during low pasteurization (say, 72 °C for 20 s); consequently, many bacteria may survive the manufacturing process. The causes for milk powder to be bacteriologically unacceptable or even unsafe can be of three kinds:

- 1. In the fresh milk, bacteria are present that are not killed by the heat treatments to which the milk is subjected before and during drying.
- 2. Conditions during the various process steps until the product is dry do allow growth of some bacterial species.
- 3. During manufacture, incidental contamination may occur. The level of contamination is generally low and remains low if the bacteria involved cannot grow.

In establishing the bacteriological quality of the powder, the species of bacteria involved should be considered. Then the cause of the contamination may be deduced, as may the measures that must be taken to improve the quality.

# 47.4. SAMPLING AND CHECKING FOR MICROBIOLOGICAL QUALITY

Bacteria originating from contamination or growth prior to drying will usually be homogeneously distributed throughout the powder and cause no problems with sampling. This is different for bacteria originating from incidental contamination of the powder, which may be distributed quite inhomogeneously. It is not possible to devise sampling schemes that guarantee detection of incidental contamination. To ensure that a product is bacteriologically safe, not only should the powder be sampled but samples must also be taken at sites that are potential sources of contamination.

#### 47.5. GROWTH OF BACTERIA DURING MANUFACTURE

- § Temperature and water activity during successive steps in manufacture are such that some thermophilic bacteria can readily grow and they are not or insufficiently killed during drying. The type of bacterium often is characteristic of the cause of the contamination.
- § In the regeneration section of pasteurizers and thermalizers (and possibly in the part of the evaporator plant where the milk is heated in counterflow), S. thermophilus, in particular, can develop. The bacterium grows fastest at 45  $^{0}$ C but scarcely multiplies at temperatures over 50  $^{0}$ C. It generally does not grow in the evaporator, because the temperature is too high in the first effects, whereas in the later effects a will be too low. Because S. thermophilus is moderately heat resistant, relatively high counts may result, especially in low-heat milk powder and in whey powder. In medium and high-heat powder the bacterium is killed during manufacture. Determination of the count of S. thermophilus in the milk just before the preheater may give a good indication of the fouling of the heating section of plant and of the moment at which it should be cleaned.
- § In some drying plants that employ a wet washer to recover powder fines, the outlet air is brought into contact with a film of milk rather than water, thereby preheating the milk and saving energy; this implies that the preheated milk acquires the wet bulb temperature ( $\sim 45^{\circ}$  S. thermophilus. C), which leads to ideal growth conditions for
- § The conditions in the second half of the evaporator and in the balance tank are not optimal for S. thermophilus. Enterococci (E. faecium), in particular, will start to grow. If the milk is properly preheated and the plant is satisfactorily cleaned and sterilized, it will take a rather long time, however, before substantial counts of E. faecium have developed. In actual practice, these prerequisites are not always met and in milk powder with a relatively high count, E. faecium often is the predominant species.
- § Likewise, the conditions in the second part of the evaporator and in the balance tank are favorable for growth of *Staphylococcus aureus*. The bacterium generally is killed by pasteurization, and strains of S. *aureus* in milk powder have been shown to have phage characteristics different from the strains in raw milk. Presumably, they originate from direct or indirect human contamination. Heat stable enterotoxins can be formed and the amounts formed at counts of  $10^7$  to  $10^8$  per ml may cause the powder to be a health hazard. Although S. *aureus* is not heat resistant, the conditions during drying appear to be such that complete killing is not achieved. Roughly  $10^{-5}$  to  $10^{-1}$  of the initial count of these bacteria have been found to survive under various practical conditions. This means that S. *aureus* can at least be found in 1 g of fresh powder if its count before the drying was so high that production of enterotoxins could have occurred.
- § Bacillus stearothermophilus (ssp. calidolactis in particular) can readily grow at higher temperatures. Its growth range is from 45  $^{0}$  C to 70  $^{0}$  C, with an optimum near 60  $^{0}$  C. It can also grow in concentrated milk and therefore throughout the equipment between preheater and drier. Moreover, the bacterium can form spores under these conditions, which further limits its killing during drying. Obviously, some growth of B . stearothermophilus will always occur during manufacture, even in a well-cleaned and disinfected plant. However, under most conditions this will not cause problems.

# 47.5.1. Measures to control the growth of bacteria

With regard to the growth of bacteria during the manufacturing process, a number of measures will have to be taken to prevent the equipment from becoming a kind of fermentor for bacteria:

- § Special attention should be paid to the temperature and the time for which the product stays in the equipment, and to matching of the capacities of the various parts of the plant. Multiple-effect falling-film evaporators are more satisfactory in this respect than are flash evaporators.
- § It is recommended that the volume of the concentrate balance tank be kept as small as possible. Mostly, there are two tanks, making possible a change every 2 h or so, and the cleaning of one while the other is in use.
- $\S$  Contamination of the product flow from outside should be prevented, with particular reference to S. aureus.
- § The concentrated milk may be pasteurized just before it enters the drier, and this is increasingly being done. It is especially successful with regard to the killing of E. faecalis and E. faecium, provided that the temperature applied is high enough. Heating for 45 s at 72  $^{0}$  C has little effect; 45 s at 78  $^{0}$  C causes a considerable reduction.

# 47.6. INCIDENTAL CONTAMINATION

A distinction can be made between contamination before drying (wet part) and during or after drying (dry part). Bacteria involved in these types of contamination generally do not grow during the process and contribute little to the count of the powder.

- § Contamination after preheating and before drying can readily occur if the equipment has been insufficiently cleaned. This is only important if the bacteria involved can survive the drying (and the pasteurization before drying, if carried out). In spite of the high inlet and outlet temperatures of the air, the concentrate droplets usually will not attain a high temperature; moreover, the heat resistance of the bacteria increases markedly with the drymatter content. About 70% of E . faecalis and E . faecium survive during drying, whereas survival of S . aureus varies widely. About  $10^{-4}$  to  $10^{-5}$  of the initial count of Salmonella spp. and E . coli will survive. Based on these facts, and due to the relatively low level of contamination, the powder may be expected to contain no appreciable counts of enterobacteria immediately after leaving the drier. This is confirmed in actual practice.
- § Contamination of the powder can occur at many places in the spray drier, during fluid bed drying, and during packaging. The species of contaminating bacteria can vary widely, but it usually concerns species that can grow in wet remnants of milk powder in the drier or in the surroundings of the manufacturing line. Contamination via (in) direct human contacts should also be considered (e.g., *S. aureus*). Bacteria can easily survive in dry powder, and undesirable bacteria can start to grow if the water content increases to over 20%. The supply of cooling air into the spray drier and into the fluid bed drier can be a source of direct contamination. It may also be responsible for indirect contamination because it gives, at certain sites, better conditions for survival and growth of bacteria in remnants of not fully dried powder. Special precautions are needed if the drier and its accessories have been wet-

cleaned. To restrict such incidental contamination, the plant and its surroundings should be rigorously freed of remnants of (wet) powder.

Among the kinds of bacteria found in this type of contamination, enterobacteria are of special interest. It generally concerns coliforms; this is possibly caused by lactose being the only carbon source in this environment. Nevertheless, the use of coliforms as an indicator organism is of restricted value for milk powder: If coliforms are absent, salmonellae or other pathogenic bacteria may still be present.

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#### Module 20

# Management of condensed and dried milk industry

# Lesson-48

#### 48.1. INTRODUCTION

The condensed and dried milk plants are no different than the other dairy operations. The general criteria followed throughout the industry can be of the following system.

#### 48.2. STYLE OF MANAGEMENT

1. Top Down Approach: All instructions from top

2. Bottom up approach: Rank has the responsibilities

#### 48.3. ORGANIZATIONAL STRUCTURE

What individual should do is shown in Figure 48.1 of the Organizational structure

# Fig. 48.1: Organizational structure of a dairy plant

# 48.4. IMPORTANT AREAS FOR A SPRAY DRYING PLANT TO CONTROL

Following guidelines will be useful for the people engaged in the drying plant system and will prove beneficial to others for planning their work according to given and tested guidelines.

# 1. Quality of the spray dried product

- (a) quality needed
- (b) The value which the manufacturer likes to incorporate in view of the purchasing capacity of the consumers with best utilization of the inputs at competitive cost.
- (c) The ratio of quality achievable per unit expenditure should be kept relatively constant as long term goal.
- (d) Ensuring excellent sanitation and hygiene to keep bacterial load well under control
- (e) Market feedback about quality control, analysis and efforts
- 2. **Productivity:** Productivity involves best possible utilization of resources like

- (1) Money
- (2) Materials
- (3) Machines and
- (4) Men

Allocation of resources is made to achieve the production of quality powder at competitive cost by cutting down unit cost and thus improving productivity.

# 3. Effort needed for utilization of resources

- § Planning person involved and necessity.
- § A detailed integrated plan of action.
- § Rank should understand responsibility in view of corporate guidance
- § Planning subject to change as per the availability of resources
- § Putting plans into action as it is a result of predetermined thought process of group of people.
- § Follow up.
- 4. **Supervision**: In view of the action plan, every supervisor should take care of:
- "Cleanliness, sanitation and hygiene
- " Efficiency of machine and manpower
- "Consistency in production
- "Process parameters and checks
- "Maintenance with the help of engineers
- "Raw material availability
- "Control of wastages
- " Quality
- "Promotion of harmonious labour management relations

# 5. Manpower effectiveness

§ Workmen should follow correct procedures.

- § Unless the workmen want to achieve error prevention as per the instruction of their supervisors or else no improvement will take place
- § Motivation through educational programmes for improving their skill
- § Setting an environment in the plant by understanding human needs on the job
- § Workmen to be given responsibility and challenge
- § Identify average, good and excellent performers and train them properly
- § Pat people when they have done good.
- § Be an active listener
- § Handle carefully negative ones for achieving your goal
- § Above all, people of average intelligence and normal personality should be there

# **6.** Introduction of error prevention programme

- § Don't tolerate problems as they will multiply
- § This involves researching, engineering, specifying and implementing procedures which when followed will assure low probability of error
- § How best errors can be reduced to zero level should be ultimate aim

#### 7. Effective communication system:

- § Most people are afraid of communication
- § An open discussion about your drying plant covering all the problems and constraints should be discussed at appropriate times
- § Discuss your problems frankly
- 8. **Control of wastages:** At the time of milk transfer, condensing, drying, packing, stacking and dispatching. Following are few Reasons for the waste:
- § Inadequate system of drying
- § Insufficient skill
- § Lack of interest among employees
- § Failure to carry out instructions
- § Indifference or laziness

- § Insufficient staff
- § Replacement of spare parts without inspection
- § Short supply of utility services like steam, hot air or water
- § Unavailability of spare parts
- § Spillage
- § Not getting the optimum parameters
- § Allowing more product in packing lines to pack
- § Frequent stoppage of p lant causing rejections
- § Not following the present plan for cleaning schedules and changeover

# 9. Analyze waste for collective action

- § Area where waste is recurring
- § Initiate collective action
- § Develop systematic plan for control
- § Trace critical control points
- § If problems are many at various places, give priority to critical ones
- § Follow up that your action plan is followed rigidly to control waste.

# 10. Ideal system for controlling waste

- § Delegate responsibility for carrying out the work.
- § Ask for suggestions of everyone in the plant
- § Develop records and analyze suggestion
- § Devote time to be with the people to show your concern
- § Recommend changes in job methods to avoid waste
- § Prevent waste through advance planning of day-to-day work schedule and job assignments
- § Develop people for predicting the problem in advance by analyzing the data bank
- § Don't forget to educate if they have not accorded proper attention.

# 11. Develop records

- § Record day-to-day problems, their cause and record remedial measures
- § Record production data along with electricity, steam, water and manpower
- § Record wastages
- § Calculate consistency in your production to ascertain where you are
- § Developing records will generate a repertoire of tricks among plant people to tackle problems

# 12. Preventive maintenance system

- § It helps to analyze the cause of failures of certain items.
- § It helps in finding out the duration or age of a co mponent like bearings, seals and rings and help to take remedial measures
- § A preventive maintenance plan n eed to be worked out for reducing wastages and making the system of drying more economic

# 13. Safety in powder plant

- § This generate confidence among the employees that management is concerned about them
- § Make sure that hydrant system has been provided in the drying chamber to extinguish fire
- § Make sure that enough vent/doors have been provided to avoid explosions
- § Make sure that all motors, gears have been guarded properly to avoid accidents
- § People to be trained for controlling fire and other untoward incidence
- § Make sure that emergency kits and ambulance are handy
- § Periodical examination of all such systems to see whether they are operative

# 14. A stepwise plan of action for economizing drying system

- § Plan work schedule.
- § Prepare detailed production schedule, make flow chart giving the sequence of operation, give time for each operation
- § Arrange materials required
- § Allocate manpower and spell out who will do what

- § Go on accounting hourly achievements
- **15. Review of production schedule: Take f** air trial for cutting cost by way of:
- § Reducing wastage
- § Rationalizing work force
- § Reduce transportation time
- § Reduce unnecessary movement of workmen
- § Reduce workers movement to tool room for bringing engineering items
- § Keep necessary items near your drying plant

# 16. Other measures to reduce cost

- § Cut down office expenses; organize layout of your office that people can approach you without wasting time
- § Make sure that your procedures are simple and avoid those which are not necessary
- § Don't add paper work too much
- § Keep only essential staff for your office
- § All out effort not follow bureaucratic system this is a costly way; follow American system and do your job yourself
- § In the matter of productivity, merely hard work does not merit commendation; remember, what is important is that the work done should yield productive results
- § Delegate responsibilities except accountability; everyone in the system should be accountable

See that time is also a valuable resource, it should be utilized most productively by all concerned

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