EVALUATION OF SEASONAL VISCOSITY VARIATIONS IN SWEETENED CONDENSED MILK

by

M.D.H.P.JAYASENA (00/AS/051)

This thesis is submitted in partial fulfillment of the requirements for the degree of

Bachelor of Science in Food science and Technology

Department of Natural Resources Faculty of Applied Sciences - Sabaragamuwa University of Sri Lanka Buttala-91100

March 2004

Declaration

The work described in this thesis was carried out by me at the International Dairy Products Limited at Polonnaruwa under the supervision of Mr. Lucky Rathnayake and Mrs.Deepika Priyadarshani.A report on this has not been submitted to any other university for another degree programme.

Hardran

M.D.H.Jayasena (00/AS/051) Date: 06/05/2004

Certified by Mr.Lucky Rathnayake External Supervisor The Production Manager International Dairy Products Limited **Gallela** Polonnaruwa Sri Lanka

Date: 06/05/2004

Mrs.W.M.D.Priyadarshani Internal Supervisor Department of Natural Resources Faculty of Applied Sciences Sabaragamuwa university of Sri Lanka Buttala SriLanka

05/2004 Date:...

Prof.M.S.Rupasinghe Head/Department of Natural Resources Faculty of Applied Sciences Sabaragamuwa university of Sri Lanka $\ddot{\cdot}$ Buttala SriLanka

MSRupern'nefre
Date 12/5/2004

Fondly dedicated to my loving parents, brother and Manjuka

 $\mathcal{L}^{\text{max}}_{\text{max}}$

 $\mathcal{L}^{\text{max}}_{\text{max}}$ and $\mathcal{L}^{\text{max}}_{\text{max}}$

 $\sim 10^{-1}$

 $\label{eq:2.1} \frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac{1}{\sqrt{2}}\sum_{i=1}^n\frac$

 Δ

 $\frac{1}{2}$

 $\label{eq:2.1} \mathcal{L}^{\text{max}}_{\text{max}}(\mathbf{X}^{(1)},\mathbf{X}^{(2)}) = \mathcal{L}^{\text{max}}_{\text{max}}(\mathbf{X}^{(1)},\mathbf{X}^{(2)})$

ACKNOWLEDGEMENT

First and foremost, I would like to express my thank to Mr. Upali Weerasinghe, The Factory Manager, and Mr.Cajetan Almeida ,The Quality Assurance Manager, for providing this opportunity to conduct this project at the Nestle Lanka Limited, Polonnaruwa.

I wish to express my deepest gratitude to my external supervisor, Mr. Lucky Rathnayake, The Production Manager for his much appreciated guidance given to me to carry out this project successfully. My gratitude is further extended to Mr.L.Y.Jayatissa, the Quality Assurance Executive and Mr.S.D.Rajapaksa, a senior member of Quality Assurance staff of Nestle Lanka Ltd, Pannla.

My sincere thank goes to my internal supervisor, Ms Deepika Priyadarshani ,The Lecturer of the Department of Natural Resources .University of Sabaragamuwa ,for the guidance, advice and encouragement given to me through out this project and in preparation of the Project Report.

Finally ,1 would like to pay my appreciation to my parents , brother and Manjuka for their valuable support and inspiration.

ii.

ABSTRACT

1

The viscosity is one of the most important quality attributes in sweetened condensed milk. The low viscosity of the product manufactured during the period of May to September extended the superheating time in manufacturing process. Therefore this study was done to find a reason *Is* for the seasonal viscosity variation in sweetened condensed milk.

Past data were recorded for the superheating time in batch wise and summarized the variation through the year 2002 and 2003.Data for the total solid content and the day old viscosity were recorded and analyzed by linear regression to find whether there is a correlation between TS and day old viscosity. Besides the data from final products ,fat and Solids non fat percentages in fresh milk during the year 2002 and 2003 were recorded and graphs were plotted to identify the variation of the fat and solids non fat.

Fresh milk samples were tested for protein and ash to determine the compositional variation in milk during a month of period in non-problematic period and analyzed by linear regression to find whether there is a correlation.

The correlation of fat and protein content in the samples in non-problematic season revealed the possibility of low protein in milk during the problematic period. This help to argue that viscosity of products produced from milk of problematic season is due to the low protein content of milk .because protein is one of the major component which affect to viscosity of milk.

CONTENT

 $\ddot{}$

 $\hat{\mathcal{A}}_1$

 $\hat{\mathcal{A}}$

Chapter 3

Chapter 4

 σ .

VII

 $\sim 10^{11}$

 \sim

 $\hat{\mathcal{L}}$

Chapter 5

 $\ddot{\cdot}$

Conclusions and Suggestions

 $\overline{}$

i.

 $\ddot{}$

 $\ddot{}$

 \mathbf{r}

 $\mathcal{L}^{\mathcal{L}}$

l.

LIST OF ABBREVATIONS

- Avg. = Average
- $^{\circ}$ C = Degree of Celsius
- cm = Centimeter
- Cone. = Concentrated
- Dept. = Department
- Dil. = Dilute
- Fig. = Figure
- $g = Gram$
- H20 = Water
- HCI = Hydrochloric Acid
- IDPL = International Dairy Products Limited
- $Min =$ Minute(s)
- $ml = Milli$ liters
- $Pg. = Page$
- SCM = Sweetened Condensed Milk
- $SH = Super heating$
- $SNF = Solids Non Fat$
- TS = Total Solids
- % = Percentage
- $>$ $=$ Greater
- $+ =$ Plus or Minus

LIST OF FIGURES

 $\ddot{}$

 $\ddot{}$

 $\ddot{}$

iv

LIST OF TABLES

 \bar{z}

 $\mathcal{L}_{\mathcal{A}}$

 $\ddot{}$

 $\ddot{}$

Chapter 01

1.1 Introduction

Milk, the first food for infant mammals (Early, 1998), is a nature's gift to mankind with balanced nutrition composition. Man used cow's milk as food from prehistoric times and found ways to preserve it for later usage when necessary. However, milk can be conserved and preserved by removing much of the water present in it, which gives it its perishable nature. (Rai, 1996)

Among two condensed milk, sweetened and unsweetened, sweetened condensed milk is basically concentrated milk to which sugar has been added. High sugar concentration with or without elimination of water was used to preserve milk in 1847.when the first sweetened condensed milk was prepared. (Rai, 1996)

In the manufacturing process of sweetened condensed milk, the nature of the heat treatment with time temperature combination determine the viscosity of the end product, and is then extremely important to the quality of the end product. Heating of either skim milk or whole milk at temperatures up to about 65^oC results in a temporary decrease of a few hundredths of a centipoises in the viscosity measured after quickly cooling to room temperature.

Studies of Whitnah et al.,1956, agree in indicating that this effect is due to a reversible change in the caseinate micelles.The recovery from the viscosity decrease produced by heating was shown by Whitnah, 1956 as to be an approximately linear function of the logarithm of age after heating.

And also, Whitnah, 1956, found high positive correlations between viscosity at 4° C of milks from individual cows and their fat and protein contents.Surprisingly, the correlation between viscosity and the content of solids other than fat or protein was negative, which may reflect an inverse effect of lactose on viscosity.

The influence of season on the composition has been studied in .India. According to Reddy et al., 2002, seasonat⁻variation in fat and solids non fat (SNF) content of milk from Punganur cows revealed that higher values of fat were observed during summer season and the higher values of the solids non fat were observed during the winter seasons.

I

As well as, results presented by Corbin and Whittier highlighted the influence of season on gross composition representing 2,426 samples from 1,482 individuals of six breeds .It emphasis that fat, protein, ash and total solids (TS) of milk showed significant reduction in content during the months of March to September. Besides, lactose shows an increasement during this period. Seasonal variations in the salt balance of raw milk and its 3:1 concentrate were studied over one year by Pouliot (1995) and a marked increase in the level of colloidal calcium was found between December and February in raw milk.

Therefore it appears that viscosity is a function of concentration of especially fat and protein and it can be said that viscosity of sweetened condensed milk can be affected by not only the time temperature combination, but the composition of the raw milk.

Hence, the study was conducted with following objectives.

 $\mathcal{L}_{\mathrm{eff}}$

1.2 Objectives

- To determine the effect of time temperature combination on viscosity development.
- To determine the effect of compositional changes in raw milk on the viscosity — development and find an instinct factor/s that change the viscosity of sweetened condensed milk during May to September.

Chapter 02

Literature Review

2.1 What is milk?

For young mammals, including human infants, milk is the first food ingested and, most cases, it continues to be the sole constituent of the diet for a considerable period of time. Milk is the normal secretion of the mammary gland and is defined by the United State Public Health Service as 'the lacteal secretion, practically free from colostrums obtain by the complete milking of one or more healthy cows, which contains not less than 8.25%of milk - solids- non-fat and not less than 3.25% of milk fat '. State and local regulations specify compositional variance from the Federal Stand ranging from 8.0 - 9.0% milk - solids - non - fat and from $3.0-3.8\%$ fat .By definition and esthetic reasons, colostrums $-$ the secretion immediately following parturition $-$ is excluded. (Fennema, 1976)

The quantities of the various main constituents of milk can vary considerably between cows of different breeds and between individual cows of the same breed. Therefore only limit values can be stated for the variations.

Table 2.1 Quantitative composition of milk

Source: R. Fennema (1976), Principles of Food Science

2.2 Milk Constituents

2.2.1 Milk Proteins

The primary structure of proteins consists of a polypeptide chain of amino acid residues joined together by peptide linkages, which may also be cross linked by disulphide bridges. Amino acids contain both a weakly basic amino group, and a weakly acid carboxyl group both connected to a hydrocarbon chain, which is unique to different amino acids. The three dimensional organization of proteins, or conformation, also involves secondary, tertiary, and quaternary structures.

The total protein component of milk is composed of numerous specific proteins. The primary group of milk proteins is the caseins .The casein content of milk represents about 80% of milk proteins. The principal casein fractions are alpha (s1) and alpha (s2) caseins, beta casein, and kappa-casein. The distinguishing property of all caseins is their low solubility at pH 4.6.The common compositional factor is that caseins are conjugated proteins, most with phosphate group (s) esterified to serine residues. These phosphate groups are important to the structure of casein micelle. Calcium binding by the individual caseins is proportional to the phosphate content.

Casein is composed of several similar proteins, which form a multi molecular, granular structure called a casein micelle. Besides casein protein, calcium and phosphate, the micelle also contains citrate, minor ions, lipase and plasmin enzymes, and entrapped milk serum .The 'casein sub micelle' model has been prominent for the last several years, and is illustrated, but there is not a defined sub micellar structure to the micelle at all.

In the sub micelle model, it is thought that there are small aggregates of whole caseins, containing 10-100 casein molecules, called sub.micelles .It is thought that there are two different kinds of sub micelle; with and without kappa casein. This sub micelle contains a hydrophobic core and is covered by a hydrophilic coat, which is at least partly comprised of the polar moieties of kappa casein.. (Hurley, 2002)

There are several factors that will affect the stability of the casein micelle system;

Salt content:

Affects the calcium activity in the serum and calcium phosphate content of the micelles,

pH:

Lowering the pH leads to dissolution of calcium phosphate until, at the iso electric point (Ph 4.6), all phosphate is dissolved and the caseins precipitate.

Temperature:

At 4C, beta casein begins to dissociate from the micelle, at OC, there is no micellar aggregation; freezing produces a precipitate called cryo casein.

Heat treatment:

Whey proteins become adsorbed, altering the behavior of the micelle.

Whey proteins are another major milk protein of two distinct types .Casein constituents over 80% of the total protein of the milk, although the relative proportion of whey protein to casein varies according to the stage of lactation. Whey protein comprised of two gene products beta lactoglobulin and alpha lactalbumin. In structure ,whey proteins are typical compact globular proteins, with a relatively uniform sequence distribution of non-polar, polar and charged residues.

Effect of heat on milk proteins

 $\tilde{\mathbb{I}}$

Casein micelles are remarkably stable at temperatures up to 140 °C.ln contrast ,whey proteins are relatively heat liable .extensive denaturation occurring at 80 °C.Denaturation is accompanied by extensive breaking and randomization of the stabilizing disulphide bonds. Beta lactoglobulin is more heat liable than alpha lactalbumin as a consequence of its one free sulphydryl group, which permits the initiation of auto catalytic disulphide exchange reactions.

Denaturation of beta lactoglobulin has further important consequences .since at 100 °C and higher temperatures .interaction occurs between beta lactoglobulin and kappa casein. (Varnam et al., 1994)

2.2.2 Milk Fat

A microscopic examination of a drop of milk will reveal the presence of an immense number of tiny fat globules, estimated to be about three million per cubic millimeter. A thin membrane, 8 to 10 nm in thickness, whose properties are completely different from both milk fat and plasma, covers these liquid fat droplets.

The fat globules in a single sample of milk may vary considerably above and below the diameter of 0.003mm.The size of the globules in the milk of cows of different breeds also varies.

Fat globules are smaller at the end of the lactation period. They are kept apart by surface tension, but exhibit the tendency to form clusters. At ordinary temperatures, milk fat is a fluid existing in an under cooled state. The specific gravity of fat is 0.93 at 15° C.

The globules being less dense than the liquid in which they are dispersed tend to rise, so a layer of cream is formed when milk is allowed to remain undisturbed (Vanstone, et al., 1975). Stokes' Law predicts that fat globules will cream due to the differences in densities between the fat and plasma phases of milk. However in cold raw milk, creaming takes place faster than is predicted from this fact alone. IgM , an immungglobulin in milk , forms a complex with lipoproteins. This complex, known as cryoglobulin precipitates on to the fat globules and causes flocculation. This is known as cold agglutination.

Fig2.1: Distribution of fat and protein

The process of homogenization of fluid milk is one where the milk fat globules are broken in to sizes small enough that they will not rise in the milk to form cream under normal milk storage conditions. Milk fat has a melting point between 29 and 35° C, it has not fixed or definite melting point because it 's a mixture of many fats.

2.2.3 Lactose

The characteristic carbohydrate of milk is lactose (milk sugar). It was formerly thought to occur only in milk, to be the only sugar in milk, and to be found in the milk of all species.

The lactose in normal cow's milk generally ranges from $4.4 - 5.2\%$ averaging 4.8% anhydrous lactose. This usually amounts to 50-52% of the total solids in skim milk. When considering the molecular structure, lactose is a disaccharide that yields D glucose and D galactose on hydrolysis. Further more it is found in forms of alpha hydrate and anhydrous beta or as an amorphous "glass" mixture of alpha - and beta - lactose.

The crystallization principle, which can be arised due to lactose, is applied in processing dairy products. E.g. Sweetened condensed milk, instant milk powders, Stabilized milk powders .In the processing of sweetened condensed milk , small crystals are desired to assure a smooth body product. Since , in general .rapid crystallization produces small crystals ,it is desirable to crystallize at the optimum temperature for rapid crystallization.(Webb et al.,1987)

2.2.4 Minerals

The minerals in milk consist principally of the bicarbonates, chlorides, citrates and bicarbonates of calcium, magnesium, potassium and sodium. All of the minerals are distributed between a soluble phase and a colloidal phase and while monovalent ions exist largely, or totally, in the soluble phase as much as 66% of the calcium and 55% of the phosphorous may be in the colloidal phase. The distribution of calcium, citrate, magnesium, and phosphate between soluble and colloidal phases and their interactions with milk proteins has important consequences for the stability of milk and milk proteins (Varnam et al., 1994).

The salts of milk and the ash of milk are not identical. The ash of milk denotes the white residue left after milk has been incinerated at a low red heat. Since the metallic elements are in excess of the non metallic, the ash is always alkaline in character .The composition of the ash doesn't represent the state of the salts as they occur in milk, since there is considerable alternation due to the chemical reactions' taking place during incineration. The ash contains substances derived from both organic and inorganic compounds of the milk. Average normal milk is considered to contain 0 .7% ash,and this amount represents a salt content of about 0.9%.(Webb et al.,1987)

2.3 Physical properties of milk

2.3.1 Viscosity

Viscosity of a substance may be defined as its resistance to flow due to internal friction between molecules as they shear each other. The unit of viscosity is the poise named after Poiseuille, one of the pioneers in this line of investigation. The poise is the force (dynes) required to move for a distance of one centimeter in one second of time a plane one square centimeter in area past another plane of the same area , which are one centimeter apart and the space filled with the liquid.

Milk is more viscous than water due to the casein, fat and albumin, listed in descending order of greatest effect. The viscosity of milk may be measured in sevecal ways .The more accurate method is to employ a viscometer. This is an instrument that measures the viscosity by measuring the resistance of a solid when rotated while suspended in the liquid .Many factors affect the viscosity of the liquid .Which would include milk .

Any factor that induces clumping of the fat globules will increase the viscosity .Low temperature and aging have this effect. Mechanical agitation of whole milk decreases the viscosity .because the fat globule clumps are partially broken up .while in case of skim milk it has no effect due to the small amount of fat present .Homogenized milk would not be affected either as the globules are already broken up. Homogenization increases the viscosity of whole milk but slightly decreases that of skim milk .The process breaks up the fat globules in to much smaller ones and there by provides a-larger surface area than previously. A film of protein is absorbed by the surface of the globules and this surface being much greater than in the case

of the non homogenized milk , a much greater adsorption takes place and this causes a greater viscosity. In regard to the skim milk, some of the protein particles would be broken and therefore the viscosity would be reduced.

Pasteurization temperature slightly lowers the viscosity through breaking of the clumps of fat globules ,but when subjected to high heat as under pressure , the viscosity is increased due to the proteins being affected. Dilution of milk with water lowers the viscosity as there is almost a direct relationship of total solids in milk to viscosity. Viscosity also increases with development of high acid and with increased fat content.(Atherton et al.,1987)

2.3.2 Acid Base equilibria

The equilibria involving protons and the substances, which bind them, are among the most important in dairy chemistry. Certain treatments, which alter the state of dispersion of proteins and salts, are reflected in the status of the protons. Thus the intensity (pH) and capacity (buffer power) factors of the acid base equilibria have come to be much used in processing control.(Webb et al.,1987)

2.3.3 Density

The density of such system as milk is a resultant of the densities of its components complicated by variations in the ratio of solid to liquid fat and in the degree of hydration of proteins. Thus the density of a given specimen of milk is determined by its percentage composition,, by its temperature, and by its previous history of temperature fluctuations and processing treatments. (Webb et al.,1987)

2.3.4 Freezing point

Milk has a freezing point, which is quite constant and is generally given as -0.55° C.lt depends on the soluble constituents .mainly lactose and chlorides. These compounds have a close inverse relation .that is, when one rises in amount the others lowers and vise versa .which fact accounts in large measure for this constancy. $\tilde{\mathbb{C}}$

Freezing points of milk may vary daily, seasonally ,as well as according to breed .feed .and many other factors .This illustrates the fact that the freezing point depends .not on the composition of the sample .but on the concentration of the dissolved substances .mainly lactose and salts. The fat existing in milk as coarse globules and the casein as colloids have no effect on the freezing point. As milk freezes it becomes very uneven in composition. The frozen portion is low in fat and other solids, while the liquid portion becomes concentrated with the milk solids.

Freezing alters the physical condition of milk and it never returns to its original state. Freezing causes the fat globules to lose their complete emulsion, to clump and become distorted and irregular in shape and size. The casein is also affected by freezing. It is partly broken from its existence in milk as calcium caseinate and precipitated as flakes. Because of the effect of freezing on milk compositional analysis, samples should not be frozen prior to testing.

2.3.4 Surface tension

 $\frac{1}{1}$

 \bullet .

The emulsion property of milk because of the colloidal solution of fat, calcium caseinate and calcium phosphate, shows its peculiar surface tension. The surface tension is lowered by the increase of fat content. The surface tension of water and milk is 5:2 meaning thereby that the surface tension of milk is lower than that of water. The accumulated soluble colloidal substances of milk chiefly proteins lower the surface tension. (Srivastava, 1993)

2.4 Factors affecting the composition of milk

Milk composition is the result of many influences within the cow and in the environment. Farmers can alter many factors to increase milk yield and improve its composition, while some factors are beyond the farmer's control. There obvious differences in milk composition between breeds, but the differences between individuals within a breed are often greater. The most obvious difference between average milk composition for breeds is fat percentage.

The nutritional factors affecting milk composition generally rations that stimulate milk yield will depress fat percentage and boost SNF percentage. Conversely, any rations that depresses milk yield will increase fat content but depress SNF content. Good

levels of feeding tend to stimulate milk yield and lactose percentage, and lower fat, protein and mineral percentage by dilution. Underfeeding tends to have an opposite effect.

With advancing age and increasing number of lactation, there is a gradual decline in milk fat percentage and SNF percentage (mainly lactose). Cows calving in good body condition tend to produce milk with a higher fiat content than cows calving in poor condition.

Stage of lactation, pregnancy and persistency will also affect to the milk composition The secretion from the udder immediately after calving, called colostrum, is considerably different from normal milk. Colostrum is higher in total solids, especially the globulin fraction, and ash. Lactose reduced, and fat and casein content are variable. The higher ash content is due to raised iron (10 times the normal level), calcium., magnesium, phosphorus ,and chlorine (Chamberlain, 1989).

Fig 2.2:A Normal Lactation Curve

Once milk composition has returned to normal after calving, the major constituents •follow a changing pattern through the lactation. Fat and SNF percentages are high in early lactation. They fall steadily until the second or third month of lactation and then gradually rise as milk yield falls with advancing lactation.

If milk is agitated in transport, especially when it has not been cooled to 10 $^{\circ}$ C or if vessels are under filled, much of the fat is "churned". At a receiving factory the milk

will be strained, removing much of the fat and lowering the fat percentage of the milk to be used. (Chamberlain, 1989)

The time of the year has an effect on the composition of the milk .although this may be indirect .since the diet of the herd and the amount of green pasture consumed varies with the time of year. Time poorest milk is produced in the spring and early summer when green pasture is bountiful and the richest in autumn and winter. (Meyer, 1987)

2.5 Concentrated milk products

2.5.1 Sweetened condensed milk

Whole milk contains approximately 87% water, which, in addition to its protein and carbohydrate content, makes it particularly susceptible to microbiological spoilage. Consequently, various methods have been develop to preserve food value, with milk concentrate manufacture being one such approach.

Borden's development of an evaporation technique, coupled with the preservative effect of sugar and Meyenberg's development of sterilized canning have given rise to the products of sweetened condensed milk and evaporated milk, respectively. Developments in technology since Borden's day have yield other milk concentrates, used mot only as substitutes for fresh milk for domestic consumption, but also as important ingredients in the industrial manufacture of many foods.

Milk concentrates provide both the nutritional and functional benefits of milk and enable a reduction in the relative transportation cost of milk solids, which is of significance to the widespread world market. Throughout the world, sweetened condensed milk, manufactured in various forms, is the most commonly consumed milk concentrate. Indeed, it is the most commonly consumed milk product, finding particular use in tropical countries where domestic refrigeration is limited and the local dairy industry is not developed.

Milk itself cannot be concentrated sufficiently to achieve a preservative effect through the osmotic pressure of lactose. As milk is concentrated, viscosity problems occur and, in particular, age gelation is encountered at solids levels of >45% for whole milk and >40% for skimmed milk. The low solubility of lactose would in any event, cause it to crystallize, defeating the object. The addition of sucrose to milk, followed by concentration to the required solids level can, however produce a shelf stable product, with an osmotic pressure sufficient to prevent microbiological spoilage. Such a product has been manufactured for many years by dairy industry and is known as Sweetened Condensed Milk .A sucrose ratio (sucrose in aqueous phase) of 62.5% or greater, calculated by

Sucrose_____ x 100 Sucrose + water

is required to achieve an osmotic pressure sufficient to prevent microbiological spoilage, other than by osmophilic yeasts. (Early, 1998) The product is yellowish in color and Sweetened condensed whole milk contains 8% fat, 45% sugar, 20% solids non fat and 27% water. (Byland, 1995)

Sweetened condensed milks are manufactured for sale in the retail and industrial market places. The confectionary industry is a major user, although in some parts of the world, such as the Far East , the West Indies and Venezuela, sweetened condensed milks are an important substitute for liquid milk for domestic consumption.

Full cream sweetened condensed milk for the retail market is commonly manufactured to two formulations, the EC and US Federal Standards , as shown in

	EC Standard(%)	US Federal Standard(%)
Non Fat milk solids	22.0	19.5
Milk Fat	9.0	8.5
Total Milk Solids	31.0	28.0
Sucrose	43.5	45.5
Total solids	74.5	73.5

Table 2.2 Standards for Sweetened Condensed Milk composition

Source:Early,R.(1998), The Technology of the Dairy Products

 $\tilde{\mathbb{I}}$

The methods for manufacturing full cream and skimmed products are comparable .although preparation of the milk is different. When manufacturing full cream sweetened condensed milk .whole milk is standardized with skimmed milk to produce the required fat to non fat milk solids ratio. For skimmed milk products .the whole milk is processed by centrifugal separation to give cream and skimmed milk.

Whichever route is used .the finished product should be homogeneous, have a smooth texture and a viscosity of 30-40 Mpa.

The shelf life of sweetened condensed milk varies from one year(although often effectively longer) for products packed in flame sterilized cans, to a couple of weeks for product transported by tanker and stored in bulk silos. In the latter case , care must be taken to condition the air in the head space above the product and to avoid condensation on the product's surface. If this is not done the surface will be diluted, which results in a reduction in osmotic pressure .This in turn make the product vulnerable to yeast and mould spoilage.

The sugar confectionery industry uses sweetened condensed milk in the manufacture of toffees, caramels and fudge. With its low moisture content , sweetened condensed milk is an ideal alternative to liquid milk as it minimizes the boiling time. This reduces the tendency for the hydrolysis of sucrose to fructose and glucose which produces a sticky confection , and avoids an over cooked dark caramel with a rough texture caused by protein denaturation.

The use of full cream or skimmed sweetened condensed milk depends on the required quality and flavor. Full cream sweetened condensed milk produces a richer .creamier flavor and is usually used in the more expensive higher quality products. Sweetened condensed milks are also used in the manufacture of milk chocolate and ice cream .In the former .full cream sweetened condensed milk can be blended with cocoa liquor to the desired composition, followed by vacuum drying to produce chocolate crumb. In the latter , sweetened condensed milks can be used as an alternative to liquid milk or milk powders for ice cream manufacture.

Other applications include fudge icing as used by the bakery industry , a source of milk solids in milky flavor gums , cereal bars and chews, and finally, in dairy based chocolate liqueur centers.

Sweetened condensed milk may be made from either whole or skim milk and either be produced for bulk industrial use or packaged in to containers for retail sale. The extended shelf life is derived not from thermal processing, but the reduced water activity level resulting from high solute concentration Refrigeration is, however, required for many types of bulk sweetened condensed milk. (Varnam et al.,1994)

2.5.1 Evaporated milk

This product is made by the evaporation of water from whole milk under a vaccum .Low percentages of Sodium phosphate, Sodium citrate, Calcium chloride ,and /or carrageenan may be added to improve stability. United States standards of identity require that evaporated milk contain not less than 7.9% milk fat and 25.9% total milk solids.(Webb et al.,1987)

2.5.2 Plain condensed milk

Plain condensed milk or concentrated milk has the same standard of identity in the United States as evaporated milk .except that it is not given additional heat processing after concentration, and the optional ingredients are not used(Webb et al.,1987).

2.5.3 Condensed skim milk

This is divided in to two groups; Plain condensed skim milk and sweetened condensed skim milk (Webb et al.,1987).

2.5.4 Condensed butter milk

Condensed or semi solid butter milk is a creamery butter milk which is allowed to ripen to an acidity of 1.6% or more , then condensed (Webb et al., 1987).

*

2.6 Milk Quality and properties of SCM Processing

2.6.1 Method of SCM processing

```
Standardization
    I
Heat Treatment
    i
Evaporation
    I
Crystallization
    i
Canning
    i
```
Storage

The quality of the raw materials for condensed milk is basically the same as that used in the manufacture of ordinary dairy products .There are two other considerations for the manufacture of condensed milk.

1 .The number of spores and heat resistant bacteria in milk.

2.Theability of the milk to tolerate intensive heat treatment without coagulating.

Condensed milk is marked with a stipulated content of fat and dry solids. The figures vary with the applicable standard, but are normally 8.5% fat and 18% solids non fat. The stipulated values percentages are minimum values which must be maintained, but for reasons of economy they should not be exceeded by more than a reasonable margin. Modern automatic standardization systems permit continuous and extremely accurate standardization of both fat content and relation between fat content and solids non fat of the basic milk. This is done so as to conform to legal standards in the finished product_s The standardization of raw milk is done to the required fat and SNF ratio by the addition of skimmed milk or full cream milk powder (Byland, 1995).

The milk has also been heat treated to destroy micro organisms and enzymes which could cause problems and to stabilize the protein complex. Heat treatment is important to the development of product viscosity during storage and is particularly important in the case of sweetened condensed milk. A gel can form if the heat treatment is severe .The milk is usually heat treated at 82°C for 2 minutes if a product with a relatively high viscosity is required. If a low viscosity product is required , the temperature/ time combination should be 116° C for 30 sec.

The addition of sugar is a key step in the manufacture of sweetened condensed milk .It is important that the correct proportion is added as the shelf life of the milk depends on its osmotic pressure being sufficiently high. Two methods are used for addition of sugar.

- **eat** 1. Addition of dry sugar before *hm* treatment
- 2. Addition of sugar syrup in the evaporator.

Å

The stage at which the sugar is added affects the viscosity of the end product. One theory maintain that early addition of sugar can cause the product to become too viscous during storage.

In evaporation , the evaporator is usually of the multistage falling film type. The milk passes through steam heated tubes under vaccum. Boiling take place at between 65- 70°C. The dry mater content of the milk increases as the water is boiled off. The dry matter content is checked indirectly by determining the density of the concentrate. This should be about 1.30 for sweetened whole milk and about 1.35 for sweetened skim milk , when the correct dry matter value has been attained (Byland, 1995).

Sweetened condensed milk must be cooled after evaporation. This is the most critical and important stage in the whole process. The water in the condensed milk can only hold half the quantity of lactose in solution. The remaining half will therefore be precipitated in the form of crystals. If the surplus lactose is allowed to precipitate freely , the sugar crystals will be large and the product will be gritty and unsuitable for many applications. It is consequently preferable to control the crystallization of lactose,so that very small crystals are obtained .The largest crystal size permitted in first grade milk is 10 Micrometer. These crystals will remain dispersed in the milk under normal stage temperatures, 15-25°C.

The required crystallization is accomplished by cooling the mixture rapidly under vigorous agitation, without air being entrapped. Seed crystals in the form of finely ground lactose crystals are added at a rate of about .05% of the total mix .either as powder or as a slurry .The mixture is cooled as quickly as possible to 15-18 °C after continuous and vigorous agitation for about one hour. The viscosity of sweetened condensed milk is high, which means that a very robust agitator is needed in the crystallization tank. The cooled condensed milk is pumped to a storage tank where it is kept until the following day to allow the crystallization process to be completed. After the cooling and crystallization step, canning is conducted by canning machines which automatically fill and seal the cans. The canning temperature is selected to give the lowest possible froth formation (Varnam,1994).

uv

Chapter 03

Materials and Methods

3.1 Past data analysis in Problematic and Non Problematic period

3.1.1 Variation in Superheating time in the production of Sweetened Condensed milk during year 2002 and 2003

3.1.1.1 Materials

Manufacturing control books year 2002 and 2003

3.1.1.2 Procedure

Past and current data for super heating time were collected for each batch .Monthly average of super heating time was calculated. Graphs were plotted between the monthly average of super heating time and the month.

3.1.2 Correlation in the viscosity of final products of SCM (Metal cans) with their TS content

.3.1.2.1 Materials

Manufacturing control files Box files

 $\tilde{\mathbb{E}}$

3.1.2.2 Procedure

Past and current data (year 2003) were recorded for the day old viscosity during the storage period; with the initial TS content of the product. Graphs were plotted to identify the variation of viscosity due to TS content. Data was statistically analyzed by linear regression (5% level of significance I) to find whether there isrcorrelation between TS content and viscosity.

3.1.3 Variation in the fat and SNF in fresh milk during the year 2002 and 2003

3.1.3.1 Materials Box files Standardization dockets

3.1.3.2 Procedure

Past and current data for fat and SNF in fresh milk during the year 2002 and 2003 were collected daily wise. Averages per month were calculated. Graphs were plotted with average fat and SNF percentage with month.

3.2 Composition analysis of fresh milk in non problematic period

3.2.1 Collection of Samples

100 ml of samples were collected randomly in to sampling bottles after agitating the milk in storage tanks. The main aim of plunging/agitating was to avoid the effect of fat separation and to get a homogeneous sample. After that the samples were stored in a refrigerator at 4°Cuntil further analysis.

3.2.2 Preparation of Samples

 \mathbf{I}

Before weighing the relevant amount, randomly collected samples were shaken thoroughly in a upward and downward movement and also using a spatula to make the sample homogeneous.

3.2.3 Estimation of protein content in fresh milk during non problematic period.

3.2.3.1 Reagents

Boric acid Sulfuric acid 95-97% Hydrochloric acid .1M Potassium Sulfate Copper Sulfate Pentahydrate Ethanol about 95% extra pure Methyl red indicator pH 4.4 - 6.2 NaOH Pellets NaOH Solution Bromocresol green Distilled water

3.2.3.2 Equipments

Mineralization apparatus, complete, Buchi 435,12 places

Gas washing bottle Buchi 412

Digestion tubes, Buchi

Distilling apparatus, Buchi 316

Beaker, tall form with spout, 250 ml

Titration stand Metrohm 719

Cylinders, graduated, low form class B, 25 ml

Volumetric flask, class A, Stopper RN 19/26, 500ml

Glass electrode for pH measurement, with Pt 1000 sensor Metrohm 6.0228

Chronometer table type

ž

Weighing boats, parchment, nitrogen free

Wash bottles

Pipettes, volumetric with single mark, class as, 5ml

Idem, 10ml

Aluminium foil

Glass beads

3.2.3.3 Procedure

Preparation of reagents

Boric acid solution with indicator

Solution A

6g of boric acid was weighed in to a 100ml of volumetric flask and 900ml of distilled water was dissolved in it.

 $\ddot{}$

Solution B

40 mg of Methyl red and 200mg of Bromocresol green were put in to a 250ml of volumetric flask and dissolved in 240ml 95% ethanol.

Working Solution

10 ml of solution B was added into solution A and mixture was filled up to the mark with distilled water.

HCI0.1M

The content of an ampoule of .1M HCI titrisol was diluted with distilled water in a 1000ml of volumetric flask.

NaOH Solution 32% w/v

640 g of NaOH pellets were weighed in to a 3000ml beaker and dissolved in about 1.5 I of distilled water. It was allowed to cool and transferred to the reservoir of the distillation unit after making up to 2 I with distilled water.

Mineralization

10g of Potassium Sulfate, 45mg of Copper Sulfate Pentahydrate and 1-2g of fresh milk samples were weighed in to digestion vessel. One digestion yessel was kept as a blank without the milk sample. Contents were mixed thoroughly. And 15ml of cone. (95-97%) sulfuric was added in to each vessel.They were transferred in to the mineralization unit. It was kept *VA* hours for complete mineralization. Then vessels were removed from the heating and allowed to cool for 10 min. Afterwards, 50 ml of distilled water was added cautiously in to each vessel and the vessels were covered by Al foils.Samples were proceeded immediately with the analysis by distilling.

Distillation and titration

Each digestion vessel was placed on the distillation device. The recipient 50 ml boric acid solution was introduced. 60ml of 32% of NaOH was added in to the digestion vessel. It was distilled for 4 minutes. Titration was conducted to the distillate with 0 .1M HCI by calibrating the pH meter to pH 4.65.

3.2.3.4 Calculations

1ml of HCI 0.1mol/l corresponds to 1.4007mg Nitrogen .The total Nitrogen content, expressed in g/100g product is,

(V1-V2) x F x 1.4007 x 100 m

- V1 Volume of HCI .1M used for the determination in ml
- V2 Volume of HCI .1M used for the blank determination in ml
- F Correlation factor for the molarity of the HCI solution
- m mass of the test portion

3.2.4 Estimation of the total ash content in the fresh milk samples

3.2.4.1 Reagents

Nitric acid Distilled water

3.2.4.2 Equipments

Quartz crucible, low form opaque, 67 x 45 mm, 80ml Tongs - Stainless steel with platinum shoe Muffle furnace, programmable (Heraeus M104 with program controller) Ceran hotplate, 300 x 300mm/water bath

3.2.4.3 Procedure

At first platinum or quartz crucibles were decontaminated by dipping them in nitric acid solution and followed by washing with distilled water. Then they were transferred in to the oven set at 130°C for 30 min They were weighed to the nearest 0.1mg (m₀). Samples were weighed in to the crucibles to the nearest 0.1mg (m1). Test portions in the crucibles were dried using a water bath. Crucibles were transferred to a programmable furnace. Afterwards, platinum or quartz crucibles were heated in a mufflefurnace at 550 °C for 30 "min.Crucibles were transferred to a decicator for about 45-min to cool to room temperature. When the ashing step is finished, product was checked whether it had been completely ashed. Crucible was weighed with the residue $(m₂)$.

3.2.4.4 Calculations

Mass percentage of total ash in the product

 $m2 - m_{o x 100}$ **m1** - m₀

 m_0 mass of the empty crucible in g

 $m1$ - mass of the crucible and test portion in g

m2 - mass of crucible and ash in g

Chapter 04

Results and Discussion

4.1 Results

 $\ddot{\cdot}$

4.1.1 Past data analysis in problematic and non problematic period in year 2002 and 2003

4.1.1.1 Variation in Super heating time in the production of SCM (Appendix i)

Graphs represent high average super heating time in problematic period(May to September) than non problematic period.

Fig 4.2 Average Super Heating time during year 2003

4.1.1.2 Correlation in the day old viscosity of the final products of SCM (metal cans) with their TS content.

(Appendix ii)

 $\ddot{}$

Correlations (Pearson)

Correlation of TS and Day old viscosity = 0.195

Since -0.55 < Correlation coefficient < 0.55

according to the past data .There is no correlation in the viscosity of the final products of SCM (metal cans) with their TS content.

4.1.1.3 Variation of fat and SNF in fresh milk during the year 2002 and 2003 (Appendix iii)

Due to the graphical representation, average fat percentage is less during the problematic period and there is no significant variation in solids non fat content during the year 2003.

Fig 4.3 Average Fat and Solids non Fat variation during year 2003

According to the graph, there is a significant reduction in average fat percentage during the problematic period in year 2002 while there is a less variation in solids non fat content.

(Appendix iv)

Fig 4.4 Average Fat and Solids non Fat variation during year 2002

4.1.2 Composition analysis of Fresh milk in non-problematic period

(Appendix v)

Correlations: SNF, Protein

H0:There is no correlation between SNF and Protein content

H1: There is correlation between SNF and Protein content

P-Value = 0.001

Since P < 0.05 .reject HO at 5% level of significance.

Therefore there is correlation between solids non-fat and protein content in fresh milk (Appendix vi) .

Correlations: Fat, Protein

H0:There is no correlation between Fat and Protein content H1: There is correlation between Fat and Protein content

y P-Value = 0.000 Since P < 0.05 reject HO at 5% of significance level. Therefore there is correlation between fat and protein content in fresh milk (Appendix vii)

Correlations: SNF, Ash

H₀: There is no correlation between SNF and Ash content H1: There is correlation between SNFand Ash content

 $P-Value = 0.006$ $P < 0.05$

Reject HO at 5% of significance level.

Therefore there is a correlation between solids non fat and ash content in fresh milk (Appendix viii)

Correlations: Fat, Ash

H0:There is no correlation between fat and Ash content H1: There is correlation between fat and Ash content

Pearson correlation of Fat and Ash = 0.323

 $P-Value = 0.222$ $P > 0.05$

Do not reject HO at 5% of significance level.

Therefore there is no correlation between fat and ash content in fresh milk

4.2 Discussion

The objective of this study has been extended to find the effect of time temperature combination of the heat treatment on viscosity while finding a instinct factor which cause low viscosity development of sweetened condensed milk during the specific time period of the year. Results of super heating time temperature combination revealed that the milk from problematic season is subjected to longer concentrating period at 70°C than non problematic period. The reason for this is to evaporate more water to adjust the concentration of the milk solids.

Even under the constant parameters throughout the year .development of low viscosity is noticed during May to September. Past and current data are recorded for fat and solids non fat in fresh milk which has been used in the production of sweetened condensed milk. A study which had been undertaken on the fat and solids non fat content on Punganur cows by Reddy(1995) revealed that fat level of milk is varied during the lactation of the cows. He revealed that high fat levels were observed during late lactation and summer season and higher values of solids non fat were observed during late lactations and winter seasons .This is in agreement with the findings by analyzing the past and current data .So, low fat percentage is observed during the problematic period while there is no significant variation in solids non fat. However, studies on variation of milk constituents of Jersey cross bred cows during different lactation by Ayyadurai(1995) .revealed that the percentages of fat and solids non fat of milk don't follow a uniform trend throughout the lactating phase.

Data pertaining to 16 samples were analyzed to assess the variation of compositional factors in fresh milk during the non problematic season within a period of one month. Significant correlation of ash and protein content with solids non fat is found as they contribute to the total solids non fat content in milk. Furthermore, a positive correlation of fat and protein is observed. The structural arrangement of casein micelles surrounding the fat globules can be the reason for that. So, the decreasement in the casein/protein content could be observed with the reduction of fat content. As casein is a one of instinct factors which affect the viscosity .It can be assumed that protein content has a possibility to show reduction within the problematic period due to the low values of fat.

Chapter 05

5.1 Conclusions

According to the statistical and graphical analysis of past and current data related to the sweetened condensed milk production and raw milk, it can be concluded that instinct factor which is responsible for the low viscosity development in SCM is protein. Because there was a correlation between fat and protein content in the milk in non problematic period. So, it can be argued that low fat content in problematic period leads to the low protein content.

5.2 Suggestions

Experiment should be continued for the compositional analysis in problematic period and compare the results with non problematic period to confirm the compositional variation.

Suitable stabilizer can be used during the problematic period and before that trials should be conducted with several stabilizers to identify the most suitable stabilizer.

Raw milk can be standardized to the normal milk composition in non problematic period concerning more about the increasement in the protein content.

References

- Atherton, H.V.and Newlander, J.A. (1987) chemistry and Testing of Dairy Products.4th edn.CBS.Delhi, pp.46-48.
- Ayyadurai.K.,Kumar,C.N.,Vijaylakshml,R.,Mohamed,M.,and Kahn,H.(1995)studies on Variation of milk constituents of Jersey cross bred cows during different Lactation.J.Indian Journal Dairy Science, v48, pp.591-593.
- Byland,G.(1995)Dairy Processing Hand Book,Tetrapack Processing Systems ABS, Sweden, pp2-4.
- Chamberlain, A. (1989) Producing and Distributing milk, Milk Production in tropics, Educational Law Priced,London, pp. 158-161.
- Early, R. (1998) The Technology of the dairy Products. 2nd Ed. Blackie Academic and Professional,pp.238-239.
- Fennema, O.R. (1976) Characteristics of edible fluids of animal origin-milk, Principles Of Food Science.2nd edn.Marcel Dekker, Newyork, pp.620-621.
- Hurley, W.L. (2002) Milk composition, Department of Animal Sciences, University of Illinois.
- Meyer,L.H.(1987)Milk and milk products, Food Chemistry, CBS,Delhi,pp.293- 297.
- Pouliot.Y.and Boulet,M.(1995)Observations on the seasonal variations in the salt balance of concentrated milk .J.International Dairy Journal,v.5,pp.75-85.
- Rai, M.M. (1996) Milk Products, Their composition and method of preparation, Dairy Chemistry Animal Nutrition.3rd edn.Kalyani,Newdelhi,pp.120-121.
- Reddy,S.R.,Murthy,P.R.S.,Rao,K.S.,and Suresh,J.(1995)Seasonal Variation in Fat and SNF content of milk from Punganur cows.J.Indian Journal Dairy Science , v.4,pp.53-54.

- Srivastava.S.M, S.M. (1993) General characteristic of milk, Milk and its Properties.2nd edn.Kalyani, New Delhi, pp.20-21.
- Vanstone, E.and Dougall, M.B. (1982) Milk fat, Principles of Dairy Science, Surjeet, Delhi, pp.35-36.
- Varnam.H.A.and Southerland,J.P.(1994)Milk and Milk Products, Technology chemistry and Microbiology, Chapman and Hall, London, pp..
- Webb, H.B.,Johnson, H.A.and Alford, A.J. (1987) Lactose, Fundamentals of Dairy Chemistry.2nd edn.CBS, Delhi, 929p.

Whitnah.C.H. (1956) J.Agr.Food Chemistry,v. 10,pp.295-298.

Appendix i

Year 2002

 \sim

Year 2003

 $\ddot{}$

 \bullet

Appendix ii

 $\hat{\mathbf{r}}$

 \overline{a}

 \overline{a}

35

 $\ddot{}$

 \mathbb{R}^2

 $\ddot{}$

 $\ddot{}$

 ψ

 $\ddot{}$

 $\ddot{}$

36

 $\ddot{}$

Appendix iii

 \bar{z}

 $\hat{\mathbf{c}}$

 $\hat{}$

 \mathbb{Z}^2

 $\bar{\mathcal{A}}$

 $\overline{}$

Average fat and Solids non Fat variation Year 2003

 $\frac{1}{2}$

 $\ddot{\cdot}$

38

 $\ddot{}$

Appendix iv

 $\ddot{}$

Average fat and SNF variation in year 2002

 \mathcal{L}^{max}

Appendix v

 $\ddot{}$

 $\ddot{}$

 $\ddot{}$

 $\ddot{}$

Appendix vi

Correlations: SNF, Protein

H0:There is no correlation between solids non fat and protein content in fresh milk H1: There is correlation between solids non fat and protein content in fresh milk

Pearson correlation of SNF and Protein = 0.775

 \tilde{z}

P-Value = 0.001

P < 0.05

Reject HO at 5% of significance level.

Therefore there is a correlation between solids non fat and protein content in fresh milk

Appendix vii

J.

Correlations: Fat, Protein

H0:There is no correlation between fat and protein content in fresh milk H1: There is correlation between fat and protein content in fresh milk

Pearson Correlation coefficient of Fat and Protein = 0.798 P-Value = 0.000 P < 0.05 Reject HO at 5% of significance level. Therefore there is a correlation between fat and protein content in fresh milk

Appendix viii

Correlations: SNF, Ash

HO: There is no correlation between solids non fat and ash content in fresh milk

H1: There is correlation between solids non fat and ash content in fresh milk

Pearson correlation of SNF and Ash = 0.651

P-Value = 0.006

P < 0.05

Reject HO at 5% of significance level.

Therefore there is a correlation between solids non fat and ash content in fresh milk

National Digitization Project

National Science Foundation

Certification of Scanning

I hereby certify that the scanning of this document was carried out under my supervision, according to the norms and standards of digital scanning accurately, also keeping with the originality of the original *document to be accepted in a court of law.*

"This document/publication was digitized under National Digitization Project of the *National Science Foundation, Sri Lanka "*