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A NEW TECHNOLOGY FOR MILKFAT

Thesis submitted for the

degree of

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at

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by

Cheng Tet Teo

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Chapter 1

Section 1 INTRODUCTION AND OBJECTIVES

1.1 Introduction

Milkfat is one of the most complex of all fats, consisting of numerous different triglycerides as well as complex fats, such as phospholipids and cerebrosides, and traces of mineral substances (Walstra and Jenness, 1984). It is liquid above 40°C and usually completely solidified below -40°C. At intermediate temperatures, it is a mixture of crystals and oil, the oil being the continuous phase. On cooling, a liquid fat usually forms small crystals at first (in rapid cooling, often in α modification) of needle to platelet shape, 0.1-3.0μm long, with a ratio length:width:thickness of 4:2:1, (Jensen, 1967). As crystals grow, similar shaped but larger crystals in aggregate, which can be as much as 50μm across, are formed.

Crystallisation is initiated by the presence of suitable crystal nuclei (centres of crystallisation) in the liquid fat as a few molecules gather in molecular aggregates where the potential energy is reduced to the minimum. Lowering of temperature strongly influences the rate of nucleus formation. Homogeneous nucleation occurs when crystals nuclei are formed spontaneously from a pure melt. Usually a supercooling of 3-5°C is needed to start a nucleation, and a further temperature decrease by a few degrees can caused rapid nucleation. In practice, for fat in bulk, nucleation is always heterogeneous, i.e. crystallisation always starts at the surface of extraneous particles, often called catalytic impurities (Skoda *el at*, 1963).

The crystallisation rate is the resultant of nucleation rate and crystal growth. Other factors maybe:

1. Crystallisation is exothermic and temperature may rise locally, hence crystallisation rate decreases, particulary in bulk fat not stirred during cooling, where temperature may increase by as much as 5°C.

2. Fat crystals being impure (that is, they contain foreign triglyceride molecules, particularly after rapid and deep cooling), solid fat may recrystallise, and the amount of crystalline fat increases if temperature is kept constant.

3. Polymorphic transitions may also take place, perhaps in the course of recrystallisation of mixed crystals.

When fat is in the form of globules, crystallisation is generally slower.

If crystallisation is very slow, large crystals are formed, particularly where few crystal nuclei are present, as is the case with mild supercooling. Rapid cooling to a low temperature leads to numerous small crystals, e.g. 10^12 ml^{-1}. But any slow crystallisation afterwards tends to increase crystal size. Small crystals are slightly more soluble than large ones, which tend to grow at the expense of small ones (Ostwald ripening). This process is enhanced by temperature fluctuations. Any slow crystallisation, whether by polymorphic transition or by separation of mixed crystals, increases crystal size. It may also lead to more or stronger solid networks.

Milkfat cream of 40-42% fat content, being the established optimum (Sammuelsson and Vikelos, 1971, Frede *et al*, 1982a and 1982b, Mercer, 1985), is the main raw material component in the process of buttermaking. Cream can be described as an oil-in-water emulsion of fat with protein, lactose and minerals dissolved in water. Ninety-nine percent of the fat in cream is present in the form of globules ranging from 1-10μm in diameter. Each fat globule is made up of 98% triglycerides which largely determine its properties; 0.8% phospholipid and the remaining traces of diglycerides, monoglycerides, sterols, lipids, and flavouring compounds. The triglycerides component, to a large extent, governs the physical properties of the milkfat, such as melting and solidification behaviour.

Butter is essentially a water-in-oil emulsion containing 80% fat or more, with fat in a plastic condition. Therefore, a phase inversion is necessary to convert cream to butter.

One of the main short-comings of butter is that it is not easily spreadable at refrigerator temperatures, as opposed to vegetable oil based margarine or low fat yellow spreads. As a comparison, the scale of butter hardness (thus spreadability) of early spring and summer butter is 950 and 2400 g respectively (MacGibbon *el at*, 1987) whereas for that of margarine is 200-300g.

If the spreadability of butter can be further improved, the commercial attractiveness of milkfat would be greatly enhanced. This has made possible continued interest in finding ways of improvement. There are mainly two areas where further improvements are being made.

The first one is by modification of the processing method augmented with suitable mechanical equipment. The processing method would involve the handling and treatment (i.e. crystallisation) of cream prior to buttermaking. The Alnarp process and its modified form has been studied by a number of workers, including Samuelsson and Peterson (1937), Dolby(1954), and Dixon(1974). The effects of longer crystallisation holding times have also been investigated by workers, such as, Precht and Peters(1982), Danmark and Bagger(1988,1989), Ulberth(1989) and Szakaly and Schaffer(1988).

Secondly, by changing the rheological properties of the milkfat or it fat fractions.
The work reported here consisted mainly of investigations into ways of changing the rheological properties of the milkfat fractions and thereafter recombining these fractions to make butter. The main consideration is in the area of butter spreadability and its textural properties.

1.2 Objectives

It is postulated that fat crystals formed in the fat globules in cream have a form of fine needles and platelets. In butter, this network of fine needles or platelets are randomly distributed. Such a structure would requires a high proportion of liquid fat for the butter to be spreadable. At low (3-5°C) or refrigerated temperature, more of the fat is solid, resulting in a harder butter texture.

Now, if the crystalline milkfat could be produced in a spherical form and the normal composition of the milkfat retained, the butter would be spreadable when there was much less liquid present.

Furthermore, if milkfat fractions which melt at intermediate temperatures could be encapsulated in a spherical form, with the milkfat fractions which melt at higher temperatures, then the butter spreadability would not changed significantly with increase in temperature up to 35°C. Any increase in flavour of such butter would be more due to enrichment into the fat with low melting temperatures through the fractionation process.

The primary objective of this research was to change the rheological properties of milkfat by different means into spherical forms and to evaluate butters made using such spheres. The various methods of producing fat spherulites are explained in the later sections.

This research work are categorized under:-

1.2.1 Homogenisation

To change the rheological properties of milkfat for buttermaking, milkfat fractions were recombined with milk powder in water and homogenised to produce a cream.

1.2.2 Different Aspects of Producing Fat Spherulites

Methods under investigation included homogenisation, creaming, spray drying, spray cooling, freeze drying, and the use of inert polymer spheres.

1.2.3 Different Methods of Buttermaking

Investigations included the use of different combination of fat fractions and acetone fractionation of milkfat to yield purer and more defined milkfat fractions; varying fat content in cream; crystallisation of fat fractions, and cream from these fat fractions; and a review of the conventional method of buttermaking to improve butter spreadability.

Of signaficant importance were the use of the milkfat spherulites produced (as mentioned in 1.2.2) in different combinations to assess its viability in buttermaking.

1.2.4 Testing the Properties of the Butters

The main parameters were sectility hardness; fat content; stand-up and oil-off; and taste.

Chapter 2

Section 2 LITERATURE SEARCH

2.1 Literature Search

Essentially, two CD-ROM (Compact Disc - Read Only Memory) literature searches, namely, Food Science and Technology (1969-1991) and Animal Production and Dairy Technology (1973-1991) Abstracts were carried out on subjects related to rheological properties, crystallisation, fractionation, emulsification and butter spreads products on milkfat.

The composition, structure, physical and chemical properties of milk have been exhaustively investigated and a comprehensive account can be found written by Walstra *el at* (1984), Fox, ed (1983), Schmidth (1982), Jenness (1988), and others.

The study of milkfat crystals from milkfat components in the process of crystallisation and subsequent fractionation to obtain different melting point fat fractions have also been widely publicized. These milkfats are in use as ingredients in specific food applications such as confectionery, chocolate, baking and other lipid-containing foods. Of particular interest is the crystallisation of recombined milkfat (i.e. emulsified state) which produces stable fat spherulites and crystallisation of milkfat fractions for the purpose of making softer butter of composition similar as normal butter.

As far as can be ascertained, where the complete composition of milk cream or recombined cream is used for buttermaking; the use of mechanical means can reduce the butter hardness to a certain extent, but it has not been shown to be possible to produce a butter of hardness which is spreadable at refrigerated temperatures (3-5°C) and at the same time having the same textural properties of normal butter.

A number of papers have indicated some degree of success in the reduction of butter hardness through mechanical means (Munro, 1982) but by and large the significant

achievement was in the control of the crystallisation/cooling of cream/milkfat commonly known as the Alnarp Method or its modified form. Some of these methods are described in more detail later as well as methods which have shown positive outcome.

Although various methods have been in use they can be generally grouped into one of the following classifications in the manufacture of spreadable butter or yellow spreads which invariably could not be strictly termed as "butter" :-

1. Low or Reduced Spreads - where the milkfat content is less than 80%.

2. Blended Spreads - mixtures of dairy and non-dairy spreads.

3. Soft Dairy Spreads - made from low melting dairy fat fractions where not all the components are utilised.

The concept of changing the crystalline milkfat from needles or platelets shaped matrix into a spherical form thus producing a butter which is more spreadable have not been well established and tested. No research work was published in the areas related to the encapsulation of different milkfat fractions into spherical form; in particular, the spray cooling methods which form part of this investigative work.

Chapter 3

Section 3 OVERVIEW

3.1 Overview

The structural heterogeneity of milkfat gives milkfat its complex and flavourful nature but it also makes milkfat less suitable in certain applications (i.e., it makes hard butters and soft chocolates, etc). The health aspects related to milkfat have also contributed to the downturn in the consumption of milkfat in general and being steadily replaced by vegetable fats. To alleviate the continual decline of milkfat in the fats market, the dairy industry fractionates milkfat to provide food ingredients with specialized characteristics for specific applications. However, only some of these fractions have applications while others, such as the intermediate fractions, have limited uses in relation to say, initial milkfat.

A high percentage of milkfat is used in buttermaking in one form or other. It is a well known fact that one of the main disadvantages of butter in comparison to that of margarine is its spreadability properties. Butter is not easily spreadable at refrigerated temperature of 3-5°C. Jebson *el at* 1989 postulated that if the crystalline milkfat could be produced in a spherical form or spherulites (instead of the normal needle or platelets shaped fat crystals) and the normal composition of milkfat retained, the butter would be spreadable when there was much less liquid fat present. Furthermore, if the fractions which melt at intermediate temperature could be sited centrally in the spheres, with the fractions which melt at higher temperatures surrounding them, then the spreadability would not change significantly with increasing temperature until a temperature of around 35°C.

The production of fat spherulites from homogenisation, spray drying and spray cooling were among some of the methods used. The use of these spherulites in buttermaking will be described in detail. In became clear that these spherulites, when incorporated into butters, give products with significantly different physical characteristics. It is

expected that the manufacture of milkfat spherulites from different types of fractions would have good potential in tailoring milkfat to specific requirements.

Further improvements have also been made in the coating of spherulites where one fraction of milkfat can be protected from its environment by a coating of another fraction. The coating of a high melting point fat particles with a lower melting liquid fat producing coated fat spherulites through a spray cooling method was most encouraging. However, in the manufacture of coated spherulites having a higher melting point surface coat a number of practical problems were encountered. This was largely overcome by placing the lower melting point solid fat particles in a liquid suspension of higher melting point fat prior to spray cooling to produce the coated spherulites. Further work is required in this area. The use of these coated spherulites in buttermaking did indeed resulted in a more spreadable butter.

In the course of the research, the following were noted:
A more spreadable butter was made by modifying the rheological properties of the milkfat fractions into spherical form and then recombining all the components into butter. Further work can be done to improve the apparent graininess of the butter by partial recycling of the single fractionation process to produce a higher percentage of the higher melting point fat fraction and investigate into the use of a colloid mill to grind the crystallised soft fraction fat crystals into smaller sizes.

By a process termed, Spray Cooling, 100% milkfat or its fractions powder can be produced. Further work can be done to look at its uses in the industry; storage and packaging problems; and its physical and chemical properties.

Using a slightly modified Spray Cooling technique, coated fat spherulites from different fat fractions can be produced.

The use of coated fat spherulites in buttermaking further confirmed the postulation that modifying the crystalline milkfat into spherical form give products with significantly different physical characteristics.

Chapter 4

Section 1 EFFECTS OF HOMOGENISATION AND EMULSIFIERS ON MILKFAT

1.1 Introduction

1.1.1 Homogenisation

Homogenisation of milkfat in serum causes disruption of the fat globules into much smaller ones resulting in a much a larger overall total surface area. Furthermore, as it subdivides the fat globules into smaller globules, becomes less polydisperse (Walstra *el at*, 1984) and thus retards the formation of a cream layer in a low viscosity cream. It also increases cream viscosity because of the increased number of the fat globules formed now have a larger surface area. Depending on the type of protein present the disruption is determined by the collision rate between these newly formed globules without a protective membrane, and the adsorption rate of surface-active material from the serum phase.

If the adsorption rate of new surface-active materials, such as casein and/or whey proteins, is much greater than the collision rate, the result of homogenisation will be a micronisation of the fat emulsion containing coated fat globules or fat spherulites. Increasing fat content increases the collision rate while at the same time decreases the adsorption rate since the amount of available surface material, relative to fat, is lowered. Finally, at very high fat content, the globules inevitably are pressed together and agitation of 80% or more of fat content in cream usually causes a phase inversion and a water-in-oil emulsion is obtained.

Homogenisation and recombination give rise to fat globules that differ markedly in properties from the natural ones (Fig 1) but these properties depend on variable conditions during treatment. Recombined fat globules have a completely new surface layer as shown on Fig 2, while homogenised globules are partly covered with natural

membrane material. Most conspicuous is the change in size, the globules usually being smaller. Smaller fat globules are more stable in almost every respect, particularly to creaming and coalescence.

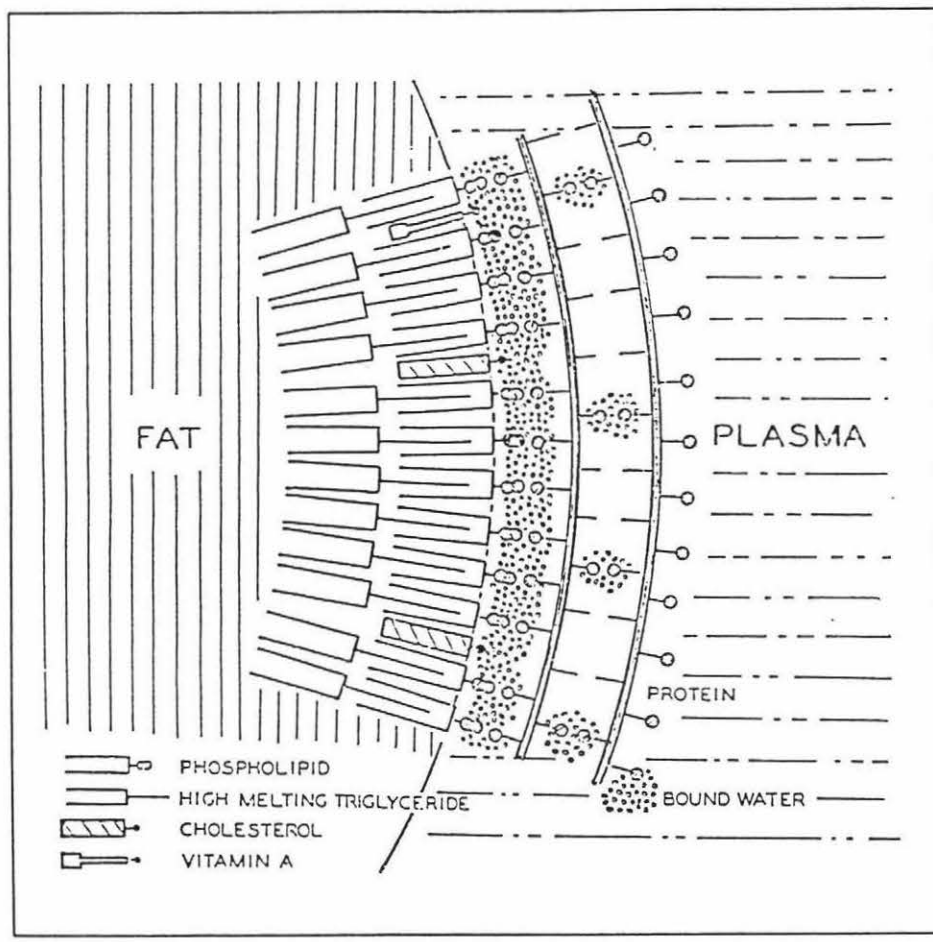


Fig 1 Diagram of Natural Fat globule (Walstra and Jenness, Dairy Chemistry & Physics)

The primary layer consists of a monolayer of radially orientated phospholipid molecules, their hydrocarbon chains anchored in the fat phase, and their hydrophillic residues, comprising two ionogenic groups, turned towards the aqueous phase. A secondary layer follows, composed of uncoiled molecules of the membrane protein, the main chain lying tangentially to the globule surface. It is possible that the membrane on the more or less solidified fat globules may under some circumstances become brittle and develop cracks, through which a part of the still liquid fraction may be squeezed out thus rendering the fat globule surface partly hydrophobic.

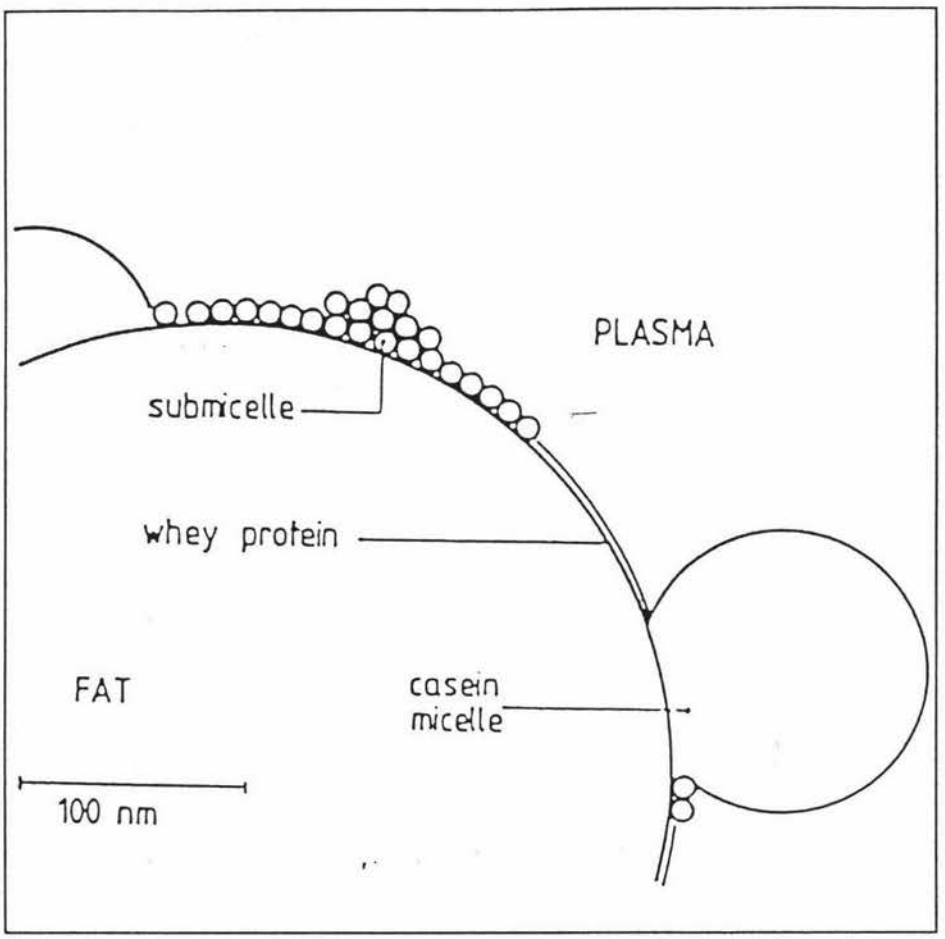


Fig 2 Diagram of Recombined Fat Globule(Walstra and Jenness, Dairy Chemistry & Physics)

Homogenisation enlarges fat surface areas thus creating denuded fat which is largely covered by plasma proteins. Fig 2 depicts a fat globule new surface layer. Adsorbed plasma proteins do not necessarily form a surface layer as coalescence can occur causing a local decrease in surface area. The membrane is composed of casein and whey proteins and the components of natural membrane (i.e. glycoproteins and phospholipid) are largely absent (Walstra *el at*, 1974). The transport of protein to the surface area is mainly caused by convection due to intense turbulence during homogenisation rather then by diffusion. The stability of the fat globules in the state of emulsion can be largely explained on the basis of their complex membrane, where proteins, phospholipid and other minor components are linked.

1.1.2 Relative Emulsifying Activity of Protein Based Emulsifiers

Emulsifiers are substance which reduces the surface tension at the interface of two normally immiscible phases, allowing them to mix and form an emulsion. The key functions of emulsifiers may be summarised as,

1. to promote emulsion stability and control of agglomeration of fat globules;
2. to modify texture, shelf life, and rheological properties by complexing with protein components; and
3. to improve the texture of fat-based food by controlling the polymorphism of fats.

1.2 Experimental

To determine the conditions and parameters of homogenising pressures, percentage milkfat content in serum, rapid cooling, and the effects of the use of milkfat protein based emulsifiers; under which stable fat spherulites can be produced.

The purpose was generally to investigate the effects of homogenising pressure, rapid cooling and the use sodium caseinate in serum on fat spherulite size distribution. Pearce and Kinsella (1978) using the Emulsifying Activity Index, $m^2\ g^{-1}$ have shown that sodium caseinate, which is highly soluble and surface active, has better emulsifying properties than milk based bovine serum albumin, β-lactoglobumin, or whey protein concentrate. Krog *el at.* (1989) showed sodium caseinate concentration (mg/m^2) as a function of the amount of protein absorbed at the surface, the so-called surface load, expressed in mg protein per m^2 surface area of emulsified milkfat. It is important to note that in practice the bulk concentration of protein required would be much higher as not all proteins in solution are absorbed.

1.2.1 Equipment

1.2.1.1 Homogeniser

A Rannie Two Stage homogeniser Model LAB Hyper: Type 12.50H with a fixed rated capacity of 100l/hr capable of operating at a maximum pressure of 6.0795 X 10^4 kPas and inlet pressure of 300-1000 kPas.

1.2.1.2 Particle Analyzer

A Malvern Master Particle Sizer M6.10[(a)] was used for the early part of the work (Table 1-5) in the determination of fat spherulite sizes. The Malvern used light scattering techniques to measure particle diameter of 1μm upwards and calculates particle size distribution on a volumetric basis.

At a later stage an updated and higher version of Malvern MasterSizer E Ver. 1.1[(b)] particle analyzer was made available which was able to analyze the fat spherulites of size and concentration in the range of 0.1μm (Table 8).

1.2.1.3 Spectrophotometer

A Varian Series 634 spectrophotometer using a 1cm pathlength cuvette with a 1nm slit and a wavelength of 550nm was selected to obtain the absorbance of the diluted homogenised fat emulsion.

1.2.2 Material

1.2.2.1 Milkfat

Anhydrous milkfat (AMF) constituting 99.9% milkfat and 0.1% moisture were obtained from the New Zealand Dairy Research Institute(NZDRI). Milkfat of various composition and fractions were obtained both from NZDRI and through New Zealand Dairy

Board(NZDB) from Bay Milk Products(BMP) Ltd in Te Puke, New Zealand. Typical nuclear magnetic resonance (NMR) solid fat profiles of the milkfat fractions used are as shown in Fig 3 to 5 for a single stage fractionation from NZDRI and three stages fractionation from both NZDRI and NZDB-BMP.

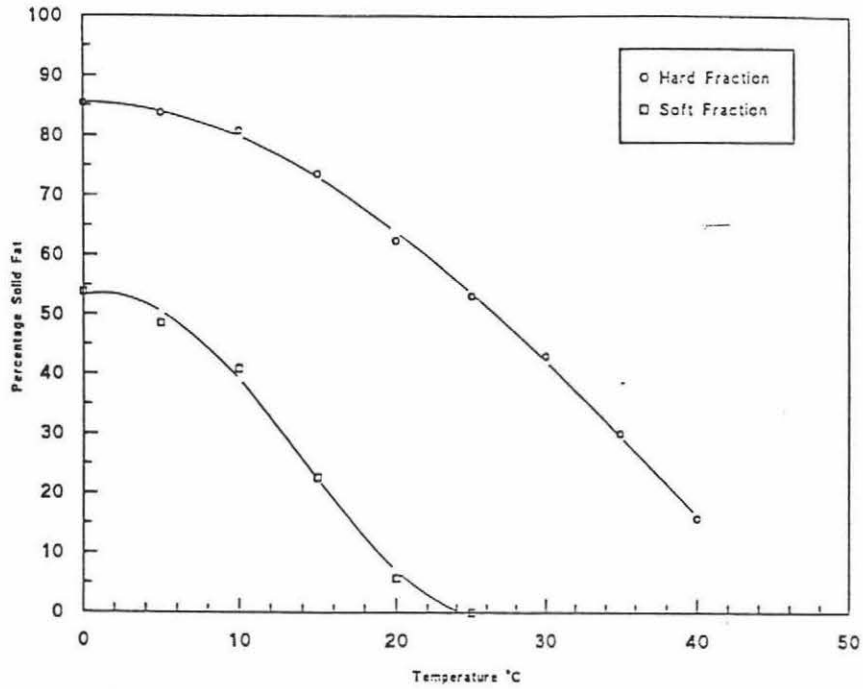


Fig 3 Solid Fat Profile of Fat Fractions from Single Stage Fractionation(NZDRI)

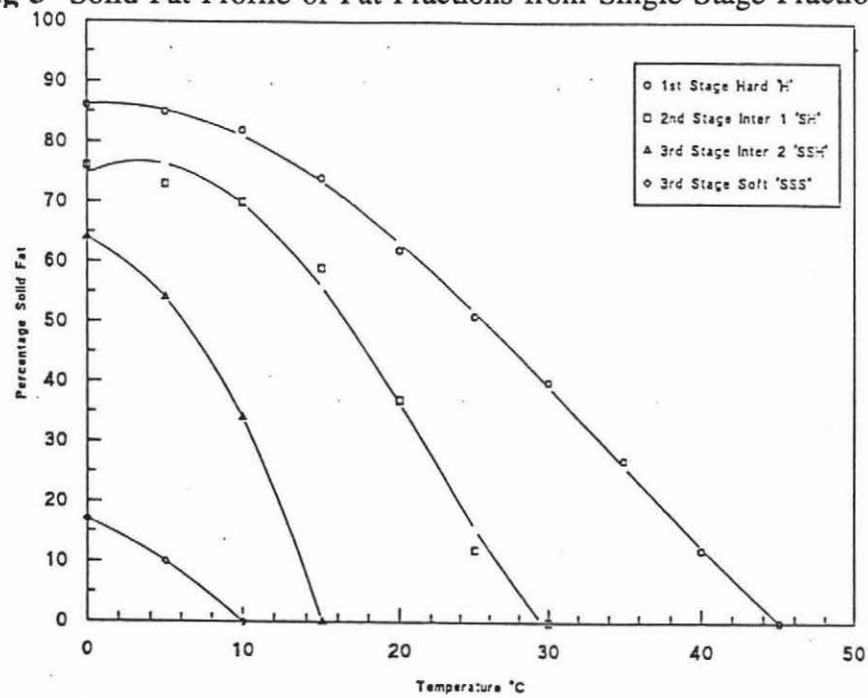


Fig 4 Solid Fat Profile of Fat Fractions from Multiple Stage Fractionation(NZDRI)

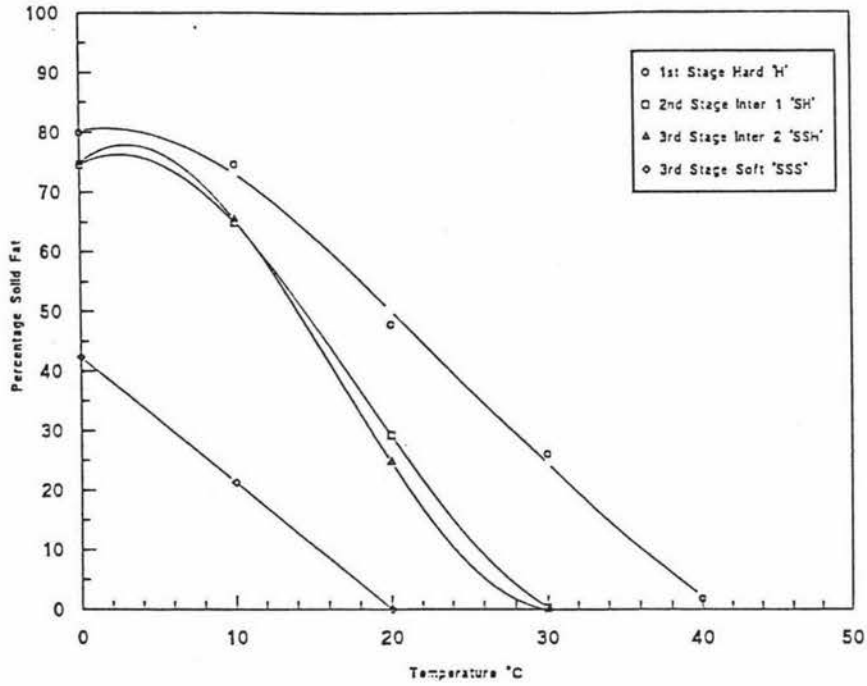


Fig 5 Solid Fat Profile of Fat Fractions from Multiple Stage Fractionation(NZDB-BMP)

1.2.2.2 Emulsifiers

In fat spreads, emulsifiers are essential to maintain a fat-continuous structure, especially during manufacture. They also contribute to the initial nucleation of higher melting triglycerides crystals during the crystallisation process. Four types of materials with good emulsifying properties, namely, sodium caseinate, buttermilk powder, lecithin (egg-based), and whey protein concentrate, were used to produce a stable cream emulsion containing "coated" fat globules or spherulites from anhydrous milkfat and selected fat fractions.

The rationale for selecting these materials was restriction to those occurring naturally in milk or derived from milk products. Lecithin constitutes 60% of phospholipid in milk, however, this is a small quantity considering that phospholipid represent only 1% of the lipids in milk (Riel, 1985). Lecithin has good emulsifying properties and egg-based lecithin was used as it was more readily available. It is important to note that adding these emulsifiers in the creaming process did not constitute an adulteration to the eventual composition of the butter as after buttermaking these components would be mostly in the buttermilk.

1.3 Methods

1.3.1 Effect of Homogenisation on Milkfat in Serum

The effectiveness of homogenisation of milkfat in serum is mainly determined by four factors; milkfat content, temperature of feed, homogenising pressure, and the type of homogeniser valve used. Most often, homogenisation is carried out in two successive phases using two valves; the clusters of fat globules formed after the milk has passed through the first valve are broken in the second one. Experimental results (Walstra and Jenness, 1984) indicate that the greater the degree of homogenisation (by repeated passages/passes), the greater would be the absorbance with formation of smaller fat globules which were highly polydisperse initially and less so after repeated homogenisation.

1.3.2 Determination of the Effectiveness of Emulsifiers

The adsorption of proteins to surface of oil droplets appears to be a two step process (Walstra and Jenness, 1984); firstly, the initial adsorption is diffusion controlled resulting in a linear decrease of the surface tension with time, and secondly, after the formation of a protein film, a penetration-controlled adsorption takes place depending on the availability of new surface areas.

In cases where oil contains surface-active lipids, which tend to adsorb more readily than proteins, this impedes the degree of protein adsorption (Darling el at, 1987).

In light scattering behaviour, an oil-in-water emulsion may be regarded as a collection of perfect, dielectric spheres, that fulfil the Mie Theory. Kerker (1987), indicates that there is a simple relationship between the turbidity and the interfacial area of an emulsion provided that certain conditions are met. Walstra (1965) made use of turbidity measurements using a spectrophotometer to study milkfat globules and colloidal dispersions.

An Emulsifying Index (EAI) was defined (Pearce and Kinsella, 1978; Haque and Kinsella, 1989) :-

EAI = 2 x T/ϕ x C, where C is the weight of protein per unit volume of aqueous phase before the emulsion is formed, turbidity (T) and volume fraction of dispersed phase (ϕ). And T = 2.303 x A/l, where A is the observed absorbance and l is the pathlength of the cuvette.

1.3.3 Rose-Gottlieb Method of Fat Determination

The fat content of a milk sample is gravimetrically determined by extraction of the fat from an ammonia-alcohol solution with diethyl ether and petroleum ether, evaporation of the solvent and finally weighing the residue. The function of the ammonia is to dissolve the protein of the milk while the ethyl alcohol breaks the fat-emulsion and the fat-protein complex. The petroleum ether is used to decrease the solubility of water, lactose and alcohol in the diethyl ether.

1.3.4 Experimental Procedures

Liquid anhydrous milkfat (AMF) and water or emulsifier in serum were mixed together, heated to a temperature of 55°C, fed to the homogeniser with both Stage I and II valves fully opened i.e, minimum pressure, to obtain a partially emulsified solution. The homogeniser valves are then preset to the required pressures before the solution was introduced to the homogeniser and sufficient sample collected in a chilled sample bottle containing chilled water to give a 1:10 mix of sample:water and placed on ice. These samples were further diluted with chilled water to 1:200 before analysis using the Malvern Particle Analyzer[(a)] was carried out.

The Malvern receptacle was cleaned thoroughly between tests with acetone solution to prevent the build up of a thin layer of fat which would cause incorrect readings.

1.4 Results and Discussion

1.4.1 Effects of Homogeniser Pressures on Fat Spherulites Size Distribution

In the following experiments, no emulsifier was used to mix with the liquid milkfat.

Composition of mixture for homogenisation :

Fat Content : 11%

Water : 89%

Sample : diluted and chilled prior to particle size analysis

Stage I homogeniser pressure varies while Stage II valve was maintained fully opened throughout the experiment.

Table 1 Effects of Homogeniser Pressure on Fat Spherulites Size Distribution.

Homogeniser x 10^3 kPas	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	S.S.A
6.89	2.90	4.70	1.60	3.00	2.30	2.7747
10.34	2.20	3.80	1.20	2.30	2.00	3.4054
17.23	2.10	4.60	1.10	2.90	2.00	3.5144
20.68	2.00	3.80	1.10	2.30	1.90	3.8741

D(v,y) - gives particle size diameter in microns below which y% (by volume) of the particles lie.

D(3,2) - refers to surface volume mean diameter (Sauter Mean). The average particle size is based on the specific surface per unit volume.

D(4,3) - refers to surface weight mean diameter (DeBroucker Mean). The average particle size is based on the unit weight of the particles.

S.S.A - refers to Specific Surface Area. It is the area of the particle size distribution of the sample divided by the total volume of the particles in the sample measured, given in square meters per cubic centimetre.

1.4.2 Determination of the Effectiveness of Emulsifiers

Four types of emulsifiers were used, and their relative effectiveness to produce a stable fat emulsion was measured by means of a simple relationship between the turbidity and the interfacial area of emulsion. The turbidity of the emulsion was determined by using a spectrophotometer.

5% of melted AMF was mixed with aqueous emulsifier solution, heated to 60°C, and homogenised at a Stage I and II pressures of $6.89 \times 10^3/1.38 \times 10^3$ kPas respectively. Aliquots of the emulsion were diluted serially with water and a buffer solution of 0.1% sodium sulfate/Dodecyle sodium sulfate giving a final dilution of 1000 times. The absorbance of the diluted emulsion was then determined in a 1cm pathlength cuvette at a wavelength of 550nm and a slit of 1nm in a spectrophotometer(Varian Series 634).

Table 2 Effectiveness of Emulsifiers

Emulsifier Concentration %	Absorbance			
	Sodium Caseinate	Lecithin	Whey Protein Conc	Buttermilk Powder
0.10	0.030	0.031	0.065	0.047
0.25	0.032	0.033	0.067	0.060
0.50	0.037	0.039	0.069	0.088
1.00	0.039	0.042	0.070	0.090
2.00	0.041	0.059	0.077	0.091
5.00	0.048	0.103	0.086	0.100
10.00	0.060	0.183	0.114	-

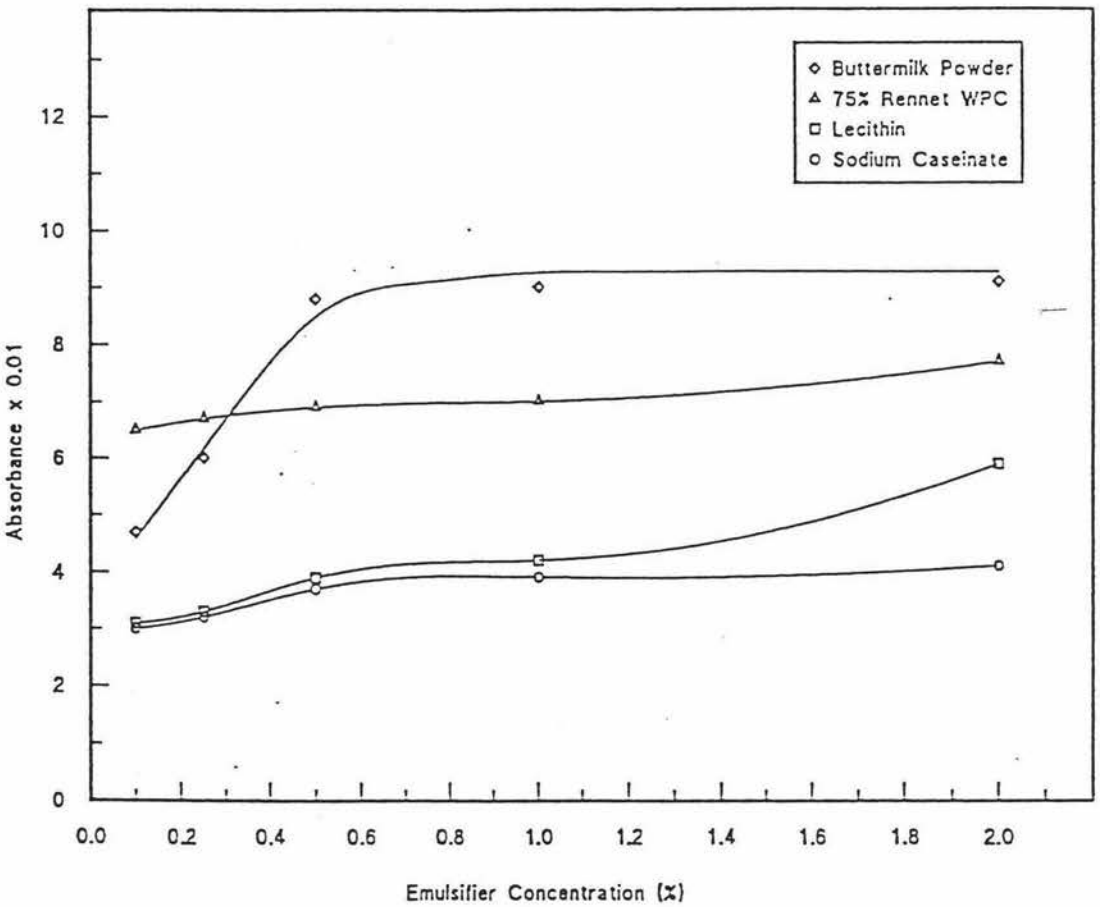


Fig 6 Effects of Emulsifier Concentration Versus Absorbances

The results in Fig 6 indicated that buttermilk powder has a better overall emulsifying properties than either lecithin or sodium caseinate at concentration of more than 0.3%. However, at concentration of less than 0.3%, the properties of 75% rennet whey protein concentrate was better than that of buttermilk powder. It has been well established that butter powder up of 80% fat content has been produced using approximately 9-10% sodium caseinate to milkfat (Hansen, 1963). This was in line with the results of the experimentation.

1.4.3 Effect of Sodium Caseinate on Fat Spherulites Size Distribution in Relation to Homogenisation Pressure at 1% Milkfat Content

In the following experiment, sodium caseinate acting as an emulsifier, was used to mix with the liquid milkfat prior to homogenisation.

Composition of mixture for homogenisation :

Fat Content : 1%

Water : 99%

Emulsifier : sodium caseinate at 0.3% concentration in water

Sample : diluted and chilled prior to particle size analysis

Stage I homogeniser pressure was varied while Stage II was maintained at 1.38 x 10^3 kPas throughout the experiment.

Table 3 Effect of Sodium Caseinate on Fat Spherulites Size Distribution in Relation to Homogenisation Pressure at Low Fat Content(1%).

Homogeniser x 10^3 kPas	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	S.S.A
0	16.40	63.30	5.70	24.50	12.70	0.5361
3.45	3.60	5.20	2.10	3.60	3.20	1.9770
5.17	3.00	4.40	1.80	3.00	2.50	2.5681
6.89	2.50	3.50	0.60	2.40	1.60	3.8446
8.62	2.30	3.50	1.30	2.40	2.10	3.1731

1.4.4 Effect of Sodium Caseinate on Fat Spherulites Size Distribution in Relation to Homogenisation Pressure at 3.75% Milkfat Content

Composition of mixture for homogenisation :

Fat Content : 3.75%

Water : 96.25%

Emulsifier : sodium caseinate at 0.3% concentration in water

Sample : diluted and chilled prior to particle size analysis

Stage I homogeniser pressure was varied while Stage II was maintained at 3.45 x 10^3 kPas throughout the experiment.

Table 4 Effect of Sodium Caseinate on Fat Spherulites Size Distribution in Relation to Homogenisation Pressure. 3.75% milkfat.

Homogeniser x 10^3 kPas	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	S.S.A
10.34*	0.90	1.80	0.50	1.00	0.80	7.7081
10.34**	1.70	2.80	0.80	1.80	1.50	4.7881
17.23*	1.70	2.90	0.80	1.80	1.50	4.8451
17.23**	2.00	3.00	1.10	2.00	1.80	3.9025

* only water used in the mixture

** sodium caseinate at 0.3% concentration in water

1.4.5 Effect of Sodium Caseinate on Fat Spherulites Size Distribution in Relation to Homogenisation Pressure at 9% Milkfat Content

Composition of mixture for homogenisation :

Fat Content : 9%

Water : 91%

Emulsifier : sodium caseinate at 0.3% concentration in water

Sample : diluted and chilled prior to particle size analysis

Stage I homogeniser pressure was varied while Stage II was maintained at 3.45 x 10^3 kPas throughout the experiment.

Table 5 Effect of Sodium Caseinate on Fat Spherulites Size Distribution in Relation to Homogenisation Pressure. 9.0% milkfat.

Homogeniser x 10^3 kPas	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	S.S.A
10.34*	1.70	3.00	0.80	1.80	1.50	4.7908
10.34**	2.60	3.40	0.60	2.30	1.60	3.9481
17.23*	1.50	2.60	0.70	1.60	1.30	5.5701
17.23**	1.60	2.70	0.80	1.70	1.40	5.0196

* only water used in the mixture

** sodium caseinate at 0.3% concentration in water

1.4.6 Effect of Sodium Caseinate on Fat Spherulites Size Distribution in Relation to a Series of Homogenisation Pressures

Composition of mixture for homogenisation :

Fat Content : 5%

Water : 95%

Emulsifier : sodium caseinate at 0.3% concentration in water

Sample : diluted and chilled prior to particle size analysis

Stage I homogeniser pressure varies while Stage II was maintained 1.38 x 10^3 kPas throughout the experiment.

Table 6 Effect of Sodium Caseinate on Fat Spherulites Size Distribution in Relation to a series of Homogenisation Pressures

Homogeniser x10^3 kPas	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	S.S.A
0	19.20	29.30	6.00	18.40	12.50	0.5621
0.69	10.40	16.90	3.10	10.10	7.10	0.9135
1.38	5.80	9.90	2.20	5.90	4.40	1.5431
2.07	4.30	7.40	2.10	4.50	3.70	1.7820
2.76	3.90	6.40	1.90	4.00	3.20	2.0435
3.45	3.60	5.20	2.00	3.60	3.20	2.0746
5.17	3.20	4.90	1.90	3.30	2.90	2.3111
6.89	2.90	4.00	0.70	2.80	2.30	2.7792
8.62	2.50	3.80	1.50	2.60	2.30	2.8411

1.4.7 Summary of Fat Spherulites Sizes for different Homogeniser Pressures

Table 7 shows a summary of fat spherulites sizes for different homogeniser pressures.

Table 7 Summary of Fat Spherulites Sizes for different Homogeniser Pressures.

Homogeniser x 10^3 kPas	From Table	% Under 1.2μm	Size(μm)100% under
6.89	1	9.10	10.50
10.34	1	10.30	10.50
17.23	1	13.20	33.70
20.68	1	15.90	10.50
0	3	0.20	118.40
3.45	3	1.80	10.50
5.17	3	6.70	8.20
6.89	3	21.70	5.00
8.62	3	7.60	8.20
10.34	4	26.20	6.40
10.34	4	23.30	5.00
17.23	4	36.00	6.40
17.23	4	28.60	6.40
10.34	5	67.20	2.40
10.34	5	25.80	6.40
17.23	5	26.70	6.40
17.23	5	14.10	6.40
0	6	0.50	118.40
0.69	6	0.30	33.70
1.38	6	2.50	17.70
2.07	6	2.10	13.60
2.76	6	3.80	13.60
3.45	6	3.90	10.50
5.17	6	4.50	8.20
6.89	6	8.70	6.40
8.62	6	4.80	8.20

The homogenisation pressures of stage I and II ranges from 0-20.68 x 10^3 kPas and 0-3.45 x 10^3 kPas respectively and the percentage of milkfat content to water were 1-10%. Sodium caseinate was used as an emulsifier and the concentration was 0.3%. In cases where emulsifier was used, there was no chilling of the samples collected.

It is important to note that the Malvern Particle Analyzer M6.10[a] results do not represent the complete analysis of the particle size distribution. Particles which are less than 1μm and more 1000μm are not included in the cumulative frequency distribution analysis. The main concern was the inability of the Malvern to detect and determine the lower range particle sizes. Table 7 gave an indication of the percentage of particle size under 1.2μm. The percentage is proportional to the homogenising pressure. However, analysis of the samples using the spectroturbidimetric method of the fat globule size determination indicated that the average particle sizes were in the 1μm or less range.

It was found that a more consistent results could be obtained if the feed, after being partially emulsified with a high speed stirrer, was passed through the homogeniser at zero pressure (or lowest homogenisation pressure setting) before homogenising at the required pressure.

Based on the results, it can be seen that the homogenisation pressure is inversely proportional to the particle size and directly proportional to percentage of fat content in the feed. This is further confirmed by the specific surface area of the particle where an increase in homogenisation pressure gave a corresponding increase in the specific surface area. The average particle size ranges from 19.20μm at 0 kPas to less 1μm or less at 17.23 x 10^3 - 20.68 x 10^3 kPas.

In Table 4 and 5, it was noted that at high homogenisation pressure (i.e, 17.23 x 10^3 kPas) the decrease in feed fat content gave a corresponding increase in the average particle size. This could be due to coalescence of the fat globules and further confirmed by the fact that a time delay in analysing the samples invariably causes an increase in the particle sizes.

1.4.8 Study to Determine the Hard Fraction Milkfat Spherulitic Sizes at Low Homogenisation Pressures

In the course of the experiments, it became clear that the important aspect to complement the overall objective was in the area of sizes of fat spherulites at low homogenisation pressures.

Milkfat hard fraction from Bay Milk Products(BMP) was used for the experiments. Feed compositions were 5%, 10% and 20% milkfat, together with equivalent concentrations of whey protein concentrate(WPC) i.e. 0.5% WPC for every 5% of fat. Homogenising pressures were varied from 0 to 3.45 x 10^3 kPas. Where Stage I pressure was 0 kPas Stage II was maintained at 0 kPas; all other homogenising pressures stage II was set at 0.34 x 10^3 kPas.

Milkfat and water containing the required concentration of dissolved emulsifier were heated separately to a temperature of approximately 60°C and mixed.

The feed was then passed through the homogeniser at 0 kPas for both stage I and II to achieve a good oil:water emulsion. The homogeniser pressure was slowly increased to the required pressure. Stage II pressure regulating valve was set at 0.34 x 10^3 kPas (for Stage I pressure above 0.69 x 10^3 kPas) throughout the experiment. Homogenised emulsion from the outlet pipe was drained off and samples collected.

Table 8 Particle Size Analysis Containing 5% Fat.

Press x 10^3 kPas	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	S.S.A
0	11.1	17.4	4.0	11.1	8.4	0.8234
0.69	6.4	9.4	2.5	6.2	5.1	1.3066
1.38	5.5	7.8	2.2	5.3	4.4	1.5220
2.07	4.3	6.7	2.0	4.3	3.6	1.8526
3.45	2.7	3.8	0.7	2.6	1.9	3.3708

Table 9 Particle Size Analysis Containing 10% Fat.

Press x 10^3 kPas	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	S.S.A
0	19.7	31.6	8.0	20.0	15.5	0.4578
0.69	7.2	10.6	2.8	7.0	5.7	1.1435
1.38	6.0	8.6	2.4	5.8	4.9	1.3433
2.07	4.5	7.1	2.0	4.5	3.7	1.7893
3.45	3.3	5.0	1.9	3.3	2.9	2.2198

Table 10 Particle Size Analysis Containing 20% Fat.

Press x 10^3 kPas	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	S.S.A
0	16.3	23.3	7.2	16.0	13.2	0.5159
0.69	6.5	9.6	2.5	6.3	5.1	1.2662
1.38	5.4	7.8	2.1	5.2	4.2	1.5697
2.07	4.2	6.5	2.0	4.2	3.5	1.8688
3.45	3.1	4.8	1.8	3.1	2.6	2.5354

Table 11 Summary of Percentage of Particle Sizes Less than 1.2μm.

Press x 10^3 kPas	5 %Fat	10 %Fat	20 %Fat
0	1.0	0.4	0.0
0.69	1.5	0.9	1.5
1.38	1.7	1.0	2.0
2.07	2.7	2.7	2.3
3.45	6.4	3.1	6.8

Fig 7a to 7c shows the relationship of the fat spherulite size distribution to homogeniser pressures at varying percentage of fat content.

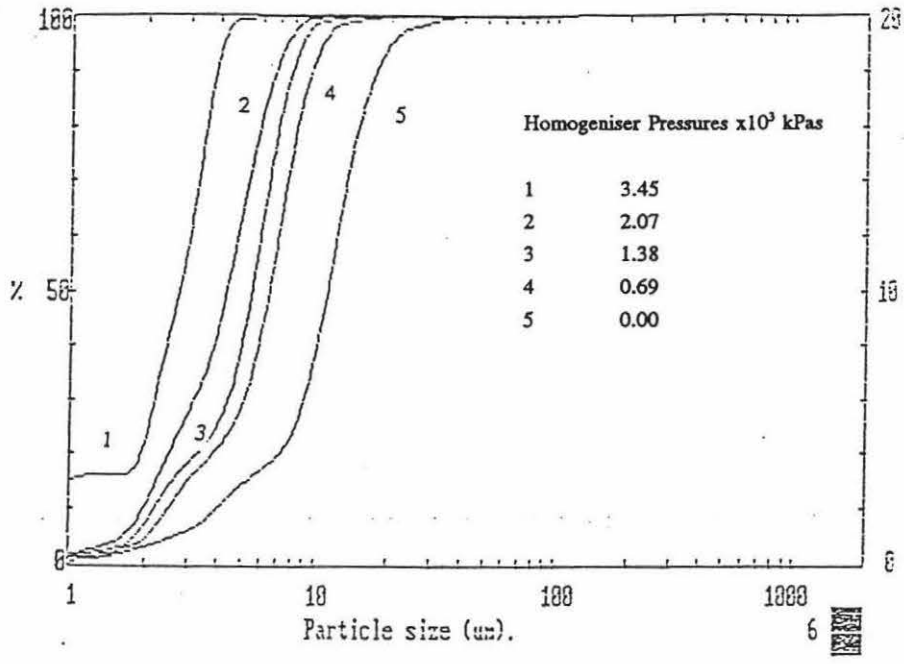


Fig 7a At 5% fat content

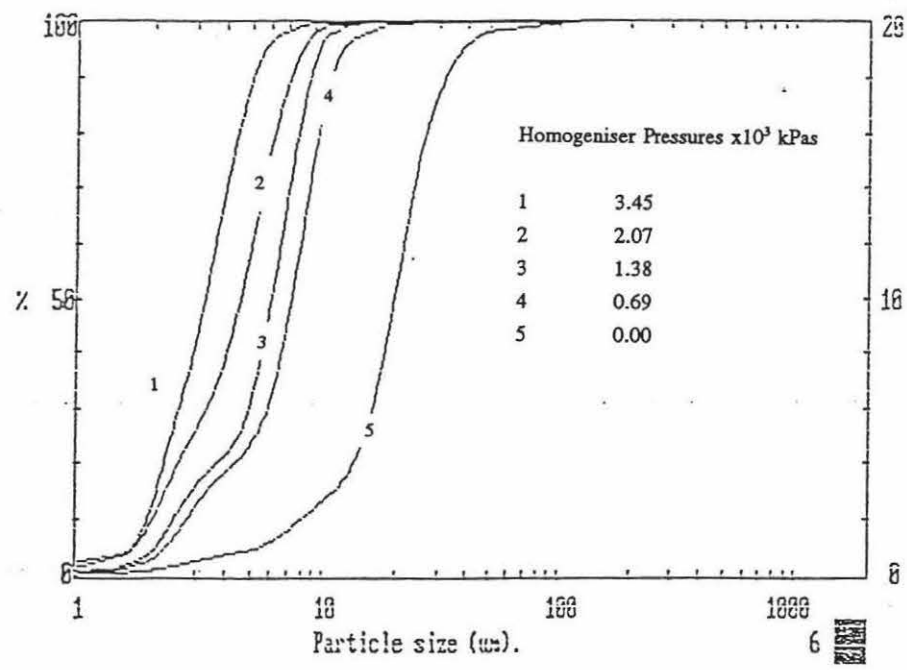


Fig 7b At 10% fat content

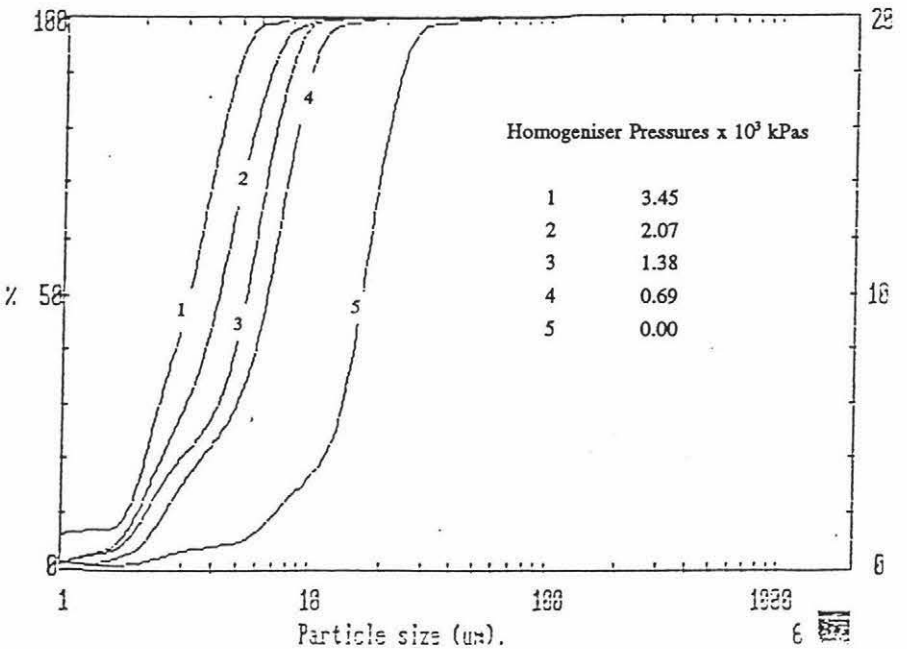


Fig 7c At 20% fat content

As expected, the fat particle sizes were inversely proportional to the homogenisation pressure. However, the fat particle sizes of 20% fat composition in the feed is smaller over the ranges than that of 10% fat in the feed. The high fat thus being more viscous coupled with the low homogenising pressure may have contributed to these results. The fluctuation of the homogeniser stage I pressure was quite significant (300-500 kPas) and it was difficult maintaining the required pressures. However, it would be unusual if all the pressure errors were in the same direction, and the reason for the smaller sizes is at present unknown.

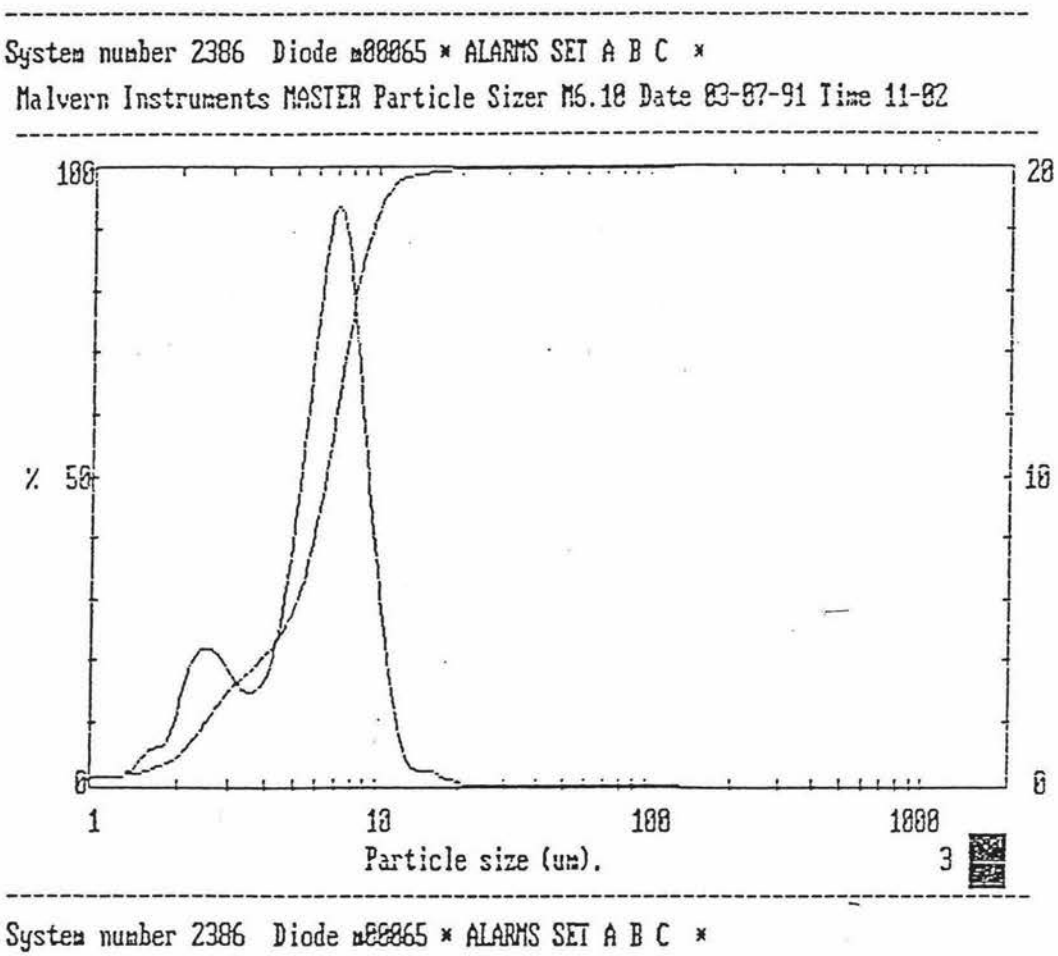


Fig 7d Summary of Particle Size Distribution

Fig 7d shows a summary of the particle size distribution of percentage under and the relative percentage frequency distribution of the particle sizes. They were generally having a bell shaped distribution with two peaks in all cases. The first peak particle size ranges from 1.5-6.0μm and the second peak ranges from 6.0-20.0μm.

1.4.9 Using an updated and higher version of Malvern MasterSizer E Ver.1.1[b] Particle Analyzer to analyze the fat spherulites of size and concentration in the range of 0.1μm.

The earlier part of the results were based on the Malvern Particle Analyzer M6.10[a] which has limited capability of detecting fat globule sizes of less than 1μm. Therefore, the interpretation of the results could not be conclusive, particularly where samples were taken at high homogenisation pressures; the concentration of smaller fat globules sizes were more prominent.

Malvern MasterSizer E Ver. 1.1[b] is capable of analysing particle of 0.1μm and upwards. As this part of the work was carried out at a much later stage, it was thought preferable to predetermined the regimes that would correlate with the main thrust of the research objectives. Also the particle analyser will provide results that will account for the distribution of fat globules which are less than 1μm. Based on the work done so far, it was apparent that low homogenisation pressures would be used, and therefore the pressure selected was from 0.35-3.45 x 10^3 kPas.

The three main parameters for investigating stable fat spherulites sizes and distribution in emulsion were the percentage fat content, percentage of buttermilk powder in serum and the homogenisation pressures.

The regime for fat content was 5-50%; buttermilk in serum from 0.5-1.0% for every 5% of fat; and, homogenisation pressures from 0.34/3.45 x 10^3 kPas.

1.4.9.1 Effects of Fat Spherulites Size Distribution in Relation to Fat content, Homogenisation Pressure and Emulsifier.

Composition of mixture for homogenisation :

Fat Content	: 5 - 50%
Water	: 95 - 50%
Emulsifier	: buttermilk at 0.5-1.0% concentration per 5% fat content
Sample	: diluted prior to particle size analysis

Stage I homogeniser pressure was varied while Stage II was maintained with valve fully opened throughout the experiment.

Table 12 Fat Globule Size Distribution of Homogenised Milkfat

Homogr. 10^3 kPas	Fat Cont %	D(v,0.5)	D(v,0.9)	D(v,0.1)	D(4,3)	D(3,2)	Results % < 1μm	S.S.A*
0.35	5	8.17	14.71	1.11	7.89	3.05	8.39	1.9681
0.69	5	6.10	12.65	1.00	6.42	2.63	10.02	2.2815
2.07	5	7.39	21.01	1.02	9.36	2.84	9.66	2.1097
3.45	5	14.11	44.17	1.11	18.79	3.42	8.62	1.7539
0.35	10	8.67	15.92	1.08	8.53	2.99	8.96	2.0037
0.69	10	6.20	11.98	0.89	6.29	2.40	11.91	2.4962
2.07	10	9.11	15.59	1.09	9.07	2.43	9.77	2.4731
2.07**	10	3.72	7.88	0.42	3.99	1.26	21.51	4.7799
3.45	10	8.39	20.45	0.58	9.84	1.94	12.81	3.0895
3.45**	10	4.03	8.69	0.40	4.40	1.28	20.26	4.7043
0.35	40	10.86	18.87	1.20	10.61	3.52	7.91	1.7039
0.69	40	8.34	18.80	1.04	9.31	3.05	9.47	1.9693
2.07	40	4.36	12.89	0.95	5.83	2.27	10.80	2.6423
2.07**	40	5.58	1.62	0.48	6.62	1.63	13.90	3.6840
3.45	40	5.18	17.16	0.95	7.41	2.22	10.57	2.7013
3.45**	40	9.24	19.55	0.64	10.18	2.07	10.52	2.9054
0.35	50	20.04	54.41	2.37	24.44	6.46	3.85	0.9290
0.69	50	9.91	26.19	1.21	12.21	3.67	7.35	1.6335
2.07	50	10.61	23.32	1.27	11.40	3.60	7.21	1.6681
3.45	50	9.90	20.07	1.25	10.40	3.31	7.85	1.8111

** - twice the concentration of emulsifier used i.e. 1% per 5% fat.

MALVERN MASTERSIZER E

Version 1.1 Thu, Sep 10, 1992 11:52AM

Homogenised Milkfat Emulsion at 5% fat and 50psi

Source: Analysed

Measured on: Wed, Sep 9, 1992 2:05PM

Presentation: (2NHD) 1.330, 1.456 + i 0.10000
Polydisperse model — Volume Result — Focus = 45 mm.

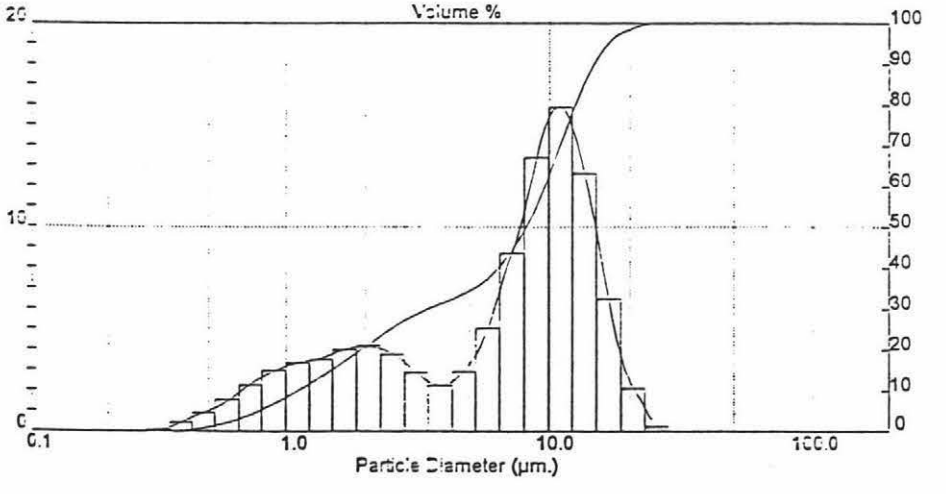
Residual = 0.234 %
d (0.5) = 8.17 µm
D [4, 3] = 7.89 µm
Sauter Mean (D[3,2]) = 3.05 µm
Specific Surface Area = 1.9681 sq. m. / gm

Concentration = 0.016 %
d (0.1) = 1.11 µm
Span = 1.68

Obscuration = 25.08 %
d (0.9) = 14.71 µm
Mode = 10.80 µm
Density = 1.00 gm. / c.c

Size (Lo) µm	Result In %	Size (Hi) µm	Result Below %
0.10	0.00	0.12	0.00
0.12	0.00	0.15	0.00
0.15	0.00	0.19	0.00
0.19	0.00	0.23	0.00
0.23	0.00	0.28	0.00
0.28	0.13	0.35	0.13
0.35	0.47	0.43	0.60
0.43	0.94	0.53	1.54
0.53	1.57	0.66	3.11
0.66	2.29	0.81	5.41
0.81	2.98	1.00	8.39
1.00	3.35	1.23	11.74
1.23	3.53	1.51	15.26
1.51	4.02	1.86	19.28
1.86	4.21	2.30	23.50
2.30	3.78	2.83	27.28

Size (Lo) µm	Result In %	Size (Hi) µm	Result Below %
2.83	2.89	3.49	30.17
3.49	2.26	4.30	32.43
4.30	2.95	5.29	35.37
5.29	5.05	6.52	40.42
6.52	8.72	8.04	49.14
8.04	13.42	9.91	62.57
9.91	15.90	12.21	78.47
12.21	12.64	15.04	91.10
15.04	6.50	18.54	97.60
18.54	2.13	22.84	99.74
22.84	0.26	28.15	100.00
28.15	0.00	34.69	100.00
34.69	0.00	42.75	100.00
42.75	0.00	52.68	100.00
52.68	0.00	64.92	100.00
64.92	0.00	80.00	100.00



Malvern Instruments Ltd.
Malvern, U.K.

MasterSizer E Ver. 1.1
Serial No.

p 1
10 Sep 92 11:53

Fig 8 Malvern Particle Sizer Data Sheet

Fig 8 shows a typical result that was obtained from the Malvern Sizer printout.

There was a general trend that fat globule sizes reduced with increase in homogenisation pressures. At low homogenisation pressures, the % of fat content in the feed can affect the proportion of the larger globule sizes i.e., d(v,0.5). There was significant reduction in globule sizes where the amount of emulsifier used was increased by two times, as in the case of homogenisation pressures at 2.07 and 3.45 x 10^3 kPas. At higher homogenisation pressures the proportion of larger sized globules tended to increase. Microscopic observations of the creams indicated agglomeration which may have been the main reason for these results, which were not in line with the expected trend.

1.5 Conclusion

The effects of the conditions and parameters of homogenising pressures, percentage milkfat content in serum, rapid cooling of homogenised cream and the use emulsifiers under which stable fat spherulites can be produced have been investigated.

It can be seen that the particle size is inversely proportional to the homogenisation pressure and directly proportional to percentage of fat content in the feed. This is further confirmed by the specific surface area of the particle where an increase in homogenisation pressure gave a corresponding increase in the specific surface area. The average particle size ranges from 19.20μm at 0 kPas to less 1μm or less at 17.23 x10^3 - 20.68 x 10^3 kPas. At high homogenisation pressure (>17.23 x 10^3 kPas) the decrease in feed fat content gave a corresponding increase in the average particle size. This could be due to the coalescence of the fat globules as a time delay in analysing the samples invariably show an increase in the particle sizes. In most cases, the particle size distribution generally have a bell shaped distribution with distinctive peaks.

At low homogenisation pressures (<3.45 x 10^3 kPas) the percentage of fat content in the feed can effect the proportion of the larger particle sizes i.e., at d(0,0.5). Increase in the amount of emulsifier used have a significant effect in reducing the fat particle sizes.

The main observations were,

a. The main process variables affecting fat particle size or spherulites distribution was that of the homogenisation pressures.

b. The fat content in the feed was a contributing factor.

c. The use of emulsifier in the feed prior to homogenisation helps to stabilise the milkfat spherulites in emulsion thus preventing coalescence and/or amalgamation.

d. Of the types of emulsifiers used, the results indicated that buttermilk powder has a better overall emulsifying properties. emulsifying properties of 75% rennet Whey Protein Concentrate(WPC) at 0.3-0.5% concentration for every 5% of fat in the feed composition gave the best results.

Chapter 4

Section 2 SPRAY DRYING OF CREAMED MILKFAT INTO MILKFAT SPHERULITES

2.1 Introduction

A spray drying process is commonly used in the dairy industry to manufacture dried milk products. Butter powder containing 80-90% of butterfat has been produced by a spray drying process using a mixture of milk constituents and other ingredients. High fat products in powder form are not new development (Fechner, 1936, Pyenson *el at* ,1946, Garrett, 1949). Butter powder has been in use mainly as a shortening in the bakery products. However, these products have not been favoured by commercial dairy manufacturers due to the fact that the powder contained emulsifiers. The proteins in emulsifiers are detrimental to baking performance (Hansen, 1963). The problems of manufacture, handling and storage of such powder in relation to its uses may have contributed to the lack of research work in this area.

It was envisaged that fat spherulites from the high melting fat fraction could be produced by spray drying process. Once these conditions for producing suitable spherulites had been determined, it was intended to produce spherulites of different sizes by varying the feed and operating conditions.

It was postulated that these fat spherulites containing a milk based protein as a stabilising agent could be produced in a fine spherical form and mixed with low melting point fat fractions, to make butter with the normal composition of milkfat retained, the butter would be spreadable when there was much less liquid fat present.

2.2 Objectives

The objectives were,

to determine the spray drying regimes and conditions under which fat spherulites from creamed milkfat fractions can be produced.

to determine the physical properties of the spherulites with respect to shape and size.

2.3 Literature Search

Butter powder containing 80-90% of fat content made from dairy fat by the spray drying process has been developed and patented by Fechner(1936). Similar work has been undertaken by Pyenson *el at*(1946), and Garrett(1949).

The process entails the homogenisation of milkfat with an emulsifying agent such as polar lipids and proteins, as well as peptizing additives like phosphates and citrates. Butter powder has been use mainly as a shortening in bakery products. Such high fat powder would have a fragile structure which poses problems not only during drying but also during subsequent handling and storage. Hansen, 1963 suggested that the fragile protein membrane coating the fat could easily be broken down by friction or mechanical shearing in the cyclones, thus causing amalgamation and stickiness of the powder. In all cases, the dry powder is cooled immediately and usually mixed with some form of free flowing or "anti-sticking" agent such as sodium-aluminium-silicate to improve handling. Improvement of the powder stability have been investigated by Townsend *el at*, 1968, Patel *el at*, 1987 with the use of emulsifying agents and the use of mechanical means by Hansen(1963) and Snow(1967).

2.4 Experimental

Equipment

2.4.1 Spray Dryer

This is an Anhydro Spray Dryer Type: Lab S1 with an evaporation capacity of 7.5 kg/hr of maximum inlet/outlet temperatures of 300°C and 90°C respectively. The liquid or slurry to be dried can either be fed through a variable high speed centrifugal disk atomizer or a nozzle atomizer with the use of compressed air for atomization (Fig 1). Air is sucked into the system by a suction fan and heated up by means of a series of electric radiator heaters. The droplets are subjected to a stream of hot air either counter currently (if nozzle atomizer was used) or concurrently (if used with centrifugal atomizer), water is removed by evaporation and powder produced. It is pneumatically conveyed to a cyclone where they are separated and removed. The exhaust air that carries the fines from the cyclone are either exhausted to the atmosphere or filtered off.

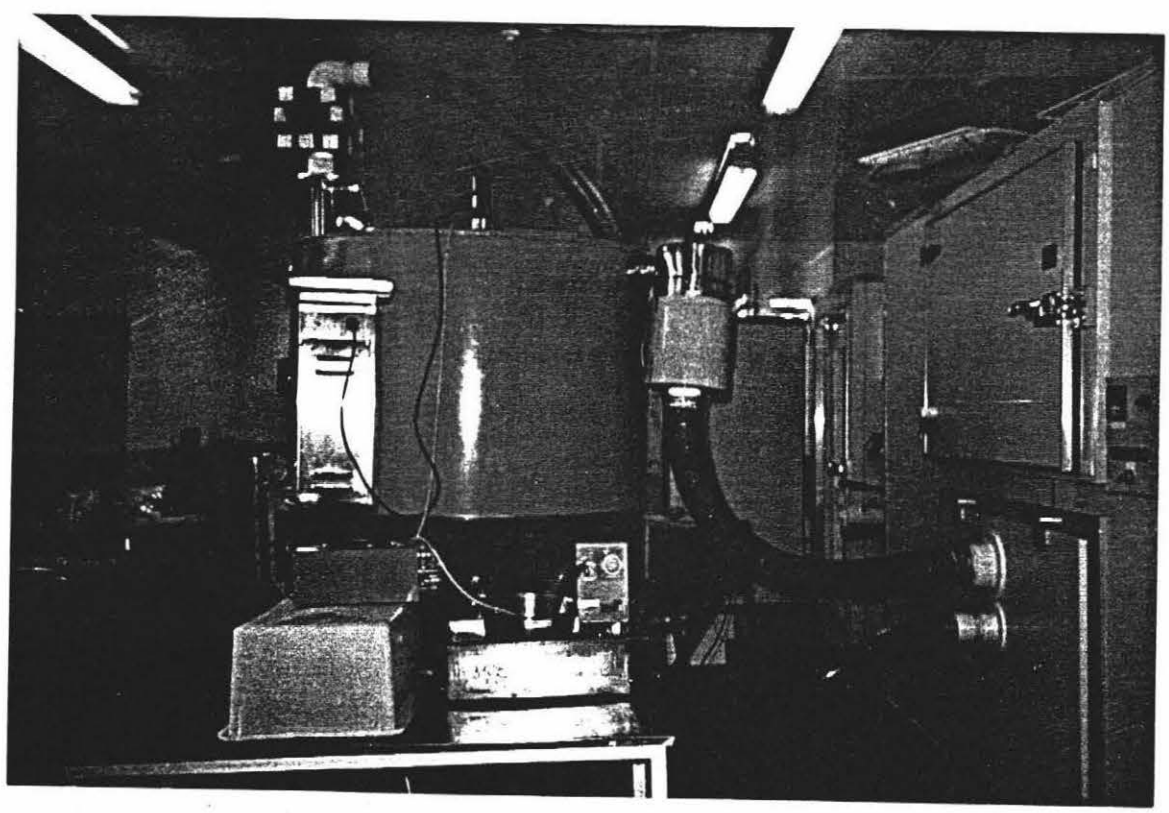


Fig 1 Photo of an Anhydro Spray Dryer Type: Lab S1

2.4.2 Homogeniser

This is Rannie Two Stage Homogeniser Model LAB Hyper: Type 12.50H with a rated capacity of 100l/hr capable of operating at a maximum pressure of 600 bars and inlet pressure of 3-10 bars. The feed rate is fixed and cannot be adjusted.

2.4.3 Bohlin Vor Rheometer

The Bohlin Rheometer System (Bohlin Rheologi AB, Lund, Sweden) was used to determine the regimes at which homogenised cream could be fed into the spray dryer. This is necessary as the feeding of the liquid or slurry into the spray dryer is by means of a Cole Palmer Masterflex Model No. 7521-25 metering pump. The pump cannot be operated with feeds of high viscosity and the viscosity of homogenised cream is largely dictated by the fat content, homogenisation pressure and the temperature.
The Bohlin measures directly the viscosity, shear rate and shear stress of a given sample at a predetermined constant temperature and time frame. The settings on the Bohlin were; measuring system - C 25, measure interval - 5 s, torque element - 4.06 g cm, strain delay time - 5 s, and sample temperatures set at 30 and 60°C.

2.4.4 Particle Analyzer

A Malvern Master Particle Sizer M6.10[a] was used in the determination of fat spherulite powder sizes. The Malvern used light scattering techniques to measure particle diameter of 1μm upwards and calculates particle size distribution on a volumetric basis.

2.5 Material

2.5.1 Milkfat

Fresh Frozen milkfat for Recombining (FFMR) constituting 99.9% milkfat and 0.1% moisture were obtained from the New Zealand Dairy Research Institute(NZDRI). Milkfat fractions of various composition and stages were obtained both from NZDRI and from

Bay Milk Products(BMP) Ltd in Te Puke, New Zealand. Refer Chapter 4, Section 1 - Figs 3-5 for details of NMR solid fat profile.

2.5.2 Milk Powder

Commercial exported grade Anchor Sweet Cream Buttermilk Powder and Skim Milk Powder from New Zealand Dairy Board(NZDB).

2.6 Methods

2.6.1 Determination of the Regimes which Homogenised Cream could be fed into the Spray Dryer

Initial trials on the spray dryer indicated that there were limitations on the extent at which the homogenised cream that could be fed by the metering pump and this is dependent of the viscosity. The viscosity of a homogenised cream is govern largely by the fat content, temperature and the homogenisation pressure. As a result, it was necessary to determine the regimes at which homogenised cream could be consistently fed into the spray dryer.

FFMR were mixed with buttermilk powder in serum. The mixture is heated to 55°C and then homogenised in the Rannie homogeniser. Homogenised samples were taken and Bohlin viscometry test carried out. A 2 x 3 x 2 (Milkfat x Buttermilk x Pressure) factorial experiment was planned and duplicates of viscometry test conducted.

Table 1 Experimental Plan

Expt No	Milkfat	Buttermilk Powder	Pressure
1	+	-	*
2	+	-	**
3	+	--	*
4	+	--	**
5	+	---	*
6	+	---	**
7	++	-	*
8	++	-	**
9	++	--	*
10	++	--	**
11	++	---	*
12	++	---	**

where,

+ - 10% Fat Content

++ - 40% Fat Content

- - 10% Buttermilk Powder

-- - 20% Buttermilk Powder

--- - 30% Buttermilk Powder

* - 3.45×10^3 kPas Homogeniser Pressure

** - 6.89×10^3 kPas Homogeniser Pressure

2.6.1.1 Results and Discussion

A total of twelve experiments were carried out and the results were as follows:

Table 2 Viscometry Results

Expt No	Viscosity (mPas) Shear Rate 100 30°C	Viscosity (mPas) Shear Rate 100 60°C
1	2.10	1.36
2	2.20	1.44
3	5.40	2.03
4	5.00	2.37
5	34.40	6.11
6	22.20	6.70
7	26.50	11.85
8	150.50	205.00
9	42.50	19.50
10	62.50	12.70
11	240.00	60.80
12	180.00	76.00

NZDRI - Food Tech Bohlin

BOHLIN RHEOMETER SYSTE
Viscometry test
1990-11-13 10:49:21

MILKFAT HF-BUTTERMILK POWDER-WATER SAM 2
1
Measuring system: C 25
Torque element: 4.06 g cm
Autozero delay time: 5 s
Strain delay time: 5 s

Sensitivity: 1 x
Integration time: 5 s

Temperature: Manual
No of measurements: 1

Measurement interval: 5 s

A:\
File name: sample2

Time s	Temp xC	no	Shear rate 1/s	Shear stress Pa	Viscosity Pas	Range %
6.7	30.0	45	4.616E+1	2.520E-1	5.460E-3	2.6
16.8	30.0	46	5.833E+1	3.191E-1	5.471E-3	3.3
26.2	30.1	47	7.335E+1	3.993E-1	5.444E-3	4.1
34.8	30.0	48	9.232E+1	5.020E-1	5.437E-3	5.1
43.0	30.0	49	1.163E+2	6.288E-1	5.407E-3	6.4
50.6	30.1	50	1.467E+2	7.942E-1	5.413E-3	8.1
58.1	30.1	51	1.846E+2	1.000E+0	5.418E-3	10.2
65.2	30.0	52	2.322E+2	1.263E+0	5.437E-3	12.9
72.2	30.0	53	2.923E+2	1.596E+0	5.458E-3	16.3
79.0	30.0	54	3.682E+2	2.016E+0	5.474E-3	20.6
85.7	30.0	55	4.634E+2	2.548E+0	5.499E-3	26.0
92.3	30.0	56	5.833E+2	3.225E+0	5.532E-3	32.9
98.7	30.0	57	7.343E+2	4.084E+0	5.562E-3	41.7
105.0	30.0	58	9.246E+2	5.227E+0	5.654E-3	53.3

Table 3 Bohlin Viscosmetry Results Showing Newtonian Fluid Behavior

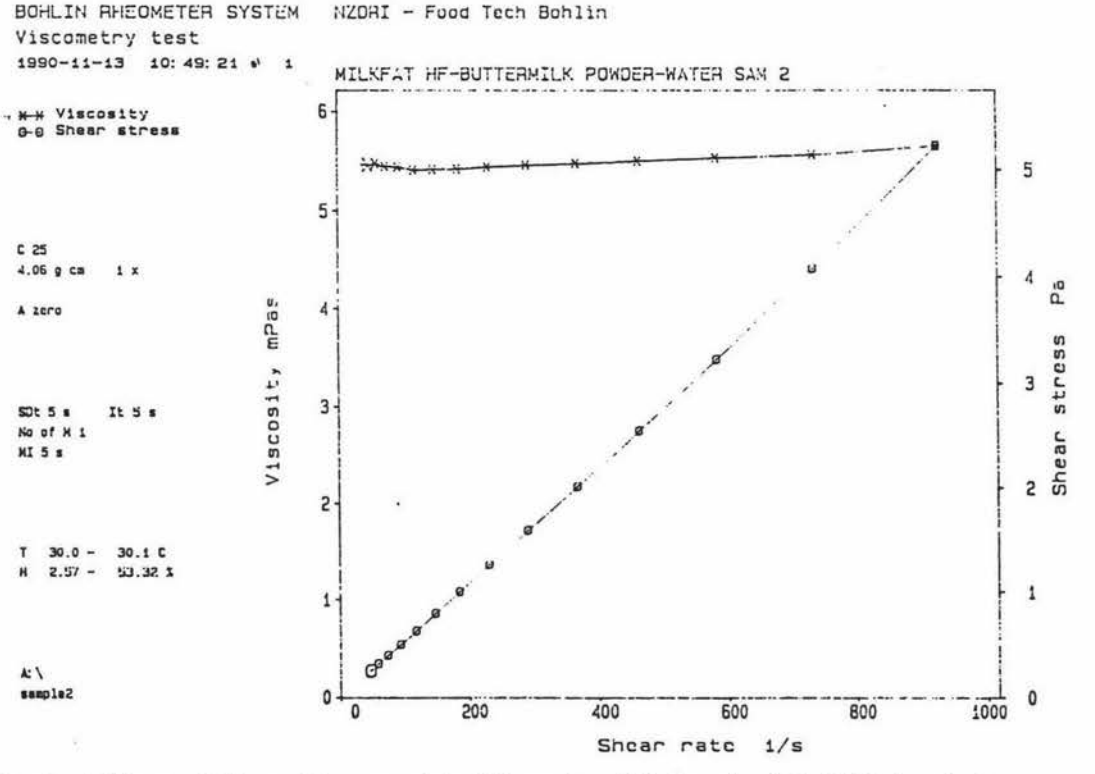


Fig 2 Effect of Shear Rate against Viscosity (Newtonian Fluid Behavior)

Measuring system: C 25
Torque element: 4.06 g cm
Autozero delay time: 5 s
Strain delay time: 5 s

Sensitivity: 1 x

Integration time: 5 s

Temperature: Manual
No of measurements: 1

Measurement interval: 5 s

A:\
File name: sample3

Time s	Temp xC	Smear rate no	Shear rate 1/s	Shear stress Pa	Viscosity Pas	Range %
6.7	30.0	32	2.322E+0	1.044E-1	4.494E-2	1.1
16.8	30.0	33	2.923E+0	1.234E-1	4.222E-2	1.3
26.1	30.0	34	3.682E+0	1.506E-1	4.090E-2	1.5
34.7	30.1	35	4.634E+0	2.002E-1	4.320E-2	2.0
42.9	30.0	36	5.833E+0	2.480E-1	4.251E-2	2.5
50.7	30.0	37	7.343E+0	2.912E-1	3.966E-2	3.0
58.2	30.0	38	9.246E+0	3.589E-1	3.881E-2	3.7
65.4	30.0	39	1.164E+1	4.266E-1	3.665E-2	4.4
72.3	30.0	40	1.465E+1	5.447E-1	3.717E-2	5.6
79.6	30.0	41	1.861E+1	6.805E-1	3.657E-2	6.9
86.3	30.0	42	2.326E+1	8.247E-1	3.546E-2	8.4
92.9	30.0	43	2.934E+1	1.012E+0	3.448E-2	10.3
99.3	30.0	44	3.686E+1	1.238E+0	3.362E-2	12.6
105.7	30.0	45	4.616E+1	1.509E+0	3.269E-2	15.4
111.9	30.0	46	5.833E+1	1.848E+0	3.168E-2	18.8
118.2	30.0	47	7.335E+1	2.259E+0	3.079E-2	23.0
124.5	30.1	48	9.232E+1	2.772E+0	3.000E-2	28.3
130.7	30.0	49	1.163E+2	3.407E+0	2.930E-2	34.8
137.0	30.0	50	1.467E+2	4.183E+0	2.851E-2	42.7
143.1	30.0	51	1.846E+2	5.149E+0	2.789E-2	52.5

Table 4 Bohlin Viscosmetry Results Showing Non-Newtonian Fluid Behavior

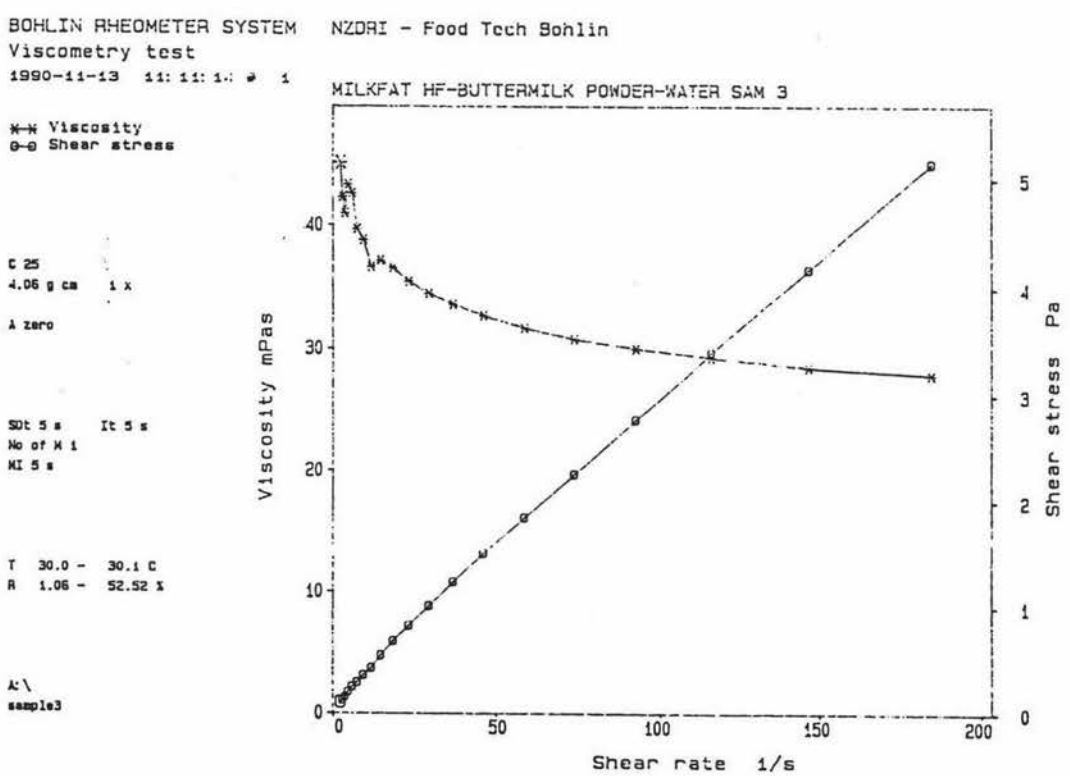


Fig 3 Effect of Shear Rate against Viscosity (Non-Newtonian Fluid Behavior)

The results of the experiment indicated that the apparent viscosities at 30°C shown inconsistencies particularly where the fat content was high. This could be attributed to the increase tendency of the cream to coalesce and amalgamate at low temperatures. Any increase in fat content, homogenisation pressure and a reduction in temperature would contribute to a proportional increase in the apparent viscosity of the emulsion. Fig 2 and 3 shows a typical Newtonian and Non-Newtonian fluid behaviour of the homogenised cream. Where the viscosity of the homogenised cream was low, the fluid behaviour was general newtonian but increasingly exhibits non-newtonian behaviour at higher viscosities (high fat and buttermilk concentration).

Regression analysis on the viscosities at 60°C of the homogenised cream against the Fat(F), Buttermilk Serum(B) and Homogenising Pressure(P) gave the following best fit Equation (1),

$$\log_e \eta = 0.53 - (0.0779 \times \% F) + (0.00067 \times P) + (0.00545 \times \% F \times \% B) + (0.000211 \times \% F \times P) - (0.000006 \times \% F \times B \times P) \quad \text{.....(1)}$$

where,

η is the apparent viscosity in mPas

The regression co-efficient was 92.1%.

Using the Masterflex metering pump, it was established that the optimum workable viscosity of the feed was between 20-25 mPas. A feeding system using a pressurised feed tank was used and did improve and make possible the feeding of higher viscosity range emulsion but the improvement was limited.

From these experiments, it was now possible to determine the limit conditions on the feed into the spray dryer.

2.6.2 Production of Fat Spherulites from Spray Drying

Having determined the conditions of the feed that could be used, a series of trials were than planned to determine the operating conditions of the spray dryer that would produced acceptable fat spherulites powder for use in buttermaking at a later stage. Earlier trials had indicated the limitations to the operating temperature ranges of the spray dryer, both inlet and outlet, which could be used without melting the fat and at the same time producing a high fat powder that has good flow properties. These trials had showed that the inlet and outlet temperature ranges of the spray dryer must be maintained at 120-130°C and 70-80°C respectively.

In these experiments, melted Fresh Frozen Milkfat for Recombining(FFMR) from NZDRI was mixed with 75% Rennet whey protein concentrate(WPC) in serum, heated to 60°C and homogenised at 6.89 x 10^3 kPas/1.38 x 10^3 kPas for stage I and II respectively to give a stable emulsion. It should be pointed out that the conditions needed to produce the butter powder applicable for this particular spray dryer (Anhydro Spray Dryer Type Lab S1) may not be appropriate for other spray dryers.

The spray dryer was turned on and allowed to achieve the desired steady state operating conditions. The homogenised cream was kept at a constant temperature of 60°C in a hot waterbath and pumped, using a Masterflex metering pump(setting at 0.8-0.9), into the centrifugal high speed disk atomizer. Atomised fat droplets were mixed with a cocurrent stream of hot air, moisture being removed from the droplets by evaporation, and the powder was separated and collected in a cyclone. The variables for each experiment were feed composition, speed of disk atomizer and feed temperatures. Half the total experiment were randomly replicated and the mean of the results used. The percentage of fat and moisture content were analyzed on the powder produced.

2.6.2.1 Results and Discussion

Table 5 shows the levels of experimental variables and the analysis of the powder collected from the drier's cyclone.

Table 5 Spray Dried Butter Powder from FFMR

Expt No	Feed Composition % Fat	% WPC	% Water	Feed % Solid	Disk Speed rpm	Feed Temp °C	Butter Powder % Fat	% Water
1	30.0	7.0	63.0	37.0	20000	50	77.50	1.43
2	30.0	7.0	63.0	37.0	20000	70	80.29	1.75
3	30.0	7.0	63.0	37.0	40000	50	78.12	1.44
4	30.0	7.0	63.0	37.0	30000	70	80.19	1.48
5	28.6	11.3	60.1	39.9	30000	50	72.69	0.91
6	28.6	11.3	60.1	39.9	20000	70	70.21	0.66
7	23.1	5.4	71.5	28.5	20000	50	75.80	1.15
8	23.1	5.4	71.5	28.5	20000	70	82.44	1.23
9	23.1	5.4	71.5	28.5	40000	50	75.68	1.12
10	23.1	5.4	71.5	28.5	30000	70	81.49	1.43
11	22.3	8.7	69.0	31.0	20000	50	71.58	0.87
12	22.3	8.7	69.0	31.0	30000	70	71.43	1.03
13	26.5	7.4	66.1	37.9	30000	50	81.04	1.01
14	26.5	7.4	66.1	37.9	20000	70	78.95	1.08
15	25.2	11.8	63.0	37.0	20000	50	66.23	1.53
16	25.2	11.8	63.0	37.0	20000	70	67.27	1.48
17	25.2	11.8	63.0	37.0	35000	50	66.32	1.36
18	25.2	11.8	63.0	37.0	30000	70	67.07	2.53
19	20.1	5.6	74.3	25.7	20000	50	77.41	1.52
20	20.1	5.6	74.3	25.7	30000	70	76.69	0.92
21	19.4	9.1	71.5	28.5	20000	50	66.11	1.77
22	19.4	9.1	71.5	28.5	20000	70	65.41	2.00
23	19.4	9.1	71.5	28.5	40000	50	71.22	0.78
24	19.4	9.1	71.5	28.5	30000	70	68.34	1.97

Table 6 Summary of Table 5

Expt No	Feed Ratio Fat:WPC	Mean % Fat Powder	Feed % Solid
1 to 4	4.29	79.02	37.0
5 to 6	2.54	72.45	39.9
7 to 10	4.29	78.85	28.5
11 to 12	2.54	71.50	31.0
13 to 14	3.60	80.00	37.9
15 to 18	2.14	66.72	37.0
19 to 20	3.60	77.05	25.7
21 to 24	2.14	67.77	28.5

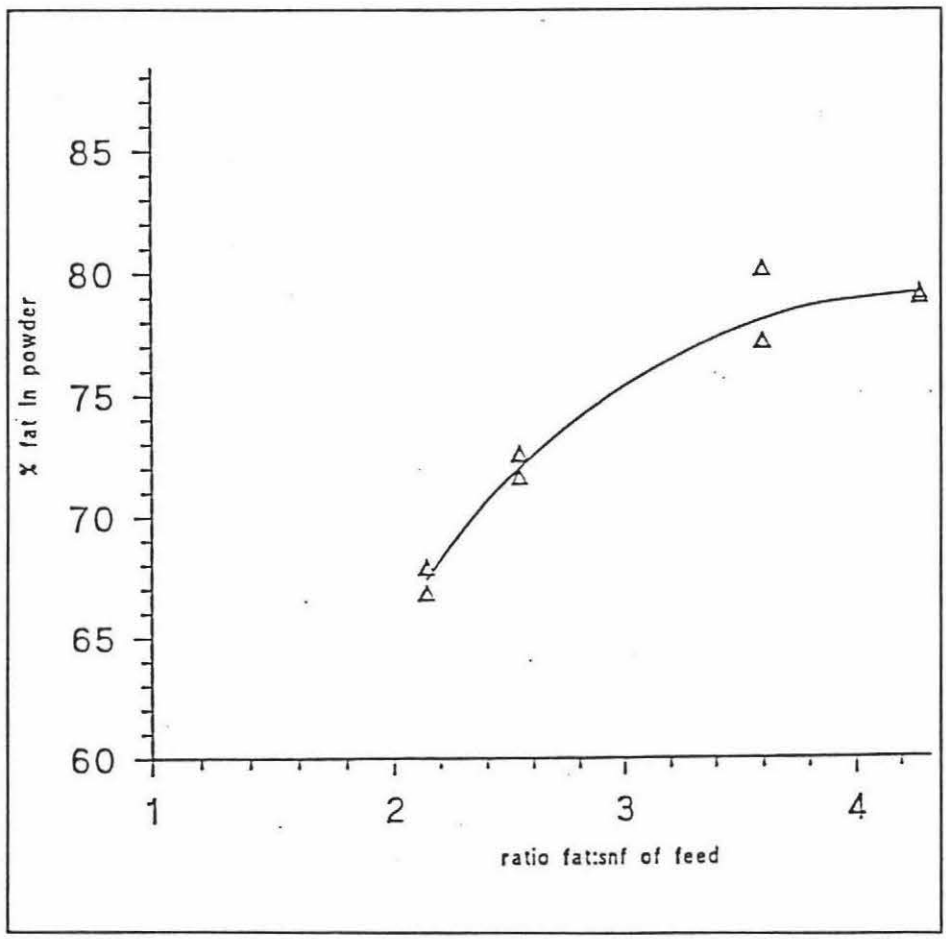


Fig 4 Spray Dried Powder - Ratio of FFMR:WPC against % FFMR in Powder

By plotting the ratio of FFMR:WPC in the feed to the percentage of fat in powder (Fig 4), it can be seen that an increase in the feed fat ratio will correspondingly increase the percentage of fat in powder. The optimum percentage of fat in powder that can be produced is in the region of 80%. Any further increase in fat content in the powder would have a detrimental effect on the physical properties of the powder in terms of free flowing and stickiness.

Based on the results of the previous experimentations, we have shown that the use of an emulsifier will help in the production of good stable butter powder and also reduces the level of solids-not-fat for coating of the fat crystals into spherulites. Table 7 gives a summary of the observed physical appearance of the butter powder produced in relation to the conditions and parameters used.

Table 7 Spray Dried Butter Powder with 75% Protein WPC

Expt No	% Fat	% WPC	% SNF	% Wat	Feed Rate	Amb. Temp	Inlet Temp	Outlet Temp	Observations of Powder
1	20	0.4	0.00	79.6	40	16	120	69	liquid
2	20	0.4	0.75	78.9	35	16	124	70	lumpy
3	20	0.4	1.25	78.5	35	17	123	73	lumpy
4	20	0.4	2.00	77.6	35	17	123	74	slightly lumpy
5	20	0.4	2.25	77.4	30	17	123	77	good powder

This work provided a guideline of the operating parameters necessary for producing a good milkfat powder. It can be concluded that, using the Anhydro Lab 1 spray dryer, fat powder of up to 80% in fat content and the remaining 18-19% solids-not-fat, with 1-2% moisture, was obtained. Rose-Gottlieb method of fat determination was used to analyze the fat content of the powder.

A higher percentage of fat was obtained in the powder with the addition of an emulsifying agent, and the powder was also of better quality. Up to 30% fat in the feed composition may be used, although the powder losses (with high fat content) sticking

on the walls of the dryer are increased. Correlation of the moisture in powder to that of the feed composition is not significant. Dryer disk speed is not highly correlated with the other parameters, although it is expected that the increase of the dryer disk speed will produce a finer powder.

At higher feed rate and higher inlet/outlet temperatures of 155°C and 74°C respectively, the powder appearance immediately ex-drier was powdery but became lumpy after a short period of time. It is possible that the high temperatures may have denatured the protein forming a hard layer on the surface of the fat globules and at the same time encapsulating the moisture in the fat globules. After cooling, breakdown of the hard surface layer results in the release of the entrapped moisture and this agglomerates the fat powder causing lumpiness.

By keeping the outlet temperature constant and varying the feed and inlet temperature, it is expected that the proportional increase in feed rate and inlet temperature will correspondingly increases powder moisture content and production of bigger particle sizes.

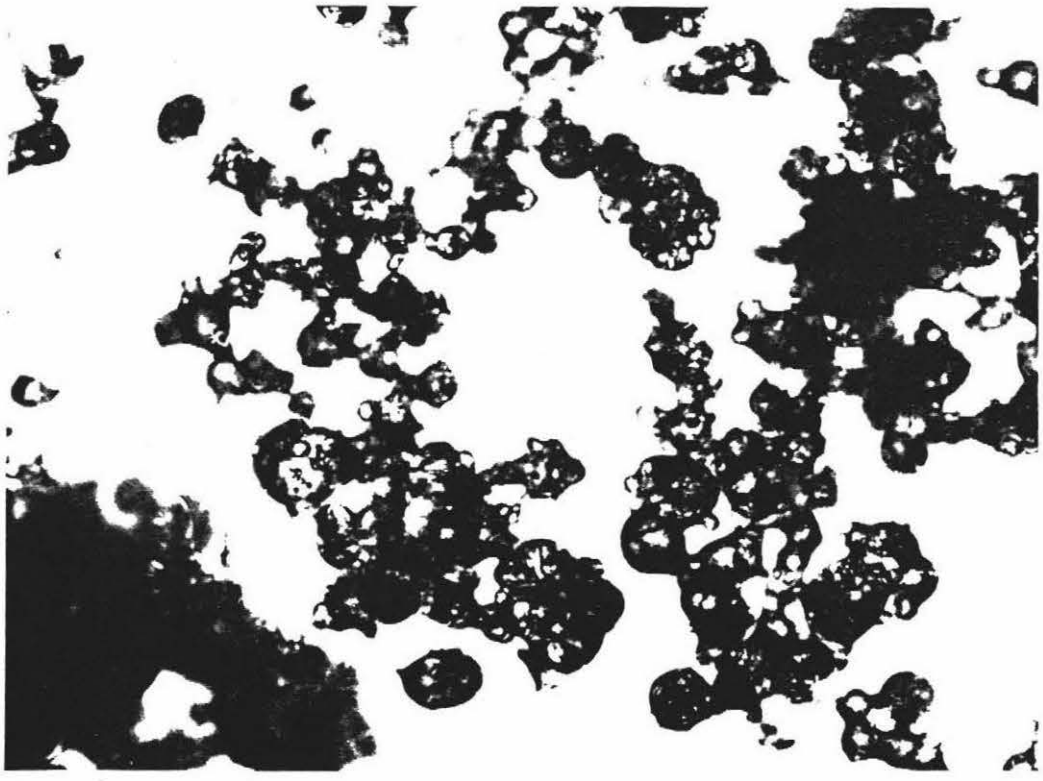


Fig 5 Photomicrograph of Buttermilk Powder

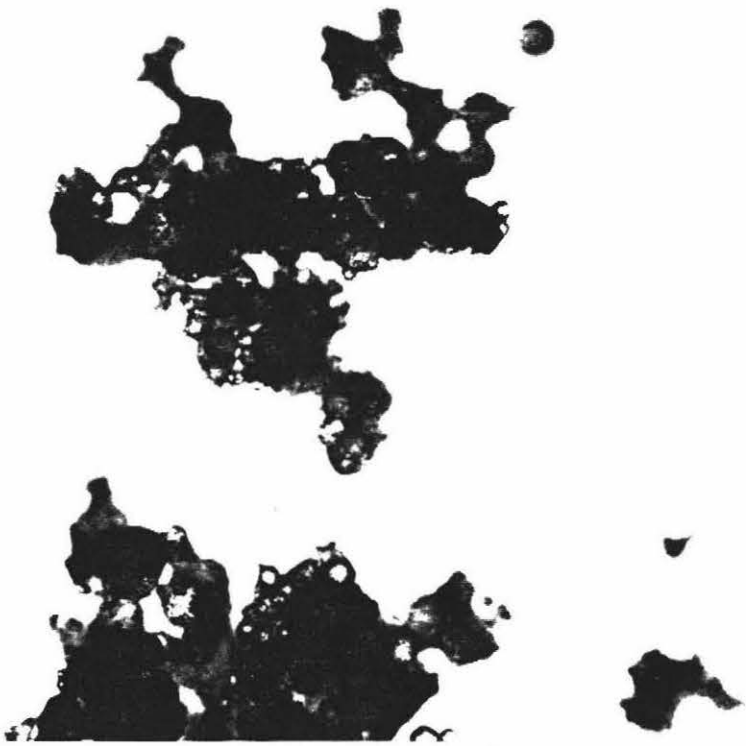


Fig 6 Photomicrograph of Fat Powder(Milkfat Hard Fraction)

Fig 7 Photomicrograph of Fat Powder(FFMR)

Fig 5-7 shows photomicrographs of commercial grade buttermilk powder, FFMR powder and butter powder from hard fraction. Buttermilk powder shows more stable physical structures and less agglomeration whereas in the case of FFMR and hard fraction powder there was more extensive destabilisation of the powder, as revealed through the presence of large irregular-shaped aggregates of fat or fused fat droplets. This is consistent with the observations by Patel *el at*, 1987. In stable powders, most of the fat appears to be intact inside the powder particle. With exposure to increase in temperature, it would appear that the FFMR powder has a higher tendency to agglomerate in comparison to that of the hard fraction powder. This was attributed to the higher melting point of the hard fraction. It can be concluded that physical integrity of the fat droplets in powder particles was mainly responsible for the tendency of powder particles to stick to the cyclone walls, the extent of damage of these droplets determine the extent of powder sticking in the dryer cyclone.

With a 20% fat and 2.25% buttermilk in serum, homogenised at 6.89 x 10^3 kPas to form an emulsion and spray dried at an inlet/outlet temperatures of 123°C/77°C, powder of about 80% fat with good physical properties was produced. Fig 8 shows the particle size of the butter powder. Fig 9a and 9b shows the size distribution of the butter powder.

System number 2386 Diode #00065 * ALARMS SET B D *

Malvern Instruments MASTER Particle Sizer M6.10 Date 20-06-91 Time 13-24

Size microns	% under	Size band microns		%	
564.0	100.0				Result source= Averaged
261.7	78.0	564.0	261.7	22.0	Record No. = 1
160.4	45.5	261.7	160.4	32.5	Focal length = 300 mm.
112.8	26.2	160.4	112.8	19.3	Experiment type pla
84.3	14.6	112.8	84.3	11.7	Volume distribution
64.6	8.9	84.3	64.6	5.7	Beam length = 22.0 mm.
50.2	6.2	64.6	50.2	2.6	Obscuration =0.1647
39.0	4.5	50.2	39.0	1.7	Volume Conc. = 0.0321 %
30.3	2.9	39.0	30.3	1.6	Log. Diff. =3.75
23.7	1.6	30.3	23.7	1.4	Model indp
18.5	1.0	23.7	18.5	0.6	
14.5	0.7	18.5	14.5	0.3	D(v,0.5) = 171.7 um
11.4	0.5	14.5	11.4	0.2	D(v,0.9) = 335.0 um
9.0	0.3	11.4	9.0	0.2	D(v,0.1) = 69.4 um
7.2	0.1	9.0	7.2	0.1	D(4,3) = 185.3 um
5.8	0.1	7.2	5.8	0.1	D(3,2) = 132.4 um
					Span = 1.5
					Spec. surf. area
					0.0521 sq.m./cc.

System number 2386 Diode #00065 * ALARMS SET B D *

Fig 8 Particle Size Distribution of Fat Spherulites Using Malvern Particle Analyser M6.10[a]

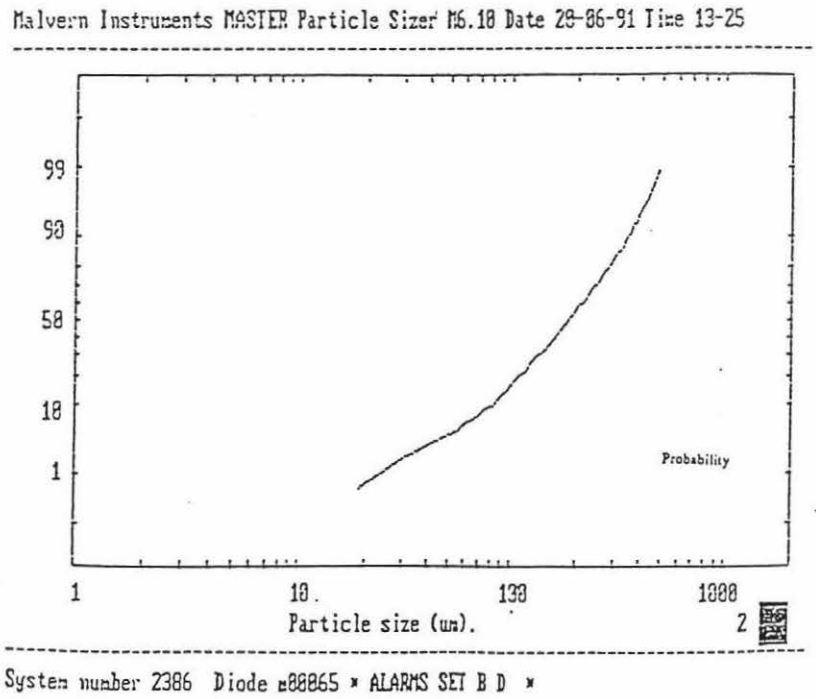


Fig 9a Particle Size Analysis of Fat Spherulites Percentage Probability

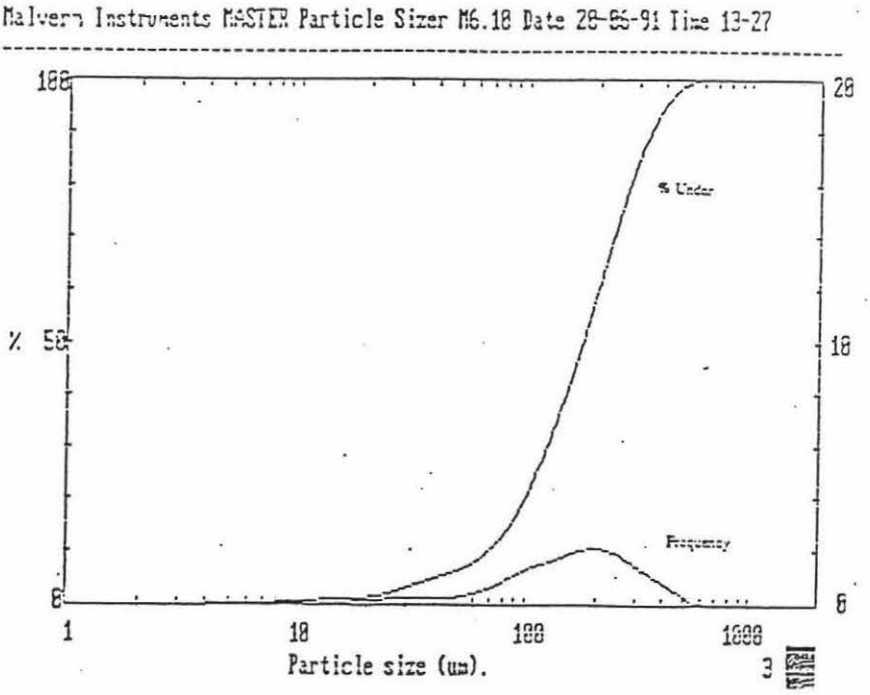


Fig 9b Particle Size Analysis of Fat Spherulites Percentage Distribution and Percentage Under

The particle size diameter below 50% (by volume) was 171.7 μm. Of the total, 22% of the particle diameters lie between 261.7 - 564μm. Finer powder will have to be produced and this could be achieved by having finer atomised fat droplets for drying. Between 5-10% of difference occurred in experimental replicates. Some attributions to the problems with the operation of the dryer were:

Spray dryer inlet temperature can vary from 120-130°C due to change in ambient temperature during operation.

Partial blockage of the pipe leading to the cyclone. As a result, there was an increase in the dryer inlet temperature and decrease in outlet temperature - i.e. higher moisture content in powder.

Cooling of powder produced is an important factor in determining the extent of powder aggregation. Cooling also prevents powder lumpiness caused by formation of lactose bridges, or by liquid fat, cementing the particles together.

By having fixed setting on metering feed pump the feed flowrate can vary due to variation in viscosity of feed composition.

There was an increase in powder moisture content at constant inlet temperature of 120°C, and when feed rate was increased, a corresponding lower outlet temperature. The powder became more lumpy as the feed rate was increased. Further increased in feed rate would increase proportionally the tendency of the "wet" powder to stick on the wall of the dryer. Samples of powder collected from the wall of the dryer do invariably have a lower moisture content. This is attributed to the longer retention time of the powder in the dryer.

2.7 Conclusion

The spray drying regimes and conditions under which fat spherulites from creamed milkfat fractions was produced using an Anhydro Spray Dryer Type: Lab S1 were

determined. The optimum percetage of fat in powder that can be produced was in the region of 80% and the remaining 18-19% solids-not-fat and 1-2% moisture. Any further increase in fat content would have a detrimental effect on the physical properties of the powder in terms of flowing and stickiness.

Up to 30% fat in the feed composition can be used. Any increase in fat, homogenisation pressure and a reduction in feed temperature would contribute to a proportional increase in the apparent viscosity of the emulsion. Using the Masterflex metering pump, it was established that the optimum workable viscosity of the feed would be in the region of 20-25 mPas. Feeding system incorporating a pressurised vessel did improved and made possible the use of higher viscosity emulsion but the improvement was marginal.

Comparison of photomicrographs show that buttermilk powder has a more stable physical structure and less aggromeration whereas that of spray dried FFMR and milkfat hard fraction show a more extensive destabilisation of the powder as indicated by the presence of large irregular-shaped aggregates of fat powder.

Fat powder of particle diameter 50% by volume in the range of 170 μm has been produced and this further reduction in particle size could be acheived by having finer atomised droplets for drying.

Chapter 4

Section 3 SPRAY COOLING OF MILKFAT INTO MILKFAT SPHERULITES

3.1 Introduction

In the proposal for this research (Jebson, 1989) it was postulated that fat spherulites could be made from milkfat fractions dissolved in propane solvent, and spray cooled using a method based on the principles of spray drying. Under suitable conditions, it is expected that the heat of evaporation of the propane will create sufficient cooling effect for solidifying the atomised fat droplets thus forming spherulites.

By applying the same principle, it was thought that atomised fat droplets could be made to solidify to form spherulites, if the temperature in the spray dryer is maintained very much below the freezing temperature of the fat fraction. Furthermore, by introducing these spherulites into the spray dryer to "collide" with atomised liquid intermediate fat fraction droplets, and provided certain conditions are met, it was expected that the fat spherulites will be "coated" with the droplets before solidifying to form coated spherulites.

Also, if the milkfat fraction which melts at intermediate temperatures could be either encapsulated or coated and made into spherical forms, with the milkfat fraction which melts at higher temperatures, and the spheres mixed with the remaining components(the low melting fractions), then the butter could be made with greater spreadability but with the same composition as normal butter, and the spreadability would not changed significantly with increase in temperature.

3.2 Objectives

Using the same spray dryer to introduce cool air circulation into the system, to determine the regimes and conditions under which spherulites can be produced by this method.

Manufacture of milkfat spherulites from different milkfat fractions and to determine the effects of spray cooling temperatures on the physical properties of the spherulites.

To investigate the effects temperature conditioning of the fat spherulites in relation to its change in physical and flowability properties.

To investigate whether fat spherulites can be "coated" with another fat fraction of lower melting point. Determine the degree of coating.

To investigate whether fat spherulites can be "coated" with another fat fraction of higher melting point and to determine the degree of coating.

To use the "coated" fat powder to make butter, thus confirming the postulation that softer butter can be manufactured.

3.3 Literature Search

No papers describing a spray cooling process of the type described were found in the literature search. However, it is known that high melting animal and vegetable fat powders can be produced and the equipment to make them are available.

3.4 Experimental

In the spray cooling process, only fresh frozen milkfat for recombining(FFMR) and/or milkfat fractions were used as feed. In the case of spray drying, recombined milk was used; that is, milkfat fraction was emulsified with protein based emulsifier in serum and homogenised.

3.4.1 Spray Dryer

The same Anhydro Spray Dryer (Lab S1) was used, except that in spray cooling, the spray dryer air inlet was connected to a blast freezer to supply regulated cooled dry air into the dryer system (refer Fig 1). The cyclone exhaust was connected in series to a filter to entrap fine particles not collected by the cyclone and the filtered air was recycled back to the blast freezer. A Centrifugal high speed disk atomizer was used for producing the fat droplets. A two fluid nozzle atomizer was not used (also available) as it would affect the cooling temperature in the dryer with the need to use compressed air. Fig 2 shows the schematic flow diagram of the process.

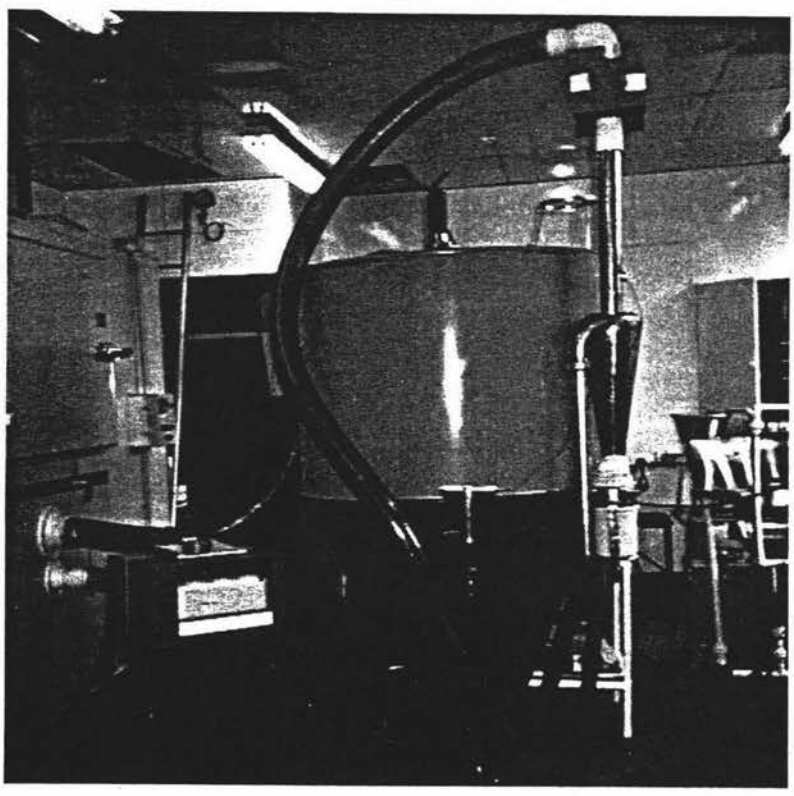


Fig 1 Photo of Spray Dryer Connected to the Blast Freezer

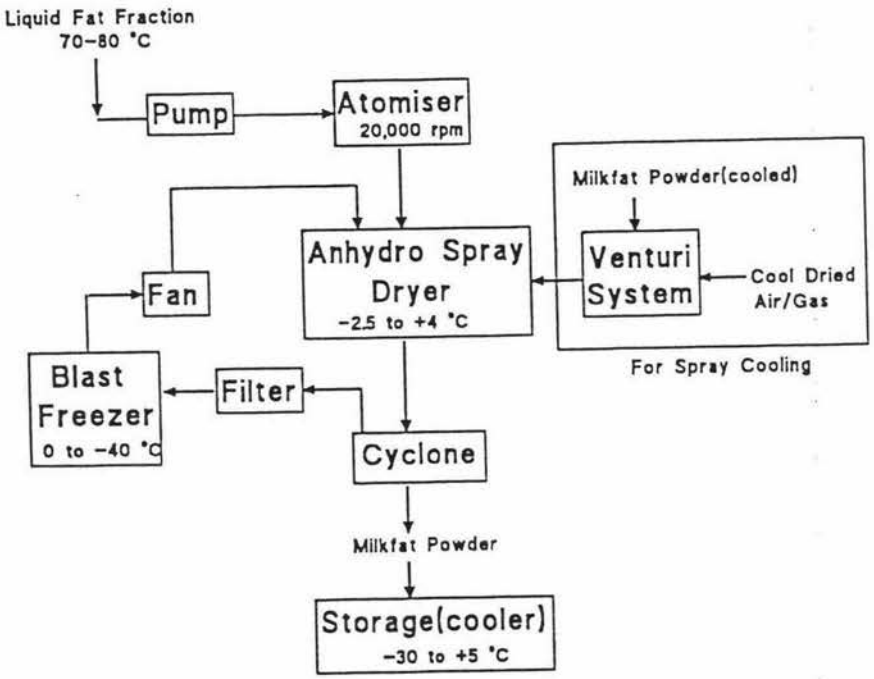


Fig 2 Schematic flow diagram of process for Spray Cooling

3.4.2 Blast Freezer

The blast freezer can be operated to provide up to $300m^3s^{-1}$ of -40°C cool dried air. Cool air is recirculated within the blast freezer compartment by means of a variable speed fan through a series of cooling coils. In the spray cooling process, a quantity of the cool air enters into the dryer via the spray dryer suction fan. The exhaust air is filtered and recycled back to the blast freezer compartment.

3.5 Materials

3.5.1 Milkfat

Fresh Frozen milkfat for Recombining (FFMR) constituting 99.9% milkfat and 0.1% moisture were obtained from the New Zealand Dairy Research Institute(NZDRI). Milkfat fractions of various composition and stages were obtained both from NZDRI and from Bay Milk Products(BMP) Ltd in Te Puke, New Zealand. Refer Chapter **1**, Fig 3-5 for details of NMR solid fat profile.

3.5.2 Milk Powder

Commercial exported grade Anchor Sweet Cream Buttermilk Powder and Skim Milk Powder from New Zealand Dairy Board(NZDB).

3.6 Methods

Section 3.4.1 outlined the production of 100% milkfat spherulites from a spray cooling process which was then used coating of these spherulites with another milkfat fraction.

Two different methods of producing "Coated" fat spherulites were tried.

3.6.1 Method One - *Using Venturi Nozzle Distributor*

In the "coating" of the high melting fat spherulites powder with liquid intermediate fat fraction, the powder was fed into the dryer system by means of a modified venturi feeder. The venturi feeding system was necessary due to the positive pressure in the spray dryer during operation. The feeder consists of a jacketed hopper connected to modified steam/water mixer venturi nozzle (refer Fig 3). Bottled high pressure carbon dioxide gas was used in the venturi system to maintain and regulate the cooling temperature in the dryer. The liquid intermediate fat fraction was fed through the centrifugal high speed atomizer where it atomises into droplets and met with an upward stream of the fat spherulites powder evenly distributed via a inverted cone shaped distributor (Fig 4). As the powder was fed very close to the atomised fat fraction, it was envisaged that prior to solidification to form powder, the atomised fat fraction would collide with the fat spherulites resulting in the coating of the atomised liquid fat onto the fat spherulites.

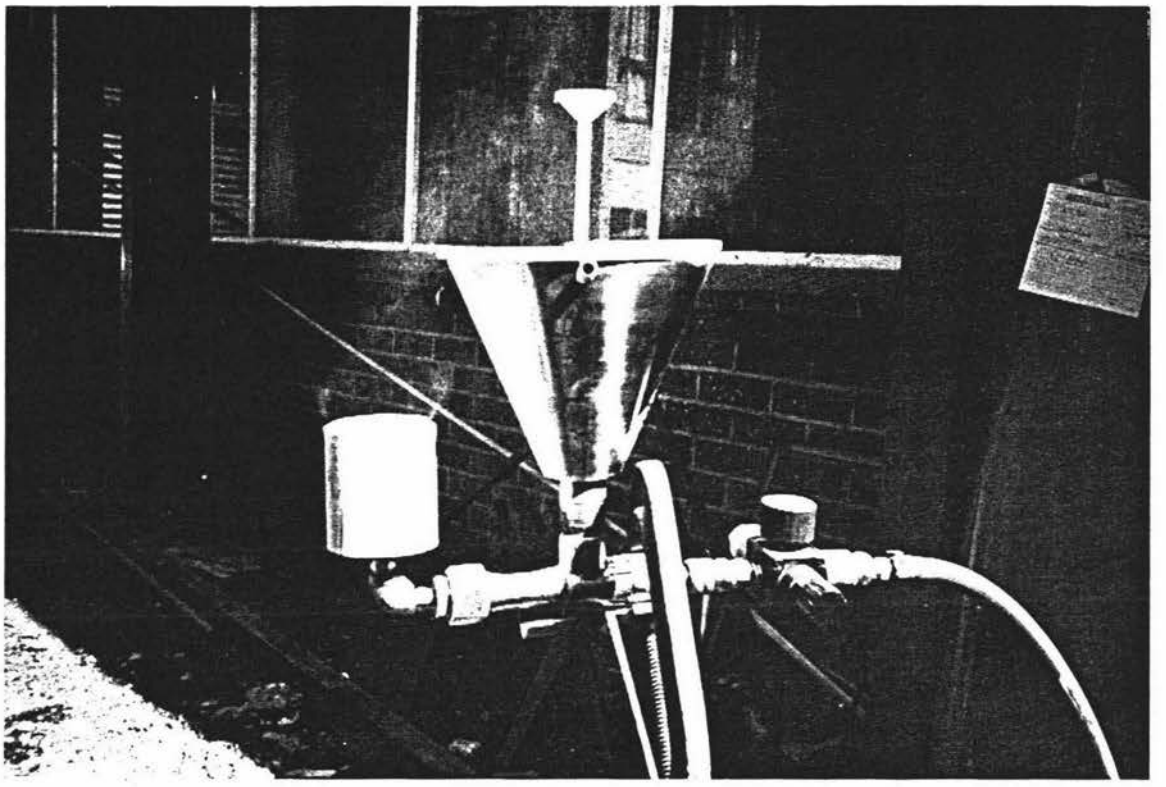


Fig 3 Venturi Mixer

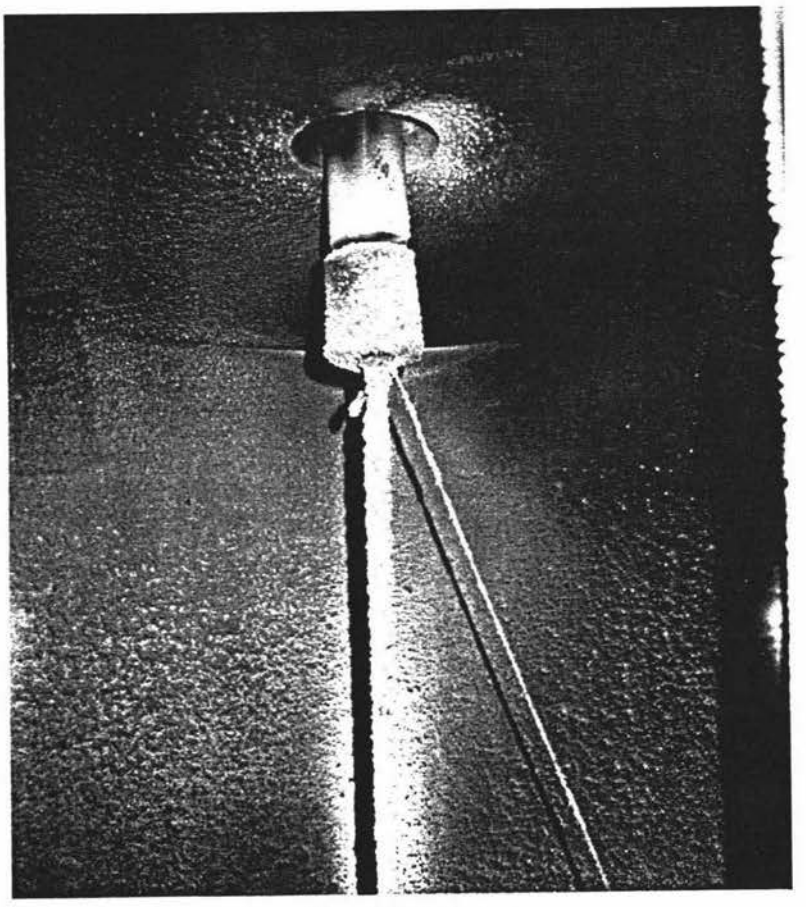


Fig 4 Diagram of Fat Spherulites Distributor

3.6.2 Method Two -*Using a Mixer to Maintain a Suspension of the Fat Spherulites and Liquid Fat prior to Spray Coating*

In Method One, the coating of the fat spherulites with a liquid fat fraction was unlikely to result in good coating efficiency decision was made to abandon this method. In order to overcome the low coating efficiency, the idea of mixing the fat spherulites with the liquid fat fraction in suspension prior to feeding was mooted. Fig 5 shows a schematic flow diagram of the method used.

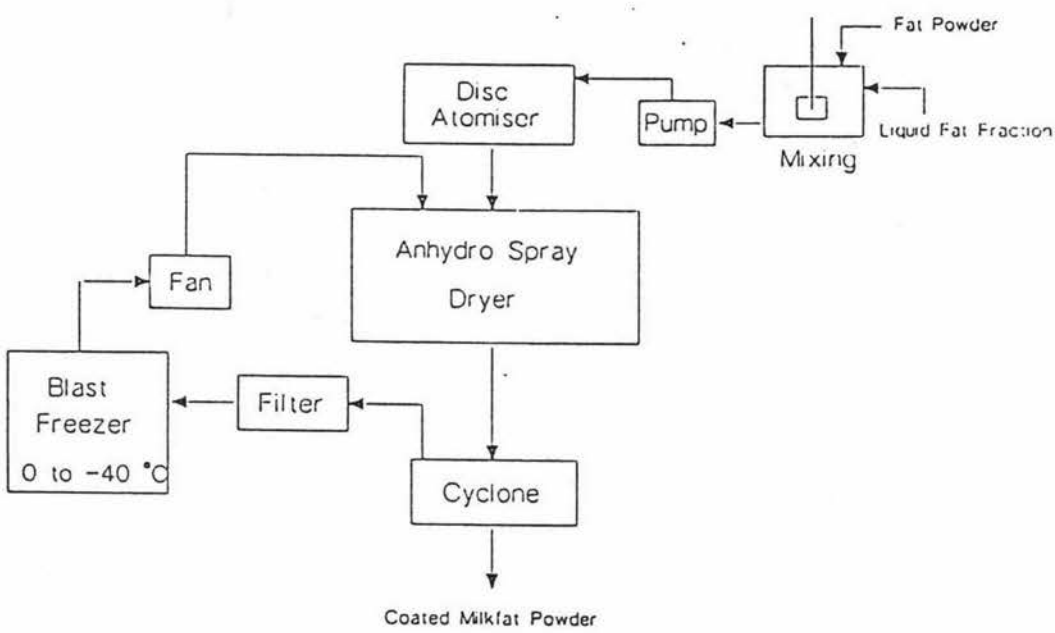


Fig 5 Schematic Flow Diagram of Spray Cooling of Milkfat Fractions

3.6.3 Determination of the Regimes at which the Milkfat can be spray Cooled to produce Fat Spherulites.

Preliminary dry runs were carried out to determine the limits of the operating temperatures that could be used in the spray dryer for spray cooling experimentations.

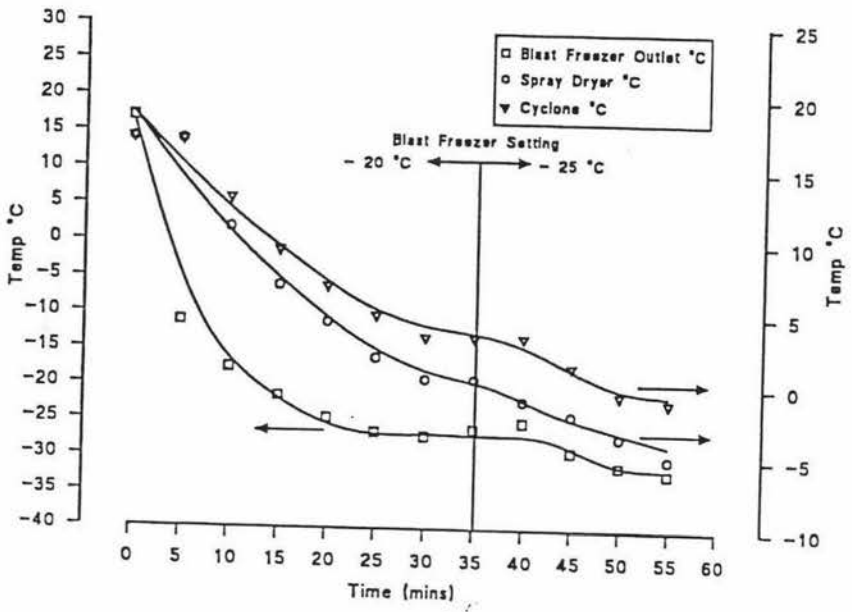


Fig 6 Effects of spray Dryer Temperatures in Spray Cooling

Fig 6 represents the temperature ranges at which the spray dryer connected to the blast freezer could be operated. Fig 2 indicates some of the parameters that were used in the production of the fat spherulites.

With operating conditions of the spray dryer at steady state, liquid milkfat heated and maintained at 70-80°C was fed to the variable high speed centrifugal disk atomizer. This atomised the fat into fine droplets which immediately cooled to below the freezing point of most of the solid fat components, and crystallised as solid spheres, forming free flowing powder. The fat powder was collected in the spray drier cyclone's container and placed immediately in a freezer room.

3.6.3.1 Results and Discussion

Initially, a number of problems were encountered in the determining the conditions required to produce a fat spherulites that would have the physical properties of good flowability and non-stickiness similar to low fat milk powder products. It is important to stress that this process is different from the normal spray drying process in that not only cooled air was used but the fact that the feed material constitutes only FFMR or milkfat fractions and hence there was no evaporation and change in feed composition. It became apparent that the factors contributing to the production of good fat spherulites were: temperature and flow of the feed, speed of disk atomizer, temperature of the dryer, and degree of exposure to ambient temperature of fat spherulite prior to storage at freezer temperature.

The liquid fat fraction was required to be heated and maintained at a temperature of at least 70-80°C prior to pumping to the disk atomizer. The high temperature is necessary to keep the fat fraction at low viscosity and to prevent solidification of the fat in the disk atomizer downpipe. The atomizer unit should be protected as much as possible in such a way that it is not unduly exposed to the low temperatures in the dryer.

Using FFMR and milkfat hard fraction, good fat spherulites with the powder-like physical properties of low fat milkfat powder could be produced when the spray dryer inlet air temperature is at 2°C or below. There were no significant differences in shapes and sizes of fat spherulites produced from FFMR and milkfat hard fraction. At higher spray dryer temperatures, there was a tendency for the spherulites to agglomerate and to stick to the walls of the dryer. This was to be expected, as at higher temperatures, the lower melting point fat components would not solidify. Fig 7 and Fig 8 show photomicrographs of the spray cooled powder made from FFMR and milkfat hard fraction.

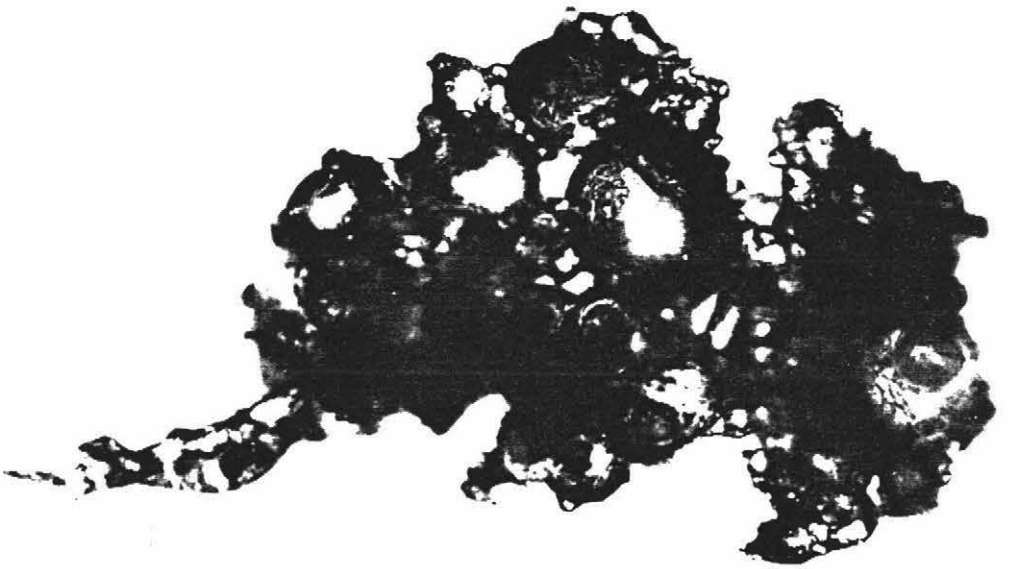


Fig 7 Photomicrograph of Spray Cooled Fat Powder (FFMR)

Fig 8 Photomicrograph of Spray Cooled Fat Powder (Hard Fraction)

It can be seen that fat spherulites, although appearing to have agglomeration taking place (largely due to the way the slides were prepared), are spherical in shape and individually formed. However, there was a significant variation in particle sizes.

When feed was introduced into the centre of the spinning disk atomizer, the concentrate on leaving the disk, forms into a thin film before breaking down into small droplets. At low dryer temperatures, fat spherulites with "tails" attached to the spherulites were formed (Fig 8). This can be explained by the fact that fat droplets solidified before they had time to form into complete spheres.

3.6.4 Production of Fat Spherulites at Different Spray Cooling Temperatures

The object of this experiment was to manufacture milkfat powder from FFMR and fat fractions by spray cooling them at different cooling temperatures. The powders were then subjected to various temperature conditionings for 1 hour and observations made in regards to any changes in the physical properties of the powder.

The feed rate through the high speed disk atomizer was set to a constant 40ml min^{-1} with a disk speed of 20,000 rpm. The spray dryer temperature was regulated by piping cool dry air from a blast freezer to the inlet of the spray dryer suction fan. Exhaust air through the dryer cyclone was passed through a paper filter and piped back to the blast freezer for recirculation.

3.6.4.1 Results and Discussion

Table 1 shows the types of fat fractions, operating conditions and visual observations made of the fresh powder products. These products were placed in a -30°C room for storage. Lower melting point milkfat powders, as expected, tend to melt and agglomerate much more easily than those of higher melting point powder. Milkfat powder with good flow properties can made from harder fat fractions and also at higher dryer temperature ranges. Microscopic views show the powder to be spherical in shape and have a narrow band of size distribution. It is difficult to distinguish differences in the physical

appearance of the powders was produced at different dryer temperatures. Generally, where the dryer temperature was higher, there was more tendency to have a higher proportion of milkfat powder sticking on the dryer wall. This powder, having been exposed longer at cooler temperature in the dryer, tended to be more fluffy in appearance.

It should be noted that the very small size of the drier means that comparatively small particles can reach the walls of the drier, and the samples collected do not contain all the larger particles generated. In general spray drying and spray cooling are easier in larger equipment.

The particle sizes were not determined at this stage, as the spherulites would start to agglomerate when exposed to temperatures higher than the dryer temperature. Viewed through a microscope and comparing the size with known milk powder size, it would appear the powder would be in the region of 150-200μm. It is expected that the sizes could be further reduced by increasing the speed of the disk atomizer.
The feed rate through the high speed disk atomizer was set to a constant 40ml min^{-1} with a disk speed of 20,000 rpm.

Table 1 Spray Cooled Milkfat Powder

Fat Fraction Used	Spray Dryer Temp °C	Fat Fraction Feed °C	Amb Temp °C	Observations of Sample
FFMR 1	1	80	18.3	powdery, dryer wall clean.
FFMR 2	3	80	18.0	powdery, some powder retain on dryer wall but removed with compress air.
H 1	1	80	19.4	powdery, small amount retained on dryer wall and had fluffy texture.
H 2	3	79	20.5	powdery, similar to powder at 1°C dryer temperature.
H 3	8	78	21.0	powdery, higher percentage of small particles attached to big ones.
SH 1	1	80	19.2	fine powder, fluffy powder retain on dryer wall, melts on contact with amb temp.
SH 2	2	80	20.6	powder similar to SH at 1°C dryer temp.
SH 3	5	80	20.6	powdery, powder sticking on dryer wall.
SSH 1	1	80	18.7	fine powder, powder retain on wall removed by compress air.
SSH 2	3	78-80	20.6	fine powder, powder sticking on dryer wall, melts on contact with amb temp.
SSH 3	5	78	20.5	powder similar to SSH at 4°C except more powder on wall.

Where,

FFMR is Fresh Frozen Milkfat for Recombining

H is the Milkfat Hard Fraction

SH is the Intermediate Milkfat Fraction I

SSH is the Intermediate Milkfat Fraction II

(refer Chapter 4 Section 1 Fig 5 for NMR Solid Fat Profile)

3.6.5 Temperature Conditioning of Milkfat Powder

The object of this experiment was to manufacture milkfat powder from FFMR and fat fractions by spray cooling them at different cooling temperatures. The powders were then subjected to various temperature conditioning for 1 hour and observations made in regard to any changes in the physical properties of the powder.

Samples of milkfat powder were placed in preconditioned petri dishes at the temperature required for conditioning of the powder. Visual observations of the powder were made after keeping the samples at the required temperature for one hour.

3.6.5.1 Results and Discussion

Table 2 Temperature Conditioning of Milkfat Powder

Fat Fraction	Conditioning Temp @9°C	Conditioning Temp @15°C	Conditioning Temp @20°C	Conditioning Temp @25°C
FFMR 1°C	powdery, normal	same as at 9°C	sticky but better than 3°C powder	partially melted and lumpy
FFMR 3°C	less powdery than 1°C powder	same as at 9°C	Sticky	partially melted and lumpy
H 1°C	powdery,normal	still powdery	slightly sticky, poor flow	sticky and some lumps
SH 3°C	powdery, normal	still powdery, sticky on touch	sticky and lumpy	liquid
SSH 3°C	normal, slight agglomeration	powder collapsing, sticky	semi liquid and lumpy	liquid

Milkfat powder can be made from normal milkfat and its fractions. However, the storage temperature must be kept lower than 15°C if stickiness is to be prevented. Softer fractions must be kept at temperatures lower than 9°C, but higher melting point fractions are satisfactory at temperatures up to 20°C. The above observations were made on the basis of a very small amount of handling of the powders. They need confirmation by

actual use of the powders under normal use conditions.

The results of the work show that FFMR or any milkfat fractions could be made into powder by this method, and such powders would have potential commercial applications particularly in the bakery industries.

3.7 Production of "Coated" Fat Spherulites from Spray Cooling

3.7.1 Introduction

In order to understand the problems that associate with the handling and feeding of the milkfat powder for spray coating, it was thought that skim milk powder could be used to be coated with liquid milkfat; skim milk powder being easier to handle, has good flowability properties and is not unduly affected by temperature change.

3.7.2 Method One - Using Venturi Nozzle Distributor

3.7.2.1 Using skim milk powder of known sizes as a substitute to solid fat particles, determine whether coating with liquid fat fraction in the spray dryer is possible

A commercial grade skim milk powder (of particle size mainly in the 80-100μm range) was used. We have shown earlier that fat spherulites could be produced and observation through microscope indicated that they are spherical in shape, and apart from exhibiting higher tendency of agglomeration and size, were not dissimilar to powder made from buttermilk and skim milk.

Investigations into coating were carried out to determine the effects of using fine classified skim milk powder. The reasoning was that the coated fat powder produced at a later stage would probably have to be in the range of 20-40μm in size for butter making to produce a smooth texture butter. Hence, very small particles would have to be used for coating.

Using an Alpine Type MZM 1/40 Zig Zag Air Classifier, two lots of fine powder were produced from commercial grade skim milk powder. Particle analysis using the Malvern Particle Analyzer show the sizes of both lots of sample to be D(v,0.5)=10.7 and 50.5μm, and D(v,0.9)=20.4 and 123.2μm respectively.

The skim milk powder of D(v,0.5)=10.7μm in particle size could not be fed at through the venturi system due to electrostatic effects of the fine powder. The powder tended to stick on the wall of the venturi hopper. Hence, only trials using skim milk powder of D(v,0.5)=50.5μm in size were conducted.

3.7.2.2 Determination of the Degree of Coating

In the initial method of determining the degree of coating of skim milk powder in fat spherulites, a reference graph was produced by using a spectrophotometer to determine the absorbance of varying concentrations of skim milk powder solutions.

The coated fat particles were washed with cold distilled water to remove uncoated skim milk powder, centrifuged, and the top fat layer removed. The fat was added to distilled water and heated to melt the fat and dissolve the skim milk powder encapsulated in the fat particles. The absorbance was determined on the supernatant liquid. This was cross-referenced with the reference graph to determine the concentration. Hence, the amount of skim milk powder encapsulated or coated in a given quantity of coated fat particles can be quantified.

Difficulties were experienced with the above method in reproducing results, and particularly in obtaining the complete removal of the centrifuged top fat layer. The concentration and thus the degree of coating is solely based on the absorbance (or turbidity) from the spectrophotometer reading.

Subsequently, a modified method was used to take a representative sample of coated fat spherulites and using Rose-Gottlieb method to determine the fat content of a "unwashed" and "washed" samples.

"Unwashed" sample means a representative sample of coated fat spherulites.

"Washed" sample means a representative sample of coated fat spherulites of known weight, placed in a bucknel funnel with filter paper. Cool distilled water is sprayed onto the sample and the supernatant is continually removed by vacuum suction. This is done until the supernatant liquid is clear and the milkfat on the filter paper has all adhering water removed.

Both "unwashed" and "washed" samples were than analyzed to determine the protein content using the Kjeltec System of protein determination. Skim milk powder was also analyzed to determine the protein content.

The percentage of Coating Efficiency is the ratio of the percentage protein in the "washed" sample to that of the "unwashed" sample.

From the above results, the actual degree of coating can then be determined.

3.7.2.3 Determination of Particle Size of Spray Cooled Coated Fat spherulites

Macintosh NIH Image 1.44 Scanner

An Image scanner is used to determine the various particle sizes and shapes of the fat spherulites. Fig 9 shows the layout of the Image Scanner consisting of a Macintosh computer connected to a video camera mounted onto a microscope. Some scanned pictures are included in this report.

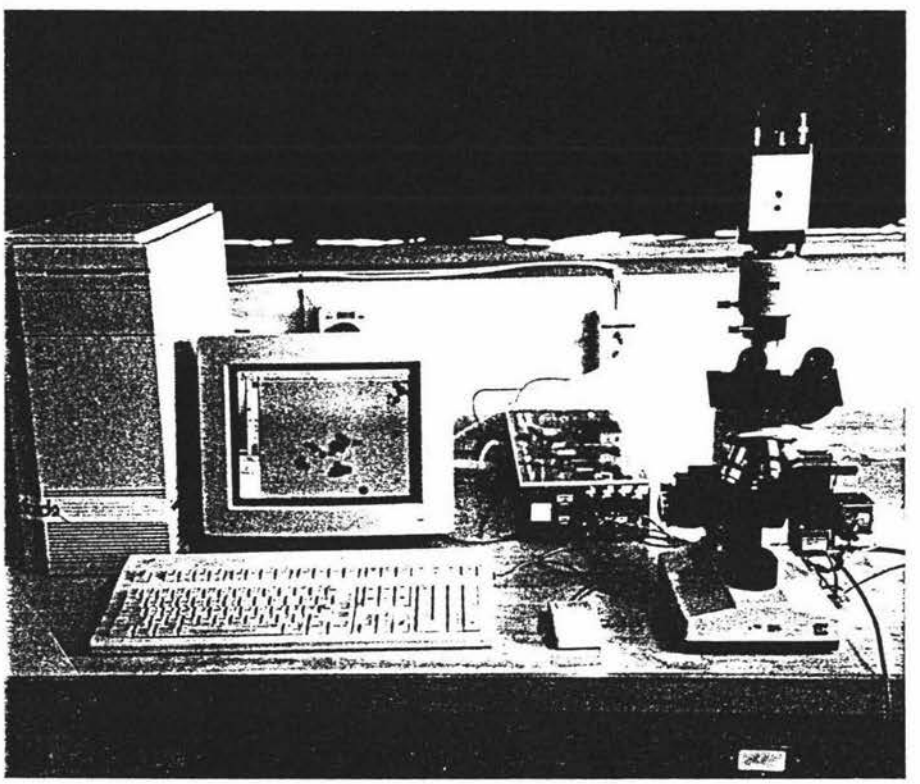


Fig 9 NIH Image Scanner

Vibratory Feeder

An AccuRate 100 series variable dry material feeder was purchased for coupling onto the venturi feeder to feed skim milk powder/butter powder into the spray dryer for encapsulation/coating with liquid fat fraction. With this feeder, it is now possible to determine and control the amount of feed powder introduced into the spray dryer.

3.7.2.4 Results and Discussion

Microscopic examination indicated that some the spheres "coated" were made up of a group of skim milk particles enclosed in an enlarged hard fraction sphere (Fig 9). However, there were a number of uncoated skim milk powder particles. This could be attributed to the fact that the proportion of skim milk powder, distributed into the dryer, was at times very high to fat droplets as it was difficult to maintain a consistent control of the powder through the venturi. It is expected that a variable feed vibratory feeder would overcome this problem. It was not expected that this method of coating fat spherulites would achieved high coating efficiency, but this work has illustrated that the concept of coated fat spherulites from spray cooling was feasible and could be used as a basis for further work.

Fig 10 Photomicrographs of Spray Cooling "coated" Spherulites with Skim Milk Powder

Fat determination showed that no significant fat was carried into the supernatant liquid during washing. This suggested that in the method used for washing the coated fat, spherulites are not lost in the washing. The skim milk powder used in the trials was found to contain 1.2% fat and 36.4% protein using Rose-Gollieb and Kjeltec system respectively.

Table 2 Coating Efficiency of Spray Cool Milkfat and Skim Milk Powder

Ratio		% Protein Content		% Coating Efficiency
Fat	Powder	Unwashed	Washed	
80	20	1.88	1.00	53
75	25	16.32	5.95	36
50	50	15.22	2.92	17

It appears that the method of determining the percentage of coated particles used in these trials gives a more accurate measurement than that used in the previous trials, and the results obtained agreed with qualitative assessments made using microscopic observation.

The above results represent an overview of the coating efficiencies that could be achieved with the equipment that was used. Other operating parameters were tested, but the results are not reported as representative samples could not be obtained. Some of these parameters (feed rates, feed rate ratio and temperatures) settings were beyond the limits for the equipment to operate consistently. It is expected that better all round results can be achieved if such experimentation were carried out during the winter months. These trials were run at ambient temperatures of 18-20°C which meant that there were problms with particles sticking to the walls of the dryer. Generally, an increase in fat to powder ratio increases the coating efficiency.

Fig 11 Scanned Picture of One "Coated" Fat Spherulite (400X mag)

Fig 11 shows a spray cool coated fat spherulite using skim milk powder and fat fraction "H". The coated particles are spherical in shape with sizes in the range of 180μm to as large as 300μm. It can be seen that some of the skim milk particles are not coated but rather attached to the fat spherulite. It was apparent that the degree of coating in this case was seen to be much better than if larger skim milk particles were used.It is apparent that the coated spherulites consists of fat encapsulating a number of skim milk powder particles.

We have shown that spray cooling of milkfat fraction of high melting points in a spray dryer to produce spherical fat particles was achievable. However, the "coating" of these particles with a liquid fat fraction under the present equipment setup could not be sustained long enough to produce a representative sample for analysis. The main problem was the difficulty of maintaining a cooled surrounding of the venturi feeding

system that would not cause deterioration of the physical properties of the fat particles. It is suggested if this system was further investigated, that either, the whole spray cooled system be placed in an enclosed freezer or that an enclosed temperature controlled spiral type vibratory feeding system be used.

However, it was thought that the spray cooling of a suspension might produce better results and this approach was tried next.

3.7.3 **Method Two** - Using a Mixer to Maintain a Suspension of the Fat Spherulites and Liquid Fat prior to Spray Coating

3.7.3.1 Production of "coated" Fat Spherulites by Mixing of Milkfat Powder with liquid Fat in Suspension

Introduction

In Method One, we were able to show that cooling the melted coating milkfat fraction in a spray dryer while pneumatically conveying spherical particles (in this case, sized skim milk powder) to be coated under the atomising disc in a manner similar to agglomeration in spray drying, was achievable. As much as 53% coating efficiency could be obtained. However, using the same equipment setup to "coat" milkfat fraction powder with another liquid milkfat fraction, it was not possible to produce a representative sample for analysis. The main problems were trying to maintain a cool temperature that will not affect the flowability properties of the fat powder, and the feeding of the powder into the disc atomizer mixing chamber. It could reasonably be expected that operation in a larger plant would overcome a number of problems that were encountered in a small pilot plant scale operation. However, it was decided to examine other procedures for producing coated spherulites.

To overcome the mixing problem and the low coating efficiency, the idea of mixing the milkfat powder with a liquid milkfat fraction in suspension prior to feeding into the disc atomizer was mooted. Fig 12 shows a schematic flow diagram of the system.

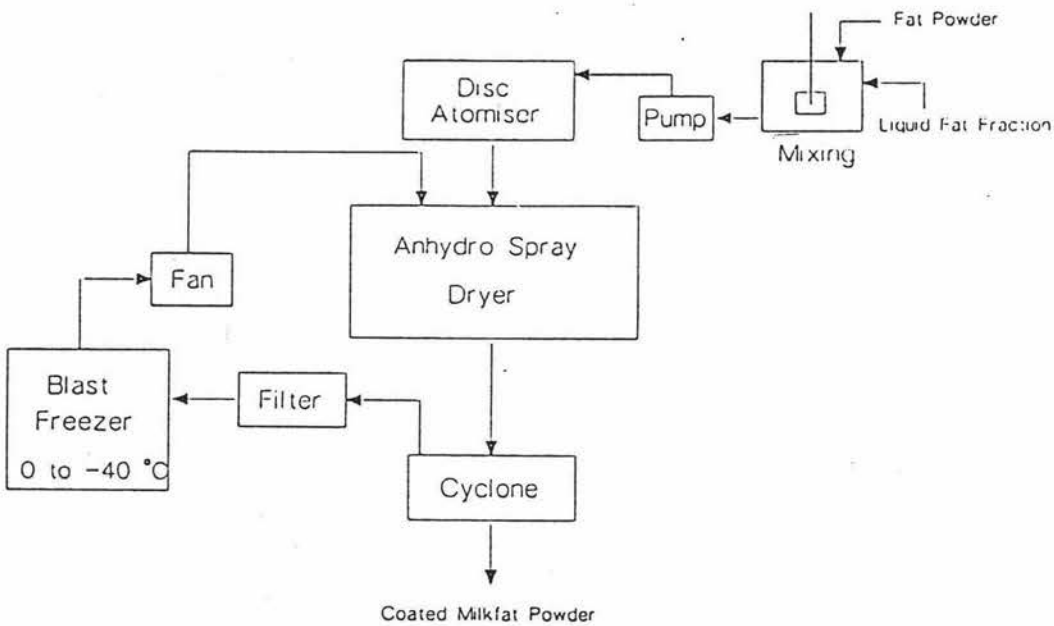


Fig 12 Schematic Flow Diagram

3.7.3.2 Objectives

To produce coated milkfat spherulites in which a *high* melting point fraction is covered with a *low* melting point fraction.

To produce coated milkfat powder in which a *low* melting point fraction is covered with a *high* melting point fraction

3.7.3.3 Mixing of Fat Powder with Liquid Fat in Suspension

Initial trials with mixing a hard milkfat fraction "H" powder with a soft milkfat fraction "SSS" liquid showed a promising outcome. This combination was chosen as there would be comparatively slow rates of solution of the one fraction to the other. By mixing the powder and the liquid together, it can reasonably be assumed that the powder produced from these mixtures would give a 100% coating of the fat powder. It also became obvious that the temperatures of the fat powder and the liquid fat prior to mixing, the resulting mixing temperature, the ratio of the solid to liquid fat, and the melting points of the fat powder and liquid fat would determine the viscosity of the feed and the degree of melting of fat powder with consequent incorporation into the liquid fat.

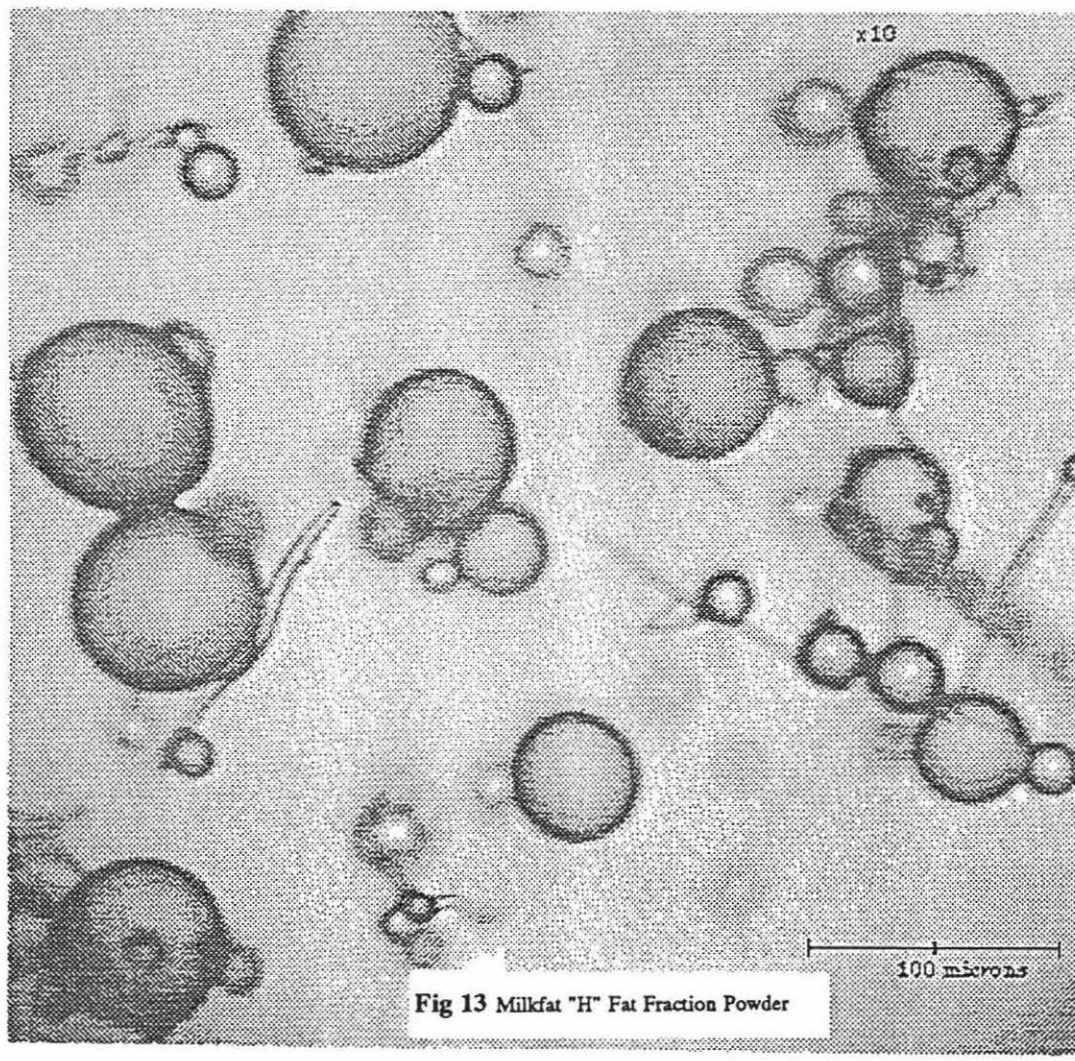


Fig 13 Milkfat "H" Fat Fraction Powder

Fig 13 shows a scanned picture of the "H" fat fraction powder spray cooled at 1°C.

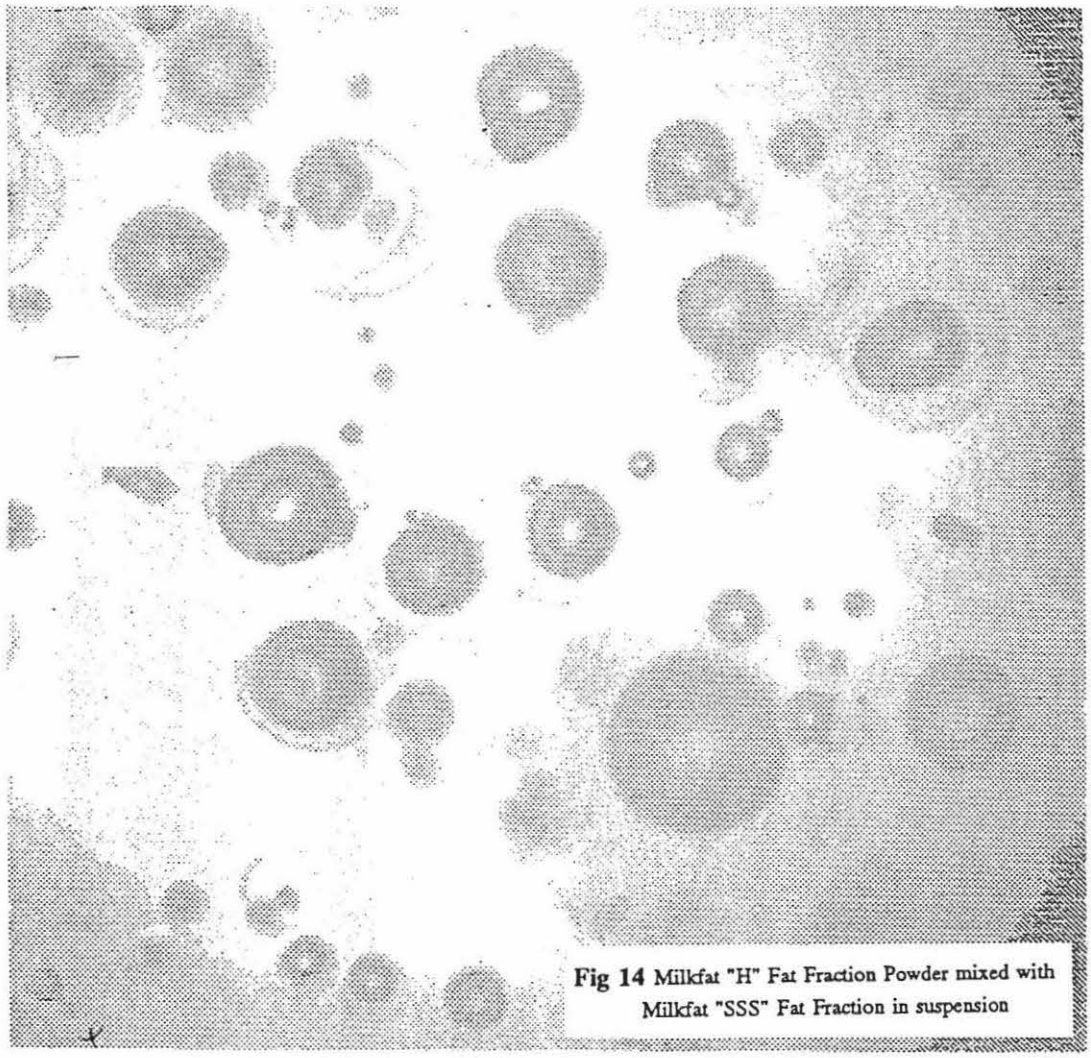


Fig 14 Milkfat "H" Fat Fraction Powder mixed with Milkfat "SSS" Fat Fraction in suspension

Fig 14 shows the fat powder(Fig 13) in suspension in a liquid fat fraction. The fat powder was conditioned at 3-5°C and mixed with liquid "SSS" fat fraction at 30°C. Ten percent solid in liquid was used.

3.7.3.4 Spray Coating of high melting milkfat fat fraction powder with low melting milkfat liquid fat fraction

In the following experiments, "H" powder conditioned at 5°C was mixed with "SH" liquid fat fraction at 30-32°C in the ratio of 10:90 w/w respectively. "H" and "SH" fat

fractions combinations are likely to be required commercially. The mixture was maintained at 25°C in a water bath. A Masterflex pump was used to pump the mixtures into the high speed centrifugal atomizer operating at a speed of 20,000-35,000 rpm. The Spray Dryer (Anhydro Type Lab S1) was maintained at various cooling temperatures by means of recycled cooled air from a blast freezer.

3.7.3.5 Spray Cooling at -3°C

Fig 15 shows a scanned picture of coated fat spherulites spray cooled at -3°C. The appearance of the spherulites was powdery, and microscopic observations indicated that particles were not spherical in shape but clusters of crystallised fat. Tail ends of particles were prominent. The tails were probably caused by the freezing of the particles before they can be formed into complete spheres.

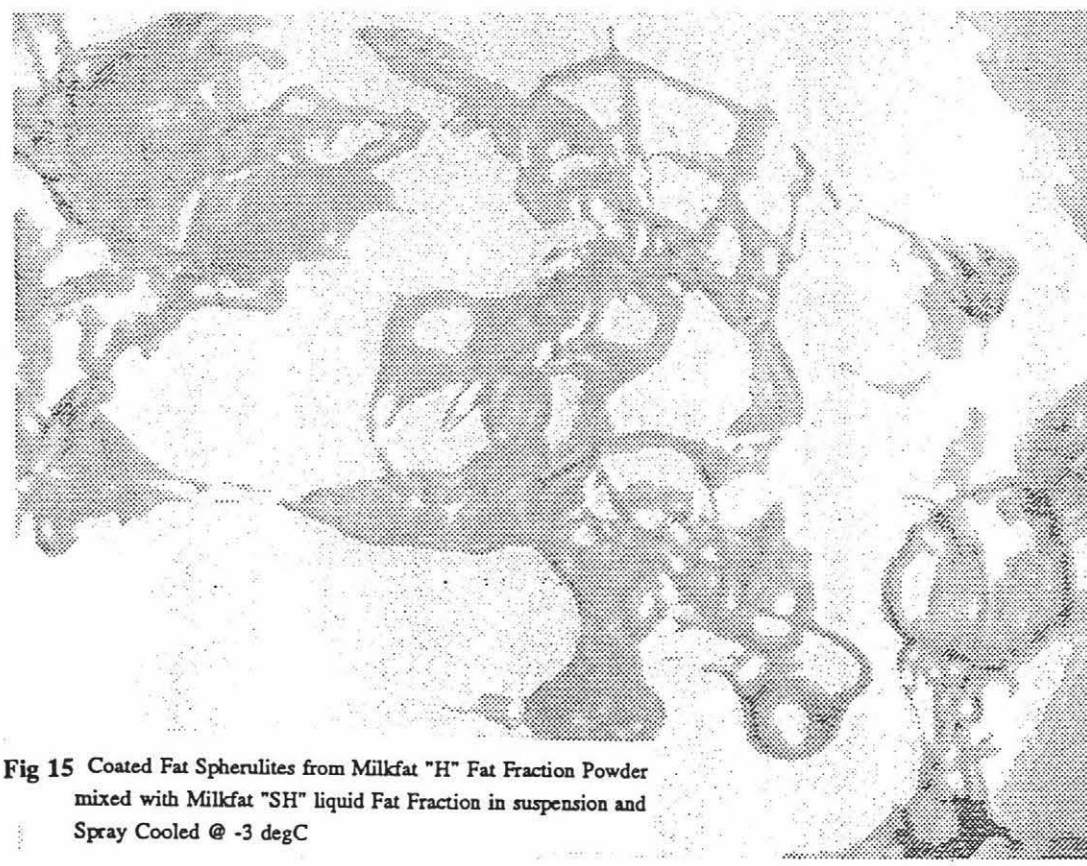


Fig 15 Coated Fat Spherulites from Milkfat "H" Fat Fraction Powder mixed with Milkfat "SH" liquid Fat Fraction in suspension and Spray Cooled @ -3 degC

Fig 15 Scanned picture of coated fat spherulites spray cooled at -3°C

3.7.3.6 Spray Cooling at 3-4°C

Fig 16 shows a scanned picture of coated fat spherulites spray cooled at 3-4 s. Formation of tail ends were more prominent on smaller spherulites and less so on the bigger spherulites. Generally, the powder has good flow properties but these will deteriorate rapidly if the powder is exposed to high ambient temperatures.

Fig 16 Coated Fat Spherulites from Milkfat "H" Fat Fraction Powder mixed with Milkfat "SH" liquid Fat Fraction in suspension and Spray Cooled @ 3-4 degC

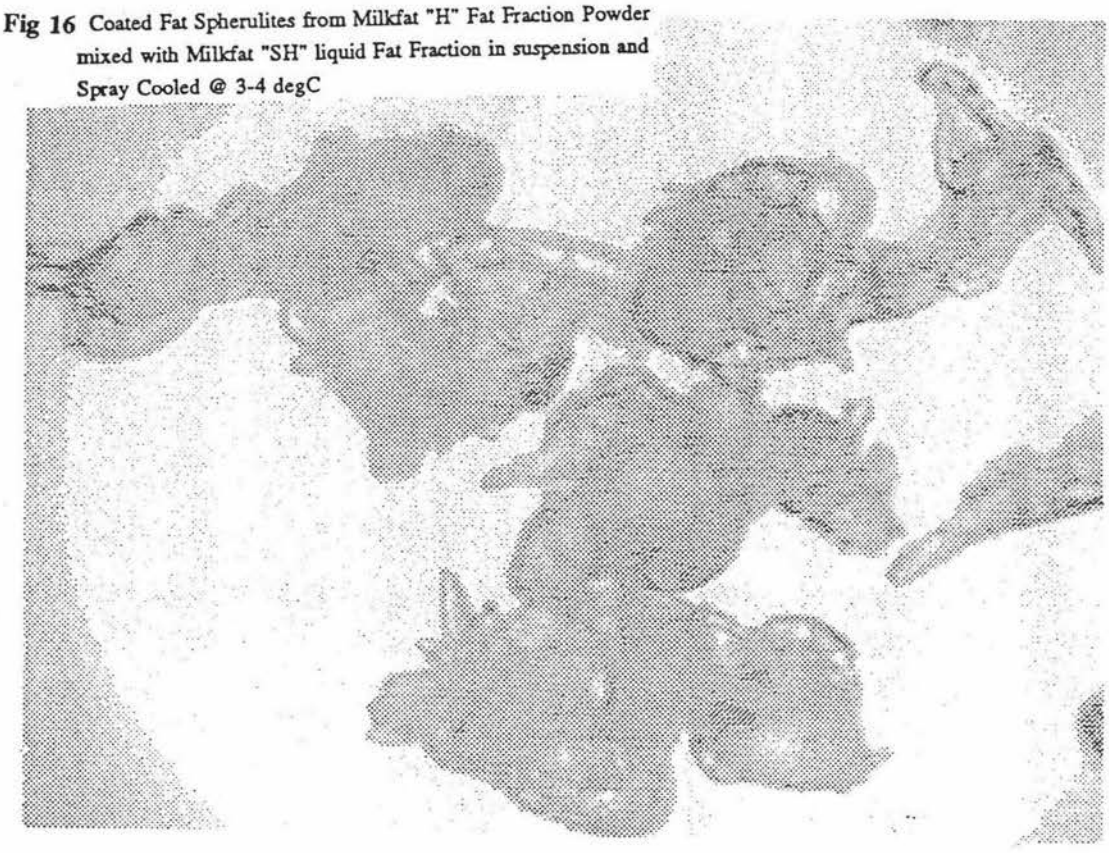


Fig 16 Scanned picture of coated fat spherulites spray cooled at 3-4°C

3.7.3.6 Spray Cooling at ~~3-4°C~~ 5-6

Fig 16 shows a scanned picture of coated fat spherulites spray cooled at ~~3-4~~ 5-6 s. Formation of tail ends were more prominent on smaller spherulites and less so on the bigger spherulites. Generally, the powder has good flow properties but these will deteriorate rapidly if the powder is exposed to high ambient temperatures.

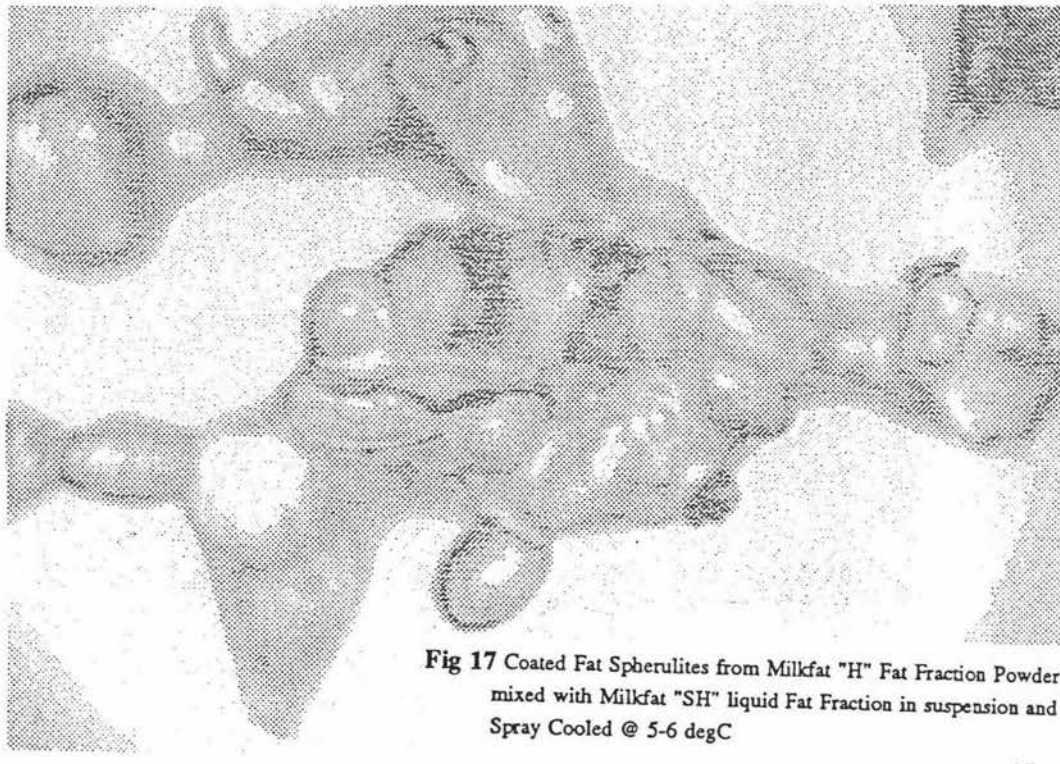


Fig 17 Coated Fat Spherulites from Milkfat "H" Fat Fraction Powder mixed with Milkfat "SH" liquid Fat Fraction in suspension and Spray Cooled @ 5-6 degC

Fig ~~16~~ 17 Scanned picture of coated fat spherulites spray cooled at ~~3-4°C~~ 5-6

3.7.3.8 Spray Cooling at 8°C

Fig 18 shows a scanned picture of coated fat spherulites spray cooled at 8°C. In this case, there were very little tail ends (if any) on the spherulites but the tendency for the powders to stick on the dryer walls and agglomerate were enhanced. These powders still retain good flow properties.

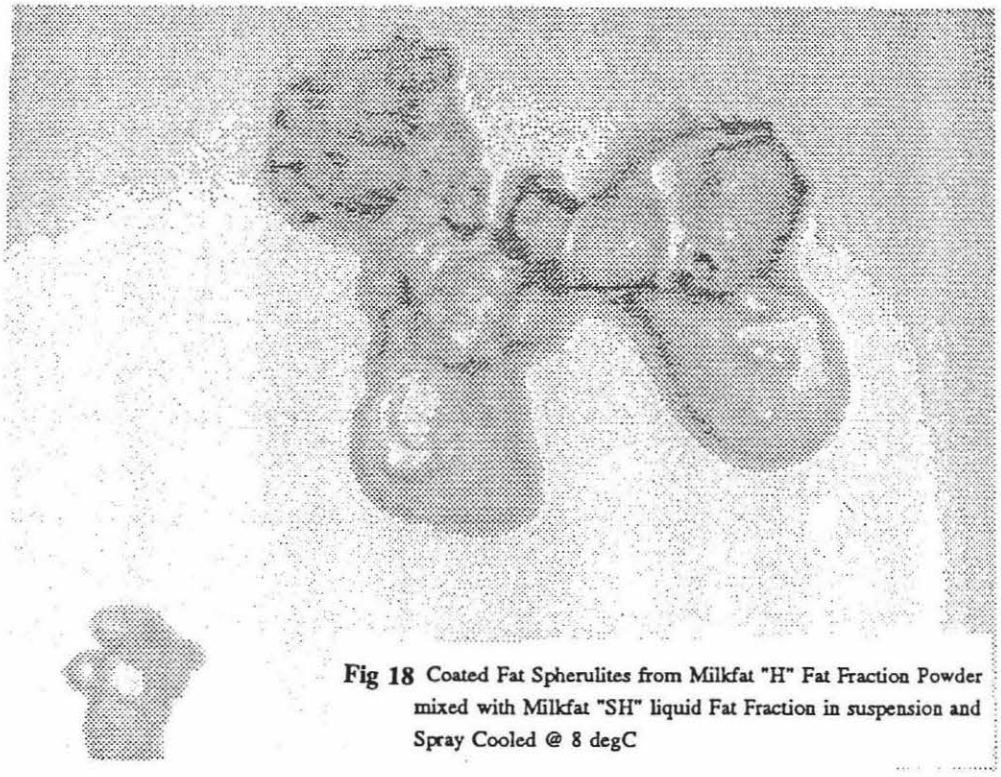


Fig 18 Scanned picture of coated fat spherulites spray cooled at 8°C

3.7.3.9 Results and Discussion

We have shown that under suitable operating conditions, fat powders from 100% milkfat or its fractions can be produced by spray cooling in a spray dryer. Provided these powders are not unduly exposed to high temperatures (refer report 9, p 7), they exhibit good powder properties.

Coating fat spherulites made by spray cooling of fat powder in suspension in liquid fat showed promising results. In this configuration, it can be safely assumed that coating efficiency of the coated powder is 100%. Obviously, there will be spherulites that do not contain any encapsulated fat powder. Depending on the types of fat fractions used for powder/liquid, it is apparent that the dryer temperature, ratio of mixtures, and the speed of the high speed centrifugal atomizer would governs the size and physical properties of the coated spherulites produced.

3.7.3.10 Spray Coating of low melting milkfat fat fraction powder with high melting milkfat liquid fat fraction

Having shown that coated fat spherulites could be produced using a high melting point fat fraction "H" powder encapsulated with lower melting point fat fraction "SH" liquid, our next objective was to invert the fat fractions and produced coated spherulites with "SH" fat powder encapsulated with "H" liquid fat fraction. The coated spherulites made from this configuration would then be in consonant with the primary objectives of producing soft butter from these spherulites. That is, the use of encapsulated intermediate fat fractions with hard fraction as components in butter making would ideally produced a butter with the full compliments of milkfat components that will be soft, as the intermediate fat fraction would not contribute to the butter hardness. At the same time, the hard fraction component would ensure that the soft butter have good stand up and oil off properties.

The equipment was therefore modified. A magnetic stirrer with thermostatic control was used for mixing and temperature control. A Masterflex pump was used to pump the

mixtures into the high speed centrifugal atomizer operating at a speed of 20,000-35,000 rpm. Spray Dryer (Anhydro Type Lab S1) was maintained at 5-8°C. This temperature was earlier determined to be suitable for producing coated spherulites with minimal tail ends.

In the early trials, it became apparent that a temperature balance between keeping a high enough temperature of the hard fraction "H" in liquid state and at the same time maintaining a temperature that must be low enough to ensure that the intermediate "SH" fat powder does not melt during mixing, was critical. It was noted that the gradual mixing of the powder into the liquid fat would results in a drop in the mixing temperature and this rapidly increased the rate of crystallisation (the powders acted as seeding crystal nuclei), such that the mixtures solidified rather quickly. Nevertheless, it was possible to produce some coated spherulites by careful control of the mixture temperature and more importantly by minimising the mixing time of the powder/liquid and feeding of the mixtures into the atomizer. At this stage, the setup of the equipment was such that it is difficult to produce a sustainable run without incurring some operational problems. In these experiments, "SH" powder conditioned at 5°C was mixed with "H" liquid fat fraction at 30°C.

3.7.3.11 Results and Discussion

It is reasonable to expect that during mixing, a small proportion of the "SH" powder would have melted into the "H" liquid fat fraction. However when the coated powder was subjected to melting tests, there was little evidence of significant melting of the coated powder. The melting tests was carried out by using a Leitz HM-LUX polarised hot stage microscope. This suggests that the actual amount of melting is small. At this stage, the ratio of powder to liquid fat fractions could not be determined but suffice to say that sufficient powdered "SH" was added to ensure that there was a suspension of powders fed to the atomizer.

In the determination of the melting tests, samples of the fat spherulites were taken in the chiller and placed on individual slides. Each sample was then placed on the hot stage

microscope and a preset heating was turn. Observation was made on the sample through the microscope and also the temperture probe to determine the temperature at which the fat spherulites showing sign of melting.

Table 3 Melting point temperature of uncoated and coated Milkfat spherulites

Sample	Melting Temperature °C
Hard "H" Fat Fraction	35-36
Intermediate "SH" Fat Fraction I	24
Intermediate "SSH" Fat Fraction II	22
"H" powder + "SH" liquid	23-24
"SH" powder + "H" liquid	36

The above results clearly suggested that the "SH" fat powder coated with "H" liquid fat fraction was successful. The encapsulated lower melting point "SH" powder did not melt until the melting temperature of the "H" fat fraction was attained. Fig 19 show a scanned picture of the coated spherulites.

Fig 19 Coated Spherulites from Milkfat "SH" Fat Fraction Powder mixed with Milkfat "H" liquid Fat Fraction in suspension and Spray Cooled @ 5-8 degC

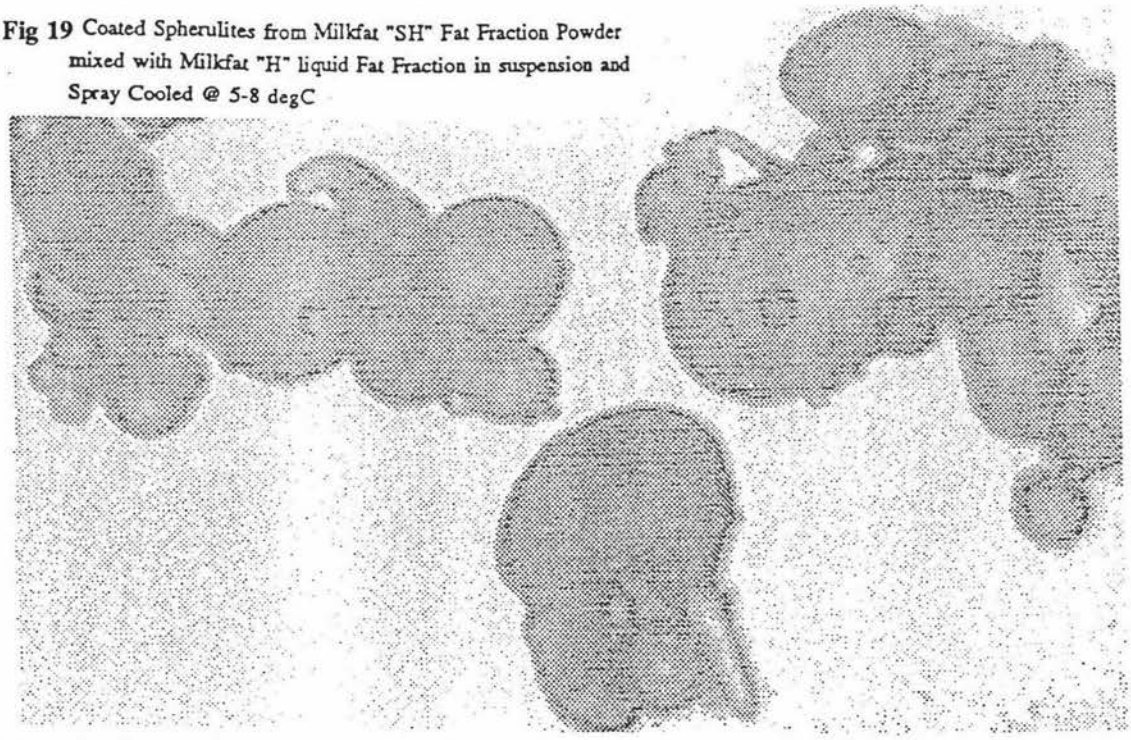


Fig 19 Scanned picture of coated fat spherulites spray cooled at 5-8°C

3.8 CONCLUSION

Under suitable conditions, spray cooled milkfat fraction powder can be made into a suspension with a different liquid milkfat fraction. The conditions are less critical if the suspension are made of a higher melting point fat fraction powder and a lower melting point liquid fat fraction instead of the reverse.

If this suspension mixture is spray cooled at suitable operating conditions in a spray dryer, coated fat spherulites in powdered form are produced. Where lower melting point fat powder are encapsulated with a higher melting point liquid fat, melting point test indicated that the "coating" of the high melting point liquid fat was sufficiently strong to "protect" the encapsulated lower melting point powder from melting at its melting point temperature. The efficiency of coating appeared to be very high, but there probably was a proportion of spherulites consisting entirely of the coating fat.

At this stage and with the present equipment setup, it would appear that there is a limit to the proportion of the fat powder that could be made in suspension in a liquid fat such that it would not increase the viscosity of the mixture unduly for feeding into the atomizer. It is expected that with a pilot size equipment and better control of the temperatures, significant improvement in the manufacture of the "coated" fat spherulites could be realised.

Chapter 4

Section 4 Buttermaking

4.1 Introduction

Traditionally butter is made by a churning process (McDowall, 1953) which includes cooling cream to crystallise partially the milk fat, then agitating in the presence of air-bubbles. These assist in destabilising the cream (an oil-in-water emulsion) and the oil droplets clump together and enclose part of the original aqueous phase of the butter product.

As early as the 1920s, van Dam (1927), observed that the temperature at which the cream is stored before churning influences the physical properties of butter and the loss of fat in buttermilk. Samuelsson and Petterson (1937) Alnarp method of cream treatment is still very much the basis for buttermaking which has the best possible spreadability particularly at the time of the year when hard fats are encountered. This involves cooling of milkfat which minimize mixed crystal formation and maximise the proportion of liquid fat at any temperature. This method has been kept under constant review with minor changes of technique being introduced to achieve improved results. Alnarp process techniques have been investigated and different modifications were made by a number of workers, Dolby (1959), Fisker (1962), Mortenson and Danmark (1982), Wood and Dolby (1965), Taylor and Jebson (1974) and Dixon (1974) generally concluded that the crystallisation temperature and holding period can give further improvement in butter spreadability of butter hardness approximately 70% of that of butter produced using the normal cream cooling buttermaking method. Although these improvements have shown positive results but it has not been sufficient to compete on the same level as vegetable based spreads in terms of spreadability.

The desire to increase the range of application of milkfat in dairy products have led to research into fractionation of anhydrous milkfat into components having different melting ranges. Reviews on the fractionation of milkfat have been written by Jebson,

1970; Esdale, 1970; Sherbon, 1974; Wilson, 1975; Walstra *et al*, 1975; and, Tirtiaux, 1983. A number of methods of separating milkfat crystals from the liquid phase have been studied. Some of these process are by detergents, liquation and tirtiaux processes.

Some work on fractionation of milkfat with acetone solution undertaken in NZDRI as described by Norris *et al*, 1973, was carried out and reported. This process has been shown to produce a much wider melting range of fractions then other more conventional methods. Butter from these purer harder and softer fractions, when mixed in the appropriate proportions, can have adequate stand-up properties as well as being spreadable from refrigerator and ambient temperatures (Norris, 1973). However, it is unlikely that such a fractionation process using solvent would be acceptable to the dairy industry. Fig 1 show a typical melting properties of milkfat and milkfat fractions.

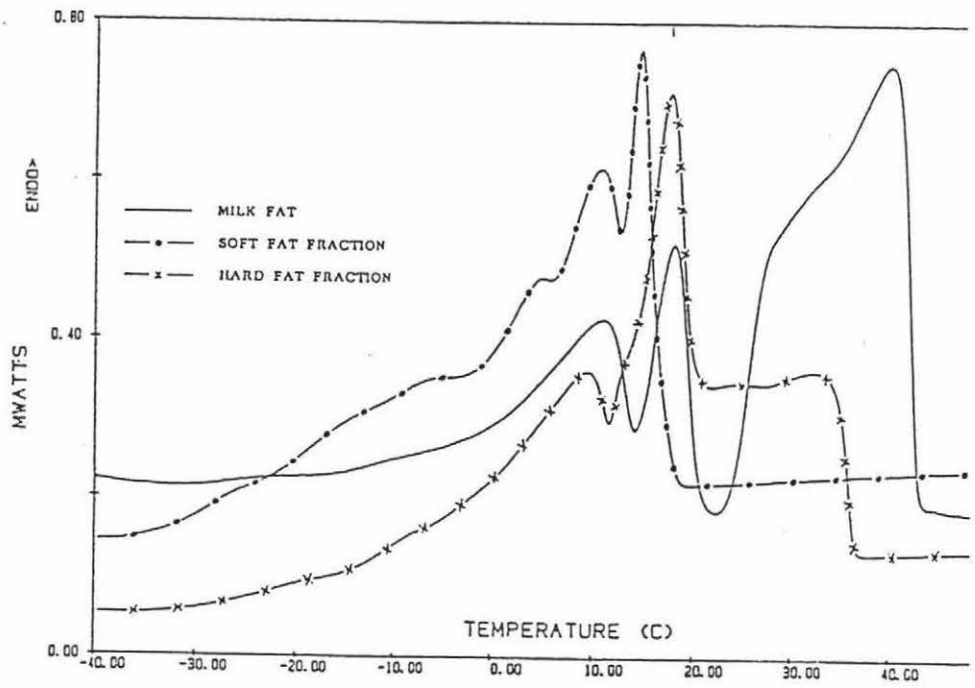


Fig 1 Melting Properties of Milkfat and Milkfat Fractions

4.2 Objectives

More spreadable butter can be made from soft fat fractions but such butters would invariable suffers poor stand-up properties and is also less stable against flavour deterioration. Furthermore, these butters do not contain all full milkfat components and could not be strictly classified as butter.

Chapter 4 Section 1, Effect of Homogenisation and Emulsifiers on Milkfat and **Chapter 4 Section 2**, Spray Cooling of Milkfat into Fat and Coated Fat Spherulites described the methods used to change the rheological properties of fat.

This section describes the various methods used, to produce butter from these fat spherulites, and to determine the physical and textural properties of such butters.

4.3 Equipment and Material

4.3.1 Equipment

An Intron Testing Instruments Series 4502 as shown in Fig 2 was used for butter sectility hardness tests. Fig 3 and 4 shows the sample ring and the attachment to which the wire was pretensioned. Results of the load in relation to displacement were plotted graphically.

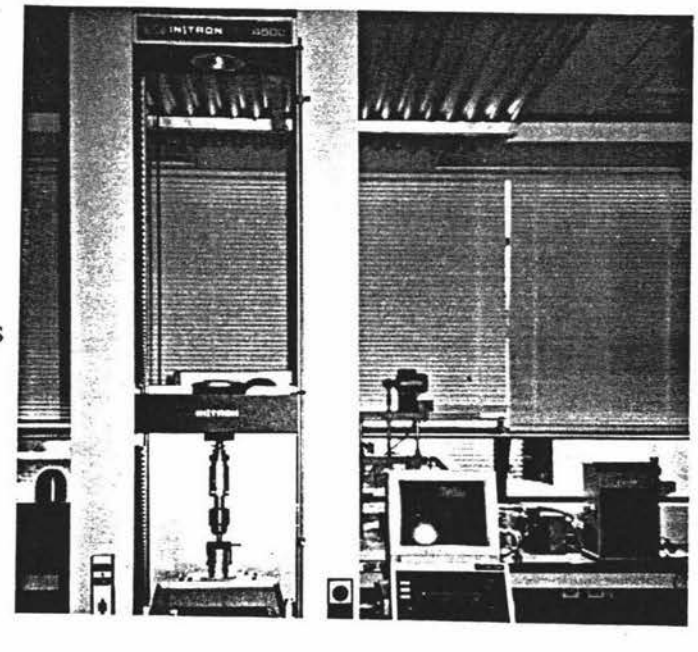


Fig 2 Intron Testing Instruments Series 4502

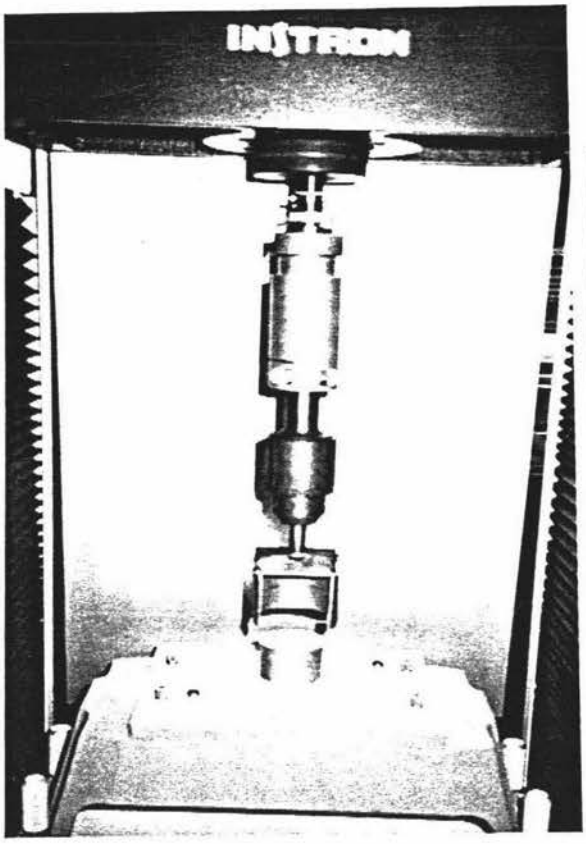


Fig 3 Sample Ring

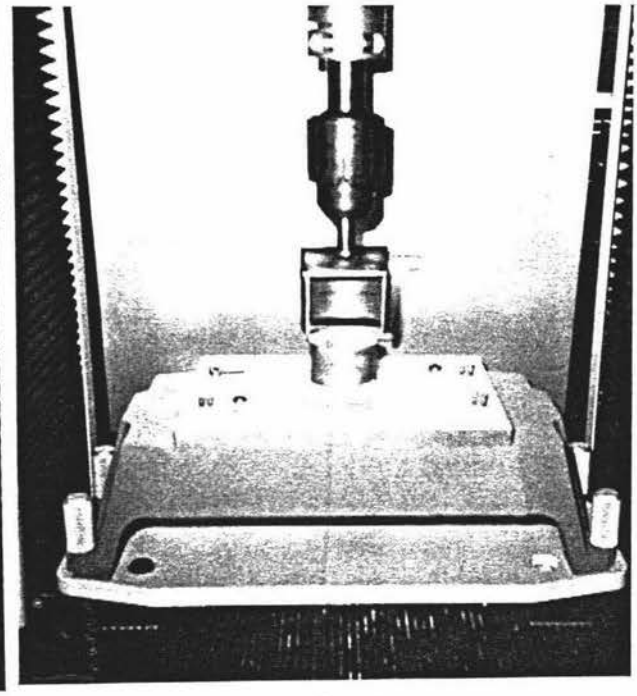


Fig 4 Wire Attachments

4.3.2 Material

Milkfat

All milkfat and its related fraction components were either from Bay Milk Products or Dairy Research Institute.

Polymer Microspheres

The dry polymer microspheres (Cat No. 444) supplied by Duke Scientific Corporation, California 94303, USA., have particle size ranges from 0.5-24μm. They are insoluble in water and have a specific gravity of 1.05.

All other agents used were of analytical grade.

4.4 Experimental

4.4.1 Sectility Hardness Measurement

Sectility testing measures the force required to cut through a sample (e.g. butter) with a wire of known diameter and length at a fixed cutting rate. The basic principle of this measurement is based on a FIRA/NIRO extruder (Prentice, 1954) and further modified as a sectility instrument by Taylor *et al*, 1971. Dixon, 1974 went further to describe ad compare the various methods of measuring the spreadability of butter.

The method used consists of a butter sample contained in a ring of 28mm high x 48mm dia. made from stainless steel tubing. Longitudinal slots were cut on opposite sides of the ring to allow the sample to be cut along the diameter by a stainless steel wire of 52mm long x 0.56mm dia. The cutting rate of the wire is fixed at 39.6mm min^{-1}.

Butter samples are usually tested at Day 1, 7 and 30. At Day 7, approximately 90% of the maximum hardness would have been reached (Taylor and Dolby, 1973). After the butter has been pressed into sample rings, the rings are then placed in a regulated waterbath at a temperature at which the samples are to be measured, usually at 10 ±1°C for a period of at least 5 hours and preferably overnight.

An Instron Testing Instrument Series 4502 is used to attach the stainless steel wire piece (Fig 3) and programmed to have a fixed crosshead or cutting rate as required and determining the force exerted by the wire on the butter sample (Fig 4) at a rate of 25 points per second. A typical graphic plot of force in gms versus the displacement is shown in Fig 5. The displacement is the distance at with the wire has travel downwards into the butter sample.

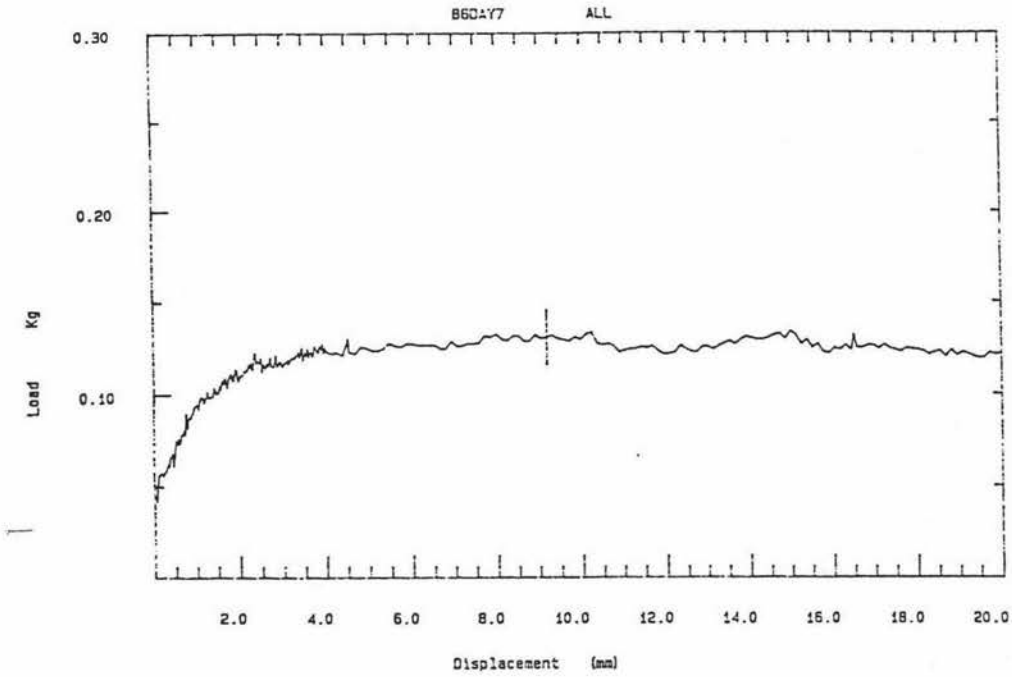


Fig 5 Instron Graphical Representation of Butter Sectility Hardness vs Displacement

4.4.2 Stand-Up and Oil-Off Test

These two tests provide an assessment of the ability of the butter to keep or maintain its shape at high ambient temperatures, Taylor *el at* 1977.

At a predetermined temperature, usually 22 ± 1°C, the Stand-Up or Slump test determine the ability of a plug of butter to retain its shape, and Oil-Off that determines the percentage of oil that drains out from the plug of butter after a period of 16 hours.

A cylindrical plug of butter measuring 25mm x 25mm made from a tube is placed on a 100mm x 100mm stainless steel gauge of 20/20 mesh. Four corners of the mesh are bent to make the platform base and this is in turn placed in a petri dish (Fig 6).
The stand-Up of butter is measured as the percentage increase in base diameter of the plug of butter.

The Oil-Off of butter is taken as the percent by weight of oil that collects in the petri dish.

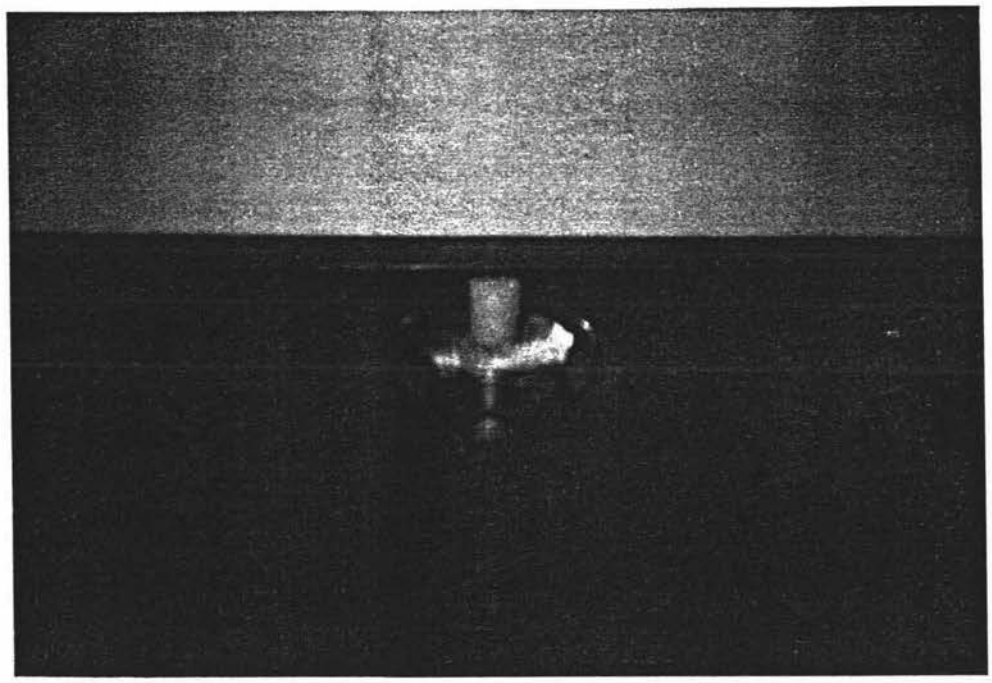


Fig 6 Picture Showing a Plug of Butter on a Wire Mesh in a Petri Dish

4.4.3 Moisture Content

A known weight of butter sample (18-20°C) is placed in a metal dish and kept in an oven at 102°C to constant weight. The dish is cooled in a desiccator and the weight of the sample is again determined. The loss in weight would be the moisture content.

Work carried out by Jebson, 1974 on the effect of moisture content on the hardness of butter conditioned for Day 1 and Day 7 showed that an increase in moisture of butter by 30% gave a corresponding decrease of only 8.6% and 6% respectively; and an increase in moisture of butter by 63% gave a decrease of hardness by 18%. The experiments concluded that the hardness of butter is inversely proportional to its moisture content in the range of 16-26% moisture, and a large increase in moisture content would be necessary to significantly make the butter softer.

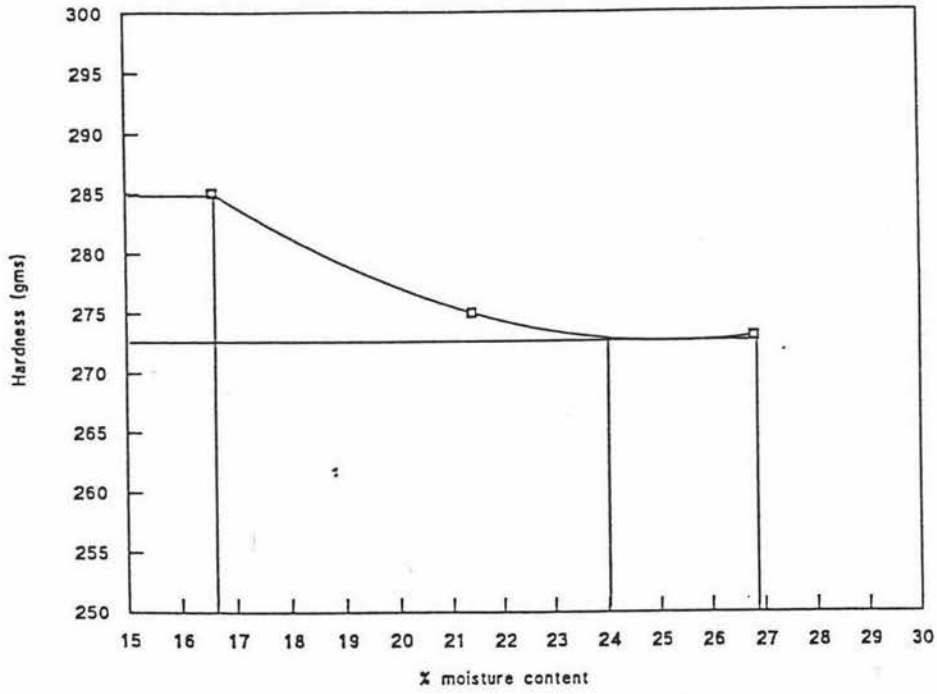


Fig 7 Effect of moisture Content on Hardness of Butter

On this basis, although in the work carried out here butters have varying moisture content and that no correction were made to take into account of the difference in moisture content. Hence, it is not necessary to make adjustment to hardness if the moisture content of the butter varies only slightly.

4.4.4 Fat Content

The Rose-Gottlieb method (Milk I.D.F. IA, 1969) was used for the determination of fat content. This is done by extracting the fat in the sample with an ammonia-alcohol solution (diethyl ether and petroleum ether), evaporation of the solvent, and weighing the fat residue. The function of the ammonia is to dissolve the protein of the milk and the alcohol to break down the fat-emulsion and the fat protein-complex.

4.4.5 Acetone Fractionation

Methods that would suit the fractionation of anhydrous milkfat were developed in the late 1960s and described in details by Norris *et al*, 1973. By far the most acceptable method would be to crystallise milkfat without additives or solvents at suitable temperature. Any benefits in the introduction of additives or solvents in the crystallisation process would be by and large overshadowed by the fact that the milkfat have been somewhat adulterated. After crystallisation over a set time, the fat fractions are separated out using filters, centrifuges or vacuum filters. Although this is a simple physical process, it is considered to be inefficient as the fractions are often not well defined components, the crystallized fraction containing a significant proportion of liquid.

4.4.5.1 Acetone fractionation Hard and soft Fractions (21°C) from BMP

One of the method under investigation in this report was to use acetone fractionation to produce milkfat fractions. This method, patented by Norris (1973), provides purer fat fractions and better fat crystallisation control. Furthermore, acetone fractionation can be used to crystallise fat of high viscosity at low relatively temperatures. The main disadvantage of this process is the use of chemical with the milkfat and subsequent removal of the chemical after the fractionation process is perceived to be unacceptable. The process is also not likely to be cost effective.

The hard and soft milkfat fractions fractionated at 21°C from Bay Milk Products(BMP) were used. To obtain a harder fraction from the hard fraction, the hard fraction was melted and than recrystallised at 40°C and filtered. Fractional crystallization with acetone solution was used on the soft fraction and filtered to produce a softer soft fraction for butter making.

Hard Fraction

Melted hard fraction in a container was placed in a waterbath maintained at a constant temperature of 40-41°C. The liquid hard fraction was stirred with a slow speed impeller

stirrer for 4-6 hours. The hard fat crystals was separated out from the liquid fat by filtration.

Soft Fraction

Melted soft fraction was added to acetone solution in equal proportions. The solution was put in a stainless steel container with glycol/water sealing (on lid) and kept in freezer at -10°C overnight. The solids were removed by straining and filtration. The acetone was removed from the liquid with a rotavapor at 50°C.

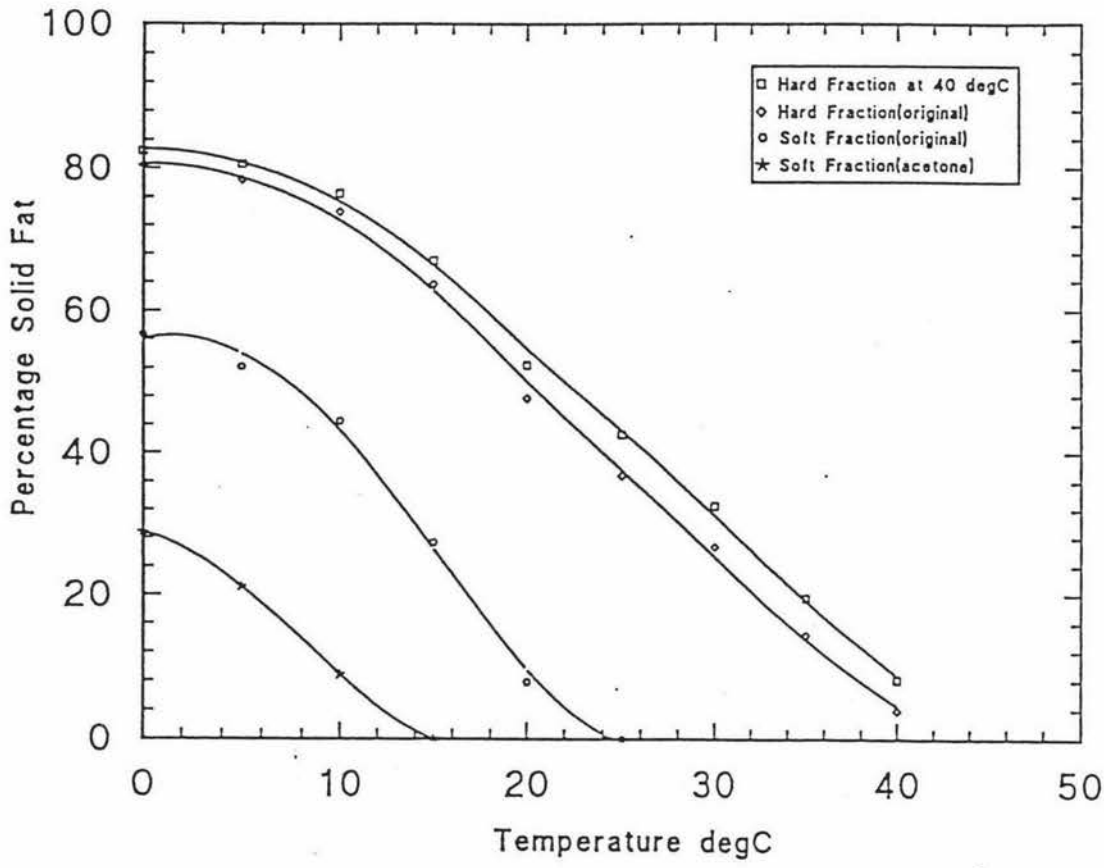


Fig 8 NMR Solid Fat Profile of the Original Hard and Soft Fractions After Acetone Fractionation Process.

The hard fraction obtained showed little improvement but a much softer fraction was achieved from the soft fraction and this compares favourably with the soft fraction from the multi stage crystallisation process (refer Chapter 1, Fig 4 and 5).

Although the temperature of crystallisation of the milkfat hard fraction crystals was set at 40-41°C, the process of filtering or separating the hard fat crystals was at ambient temperature (i.e. much lower temperature) resulting in very little removal of the lower melting point fat crystals.

Removal of acetone from soft fraction has had limited success. Normal evaporation under vacuum did not completely remove the last traces of the acetone from the fat fractions. This was overcome by using steam stripping in a glass tubes bedded column under vacuum, but this process introduced a certain amount of moisture to the product.

During the course of the work, it was thought that since milkfat fractions was available from the NZ Dairy board and their melting point characteristics are similar to fractions from acetone fractionation, it was decided to use the fat fractions from the NZ Dairy Board instead. Due to the confidential nature of the fractionation process the fraction yields and other sensitive data will not be reported.

4.5 Methods of Buttermaking

Having investigated and produced fat spherulites as described in Chapter 1 and 2, these fat spherulites were then used for buttermaking to find out its effects on the hardness and textural properties on butter.

4.5.1 Butter with Hard and Soft Fraction - Standard Method

The melted hard and liquid soft fractions were mixed with buttermilk powder in serum. The compositions by weight were 40% combined milkfat, 6% buttermilk powder and 54% water. The mixture was heated to approximately 55°C and homogenised at 1.70/1.38 x 10^3 kPas for stage I and II respectively and the homogenised sample conditioned at 10°C overnight before churning to make butter. This is termed "Standard Butter" for the purpose of comparison with butters made by the following methods. Using the same procedure, further experiments were carried out to determine the effect of homogenising pressures on butter hardness.

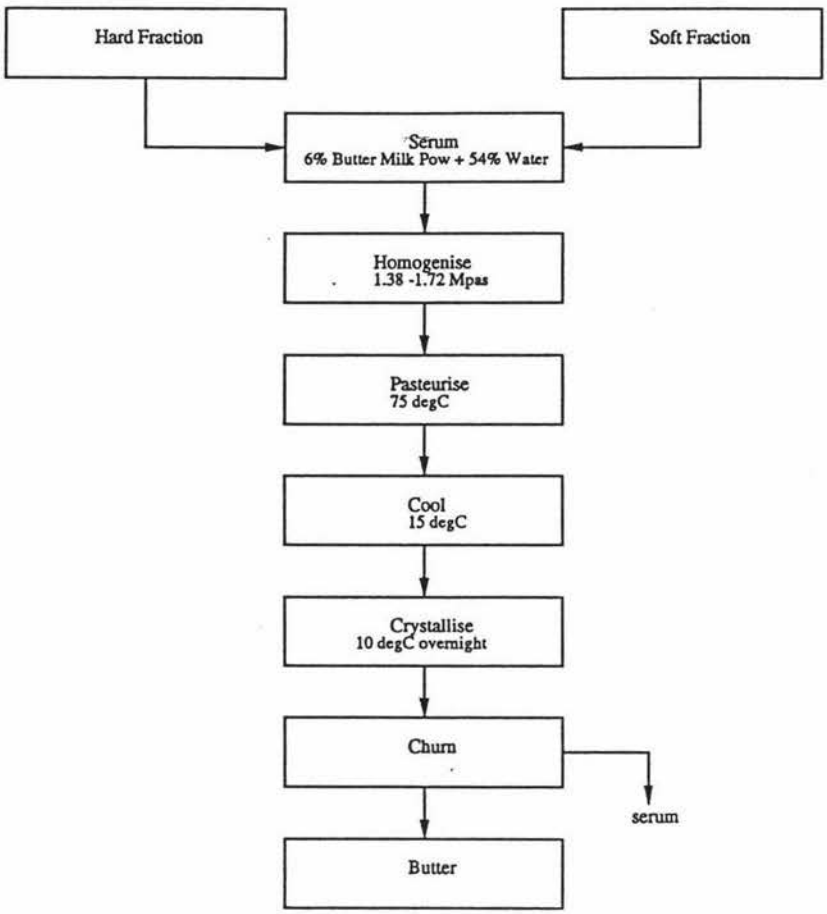


Fig 9 Standard Method of Buttermaking

4.5.1.1 Results and Discussion

It should be noted that the Standard Butter made in these trials were from milkfat hard and soft fractions mixed with water and homogenised to form milkfat cream prior to churning. No intermediate fraction was added. Table 1 showed the butter hardness at day 1 and 7 for the various hard fraction to total fat fraction. Butter hardness is directly proportional to the hard fraction in total fat fraction.

Table 1 Sectility Hardness of Butter

Hard Fraction to Total Fraction	Hardness Day 1	Hardness Day 7	% Moisture
0.25	124	179	15.83
0.35	270	294	16.48
0.50	462	570	16.60
0.75	1236	1976	16.08

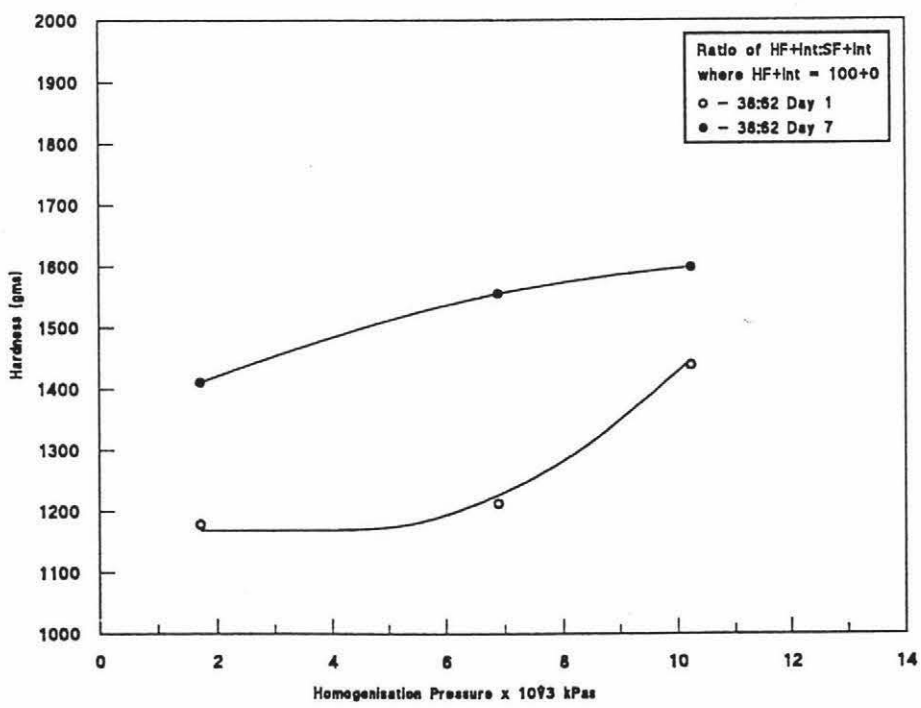


Fig 10 Effects of Homogenising Pressures on Butter Hardness

Fig 10 shows the effects of homogenising pressures on butter hardness using the standard buttermaking method. Increase in homogenisation pressure results in an increase in the butter hardness. This was expected as the increase in homogenisation pressure produced a thicker cream with fat globule size or spherulites distribution which are correspondingly smaller in sizes. With thicker cream the effects of clumping and gelling would be more pronounced and this would be important in a commercial process.

4.5.2 Butter from Filtered Hard Fraction Spherulites and Creamed Soft Fraction

The hard fraction fat spherulites was made by mixing the hard fraction with warm water to give 10% fat in the mixture. The mixture was then homogenised at 6.89/1.38 x 10^3 kPas for Stage I and II respectively. The emulsified sample collected was immediately chilled with iced water to a ratio of 1:1. The fat spherulites is filtered out in a buckner funnel under vacuum suction.

Melted soft fraction was mixed with buttermilk powder in serum, heated to 55°C and homogenised at between 0.35-0.69 x 10^3 kPas. After homogenisation, the emulsion was pasteurised (where necessary) at 72°C and then cooled slowly to 15°C. The cream was then conditioned at 10°C overnight, mixed with the hard fraction fat spherulites and churned to make butter.

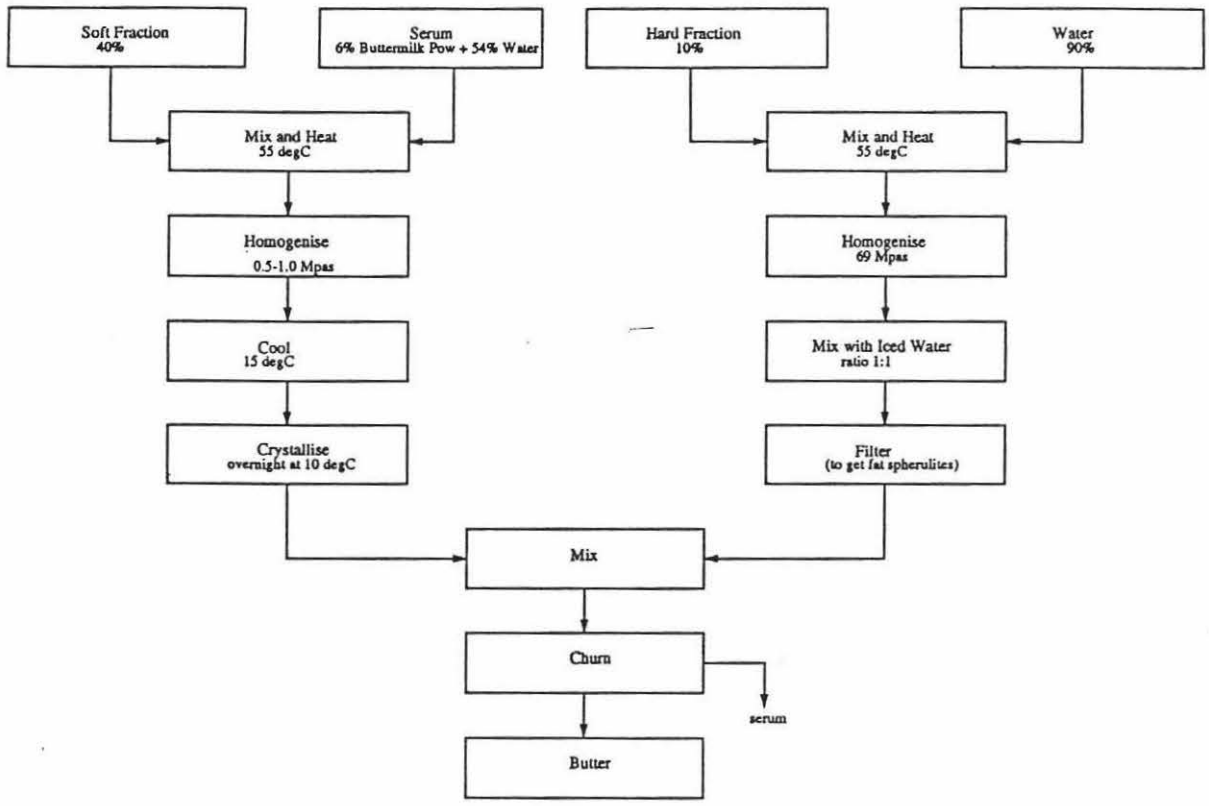


Fig 11 Buttermaking with Filtered Hard Fraction Spherulites and Creamed Soft Fraction

4.5.2.1 Results and Discussion

The filtered fat spherulites were lumpy and sticky. It was apparent that the sample probably constituted an amalgamation of pure fat spherulites with water molecules within the free space matrix. This was to be expected as there was no stabilising agents to provide a coating of the fat spherulites. This fat could not be blended with the soft fraction cream during the churning process. No effort was made to determine the sectility hardness of the butter made from this process.

The experiment was useful in that it confirmed the need to have some form of surface active agent or emulsifier to stabilise the fat spherulites in emulsion. Secondly, the necessity to concentrate the stable fat spherulites so that this could be mixed with the soft fat fraction to make butter.

4.5.3 Butter with Freeze Dried Hard Fraction Cream and Crystallised Soft Fraction

A hard fraction was creamed, homogenised, and the cream frozen and freeze dried. This was then mixed with crystallised soft fraction emulsion and churned to make butter. The temperature was maintained at 10°C during the churning process.

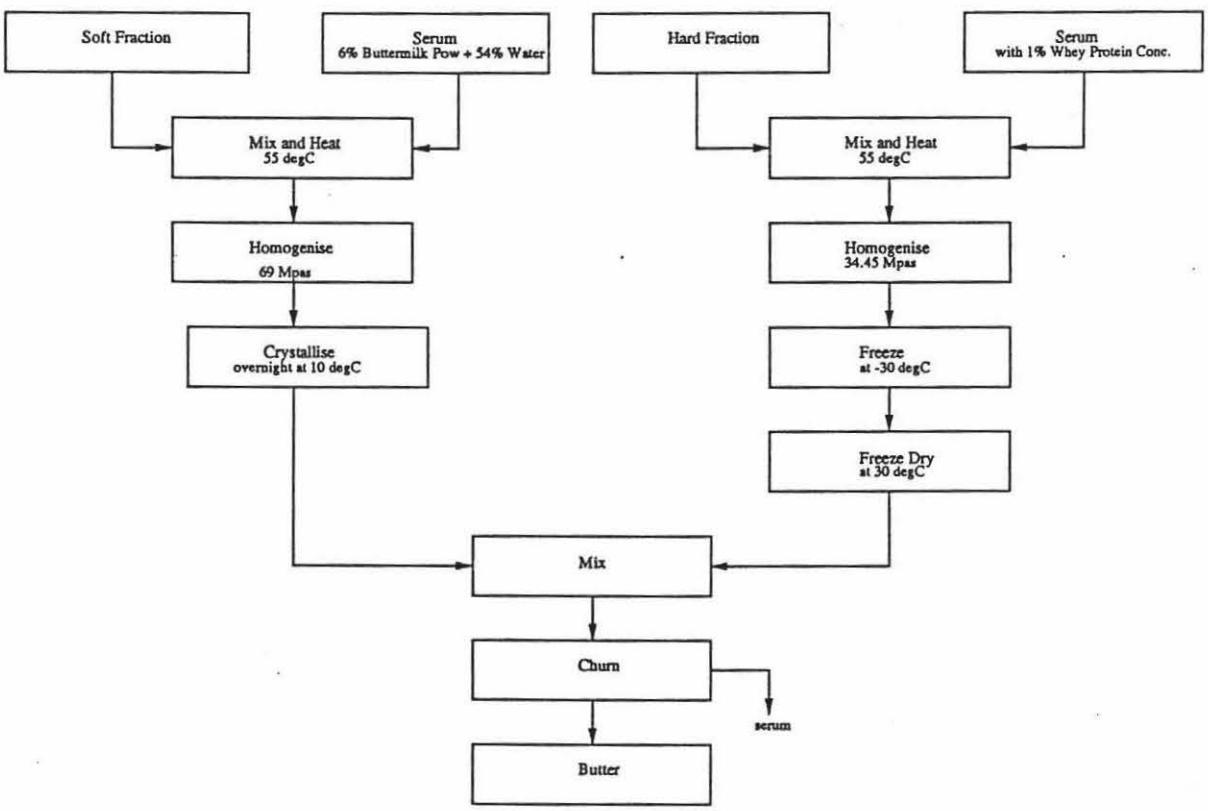


Fig 12 Buttermaking with Freeze Dry Creamed Hard Fraction and Creamed Soft Fraction

4.5.3.1 Results and Discussion

The freeze dried powder produced was flaky in texture with an oily surface. It appeared that the act of freezing had disrupted the milkfat globules, causing leakage of milkfat and making the powder unsuitable for butter making. During churning, the mixture was lumpy and the powder could not be mixed properly. No attempt was made to determine the hardness of the sample.

4.5.4 Buttermaking with Polymer Microspheres

The purpose of using polymer microspheres was to confirm whether the "butter" made with a proportion of fat spherulites (in this case, polymer microspheres) in solid spherical forms would be softer than "standard butter" and to determine the extent of relative "softness".

The microspheres came in a powdered form which also contained a percentage of polystyrene divinylbenzene and polymethymethacrylate. They were washed with acetone solution, filtered and dried before using. The diameter of the microspheres ranges from 0.5-24μm. This size range (Hartel, R., personal communication) was selected on the basis that they were sufficiently small not to be detected by mouth feel. Most fat spreads are fat continuous, with the dispersed aqueous phase droplets, 2-60μm or more in diameter.

Chilled water was blended with liquid soft fraction at 6°C. A small quantity of microspheres was then slowly added and mixed well to give a final product having 16% moisture content. The samples are placed in rings, conditioned at 10°C overnight, and sectility hardness test carried out using the Instron testing instrument.

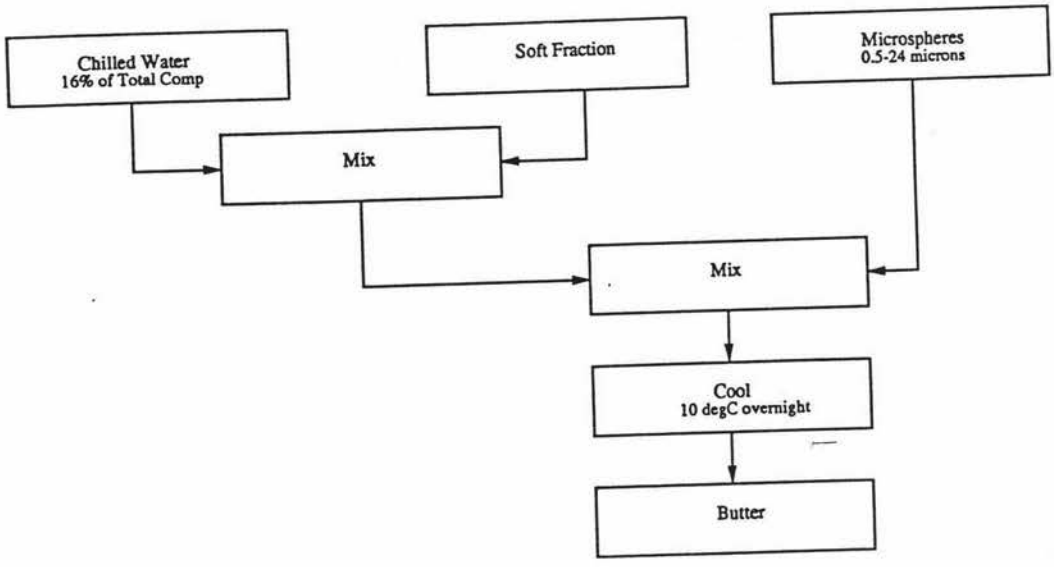


Fig 13 Buttermaking with Polymer Microspheres

4.5.4.1 Results and Discussion

Table 2 shows the comparative sectility hardness of the product after day 1 in relation to the proportion of microspheres used.

Table 2 Sectility Hardness of Butter from Microspheres

Microspheres to Total Fraction	Hardness(gms) at Day 1	% Moisture Content
0.25	87.00	16.00
0.35	130.00	16.00
0.75	298.00	16.00

Where the proportion of microspheres was in the range of 25% to the total fractions, there was no significant difference in hardness from that of other types of butter (refer Fig 31). As the microspheres proportion was increased, the corresponding increases in hardness became more apparent, and the "butter" was lumpy. The lumpiness was most

likely due to the high microspheres content and difficulties in obtaining a consistent mix. The experiment confirmed that if the hard fat fraction could be made into solid fat spherulites akin to polymer microspheres, then the butter would be softer.

4.5.5 Buttermaking with Hard Fraction Crystallised Cream and Crystallised Soft Fraction

In this experiment, 40% fat in buttermilk serum, similar to percentage fat in cream in the conventional method of butter making, was made up from hard fraction cream and crystallised soft fraction. The 40% fat content was made up of 10% hard fraction and 30% soft fraction. The hard fraction in serum was heated to 55°C and then creamed in a homogeniser and cooled to 10°C. For the soft fraction, it was first melted and then crystallised at 10°C overnight. Both these were mixed and churned to make butter.

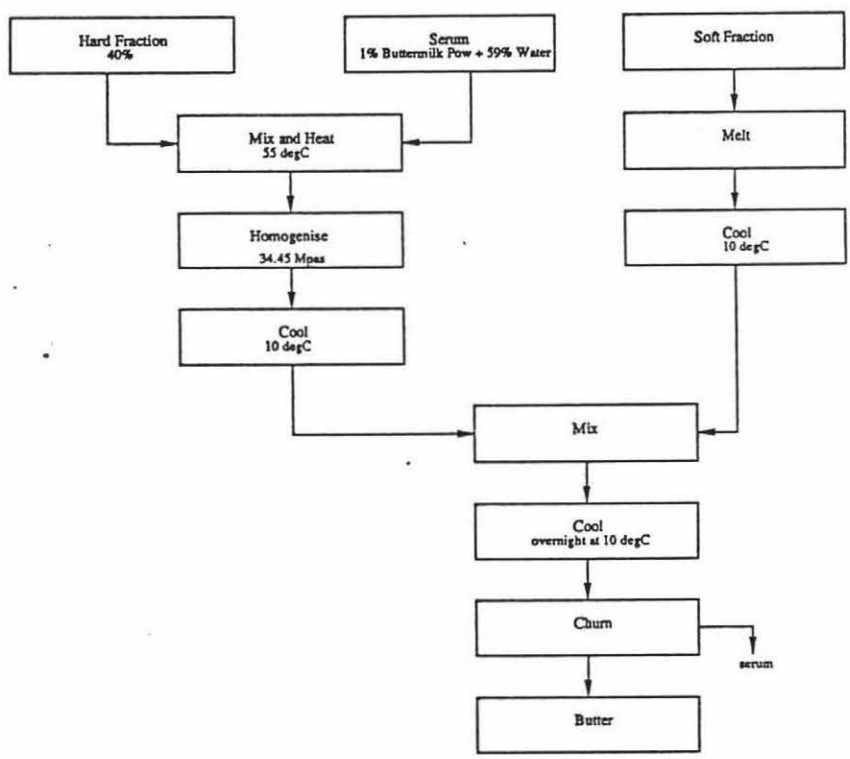


Fig 14 Buttermaking with Hard Fraction Crystallised Cream and Soft Fraction

4.5.5.1 Results and Discussion

Although there was some degree of phase inversion and serum removal during churning,

the butter was soft and leaky in texture. The serum could not be completely removed and prolonged churning increased the proportion of liquid fat due to increase in temperature. It would appear that reducing the churning temperature may improve the churning process but the butter would still be leaky. This being the case, no further tests and experiments were carried using this method.

4.5.6 Buttermaking with High Fat Creamed Hard Fraction and Soft Fraction with No Serum Removal

In this experiment, a proportion of hard fraction was creamed with buttermilk powder in serum, homogenised, and the cream mixed with crystallised soft fraction in a proportion that will yield a butter with 16% moisture after churning with no removal of water in serum. Relatively low homogenisation pressure was used as the percentage of fat to total mixture is high at 50%.

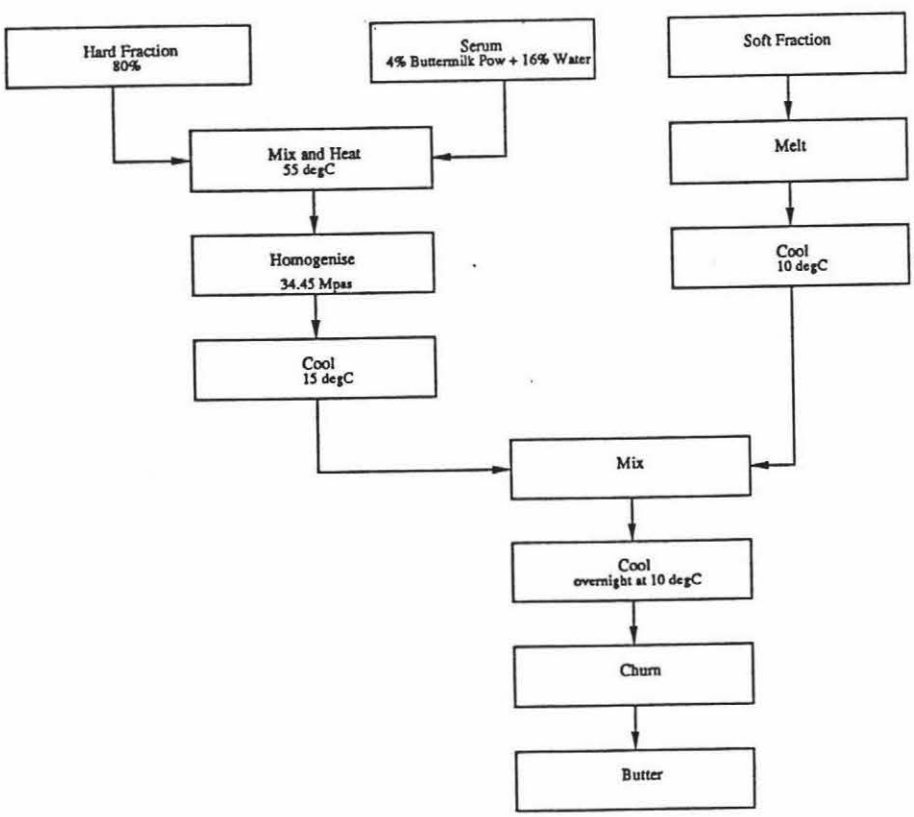


Fig 15 Buttermaking with No Serum Removal

4.5.6.1 Results and Discussion

The homogenised cream was quite viscous and the viscosity increases with decrease in temperature. There were difficulties in obtaining a consistent mix from the cream and the soft fraction. On conditioning at 10°C overnight prior to churning, the mixture was quite hard and difficult to churn. The churned butter was grainy in texture and somewhat lumpy. Although sectility hardness test on the samples indicated that the hardness are up to 50% lower than the standard butter with the same fat fraction compositions, no further test was carried as the butter produced had poor textural properties.

4.5.7 Buttermaking with Spray Dried High Fat Hard Fraction and Soft Fraction in Serum

The rational of the experiment was to confirm the postulation that the butter from butter powder in spherulites would be more spreadable than the normal needle to platelet shape fat crystals matrix.

In Chapter 4 Section 2 , the production of high fat powder from spray drying was reported. Milkfat suitably mixed with a proportion of emulsifier, homogenised and spray dried will produced a butter powder of up to a maximum of 80% fat content. In this method of buttermaking, milkfat hard fraction was spray dried to butter powder and this powder was then mixed with soft fraction in serum and churned to make butter.

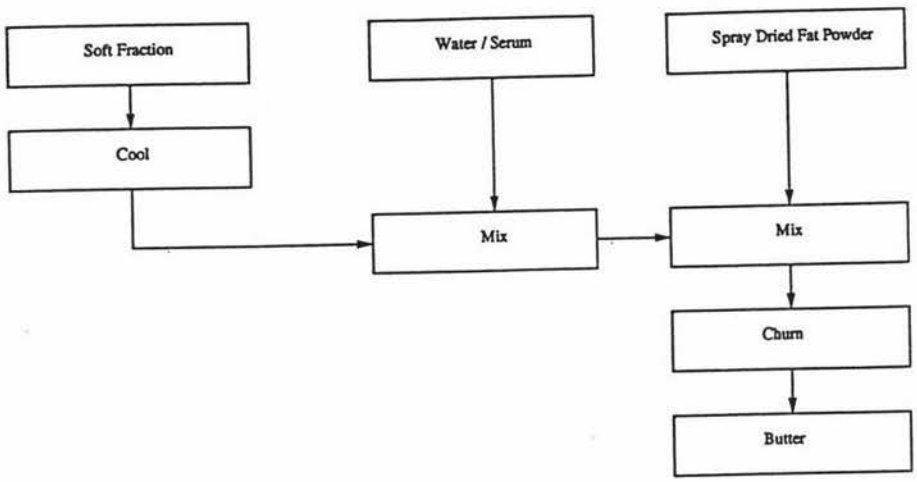


Fig 16 Buttermaking with Spray Dried High Fat Hard Fraction and Soft Fraction

4.5.7.1 Results and Discussion

As expected, butter hardness increases with increase in the percentage of hard fraction used for butter making as shown in Table 3. Hardness tests carried out after Day 7 showed an increase in hardness compared to tests done on Day 1 from 10 to 27%. The comparatively higher percentage of 27% between Day 1 and Day 7 results for milkfat fraction ratio of 25:75 may have been due to preparation of samples. The remaining samples showed an increase in hardness ranges from 10 - 16%. Day 30 tests will be carried out in due course but the increase in hardness are not expected to much higher than Day 7 results. Although the butter hardness has been reduced considerably from that of normal commercial butter, it has a somewhat grainy texture. It is possible that the range of sizes of the spherulites hard fraction powder was too big.

With the use of an emulsifying agent in the hard fraction powder, the percentage of solids not fat was significantly reduced. However, this is still higher than the percentage of solids not fat in normal commercial butter.

Table 3 Sectility Hardness Test on Butter

Fat Frac Ratio* HF:SF	Hard Frac to Total Frac	% Fat	% Whey Protn Conc	% Solids Not Fat	% Water set	% Water act	Hardness(gms) Day 1	Hardness(gms) Day 7	Hardness(gms) Day 30
Soft Frac	0.00	100.00	-	-	-	-	62	-	-
25:75	0.25	79.73	0.64	3.36	16	15.83	103	131	142
35:65	0.35	78.14	0.88	4.98	16	16.48	104	115	125
50:50	0.50	75.86	1.23	6.91	16	16.60	122	139	146
75:25	0.75	72.37	1.75	9.88	16	16.08	479	559	589

* From analysis, the hard fraction powder constitute the following compositions:

Milkfat	= 82.35%
Whey Protein Concentrate	= 2.66%
Solids Not Fat	= 14.99%

Fig 17 shows a graph of sectility hardness test of butter taken at day 7; conditioned at 10°C, using the Instron Testing Instrument. The load refers to the sectility hardness in kg and the displacement is the depth of the cross sectional wire that was forced down the butter sample. Plot 1 corresponds to 0.25 hard fraction to total fat fraction and Plot 4 to 0.75 of hard fraction to total fat fraction.

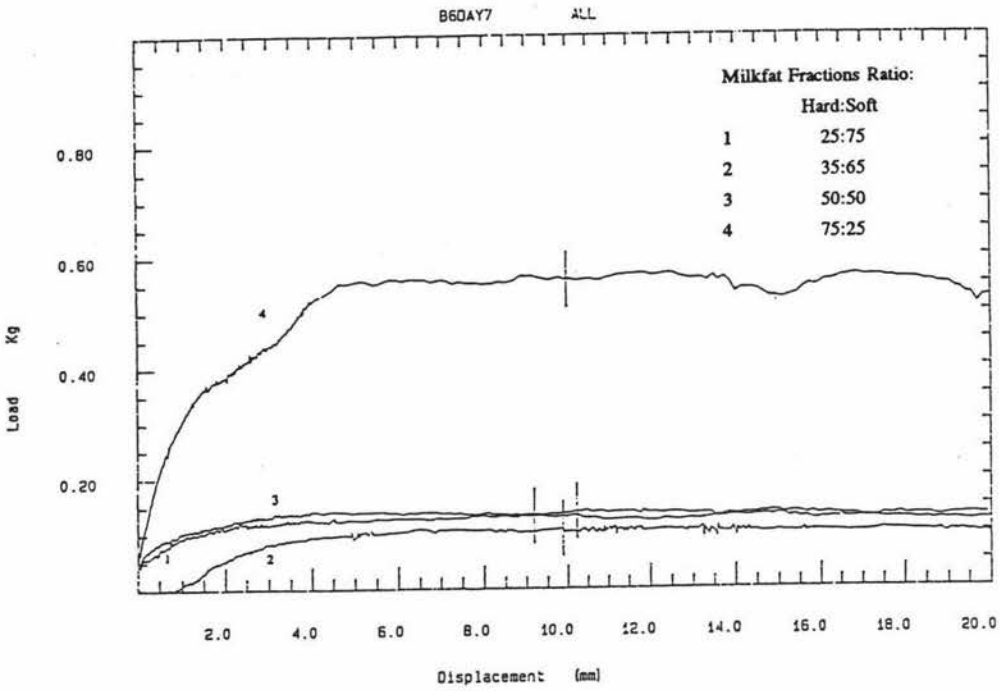


Fig 17 Instron Sectility Hardness Test on Butter (Load vs Displacement)

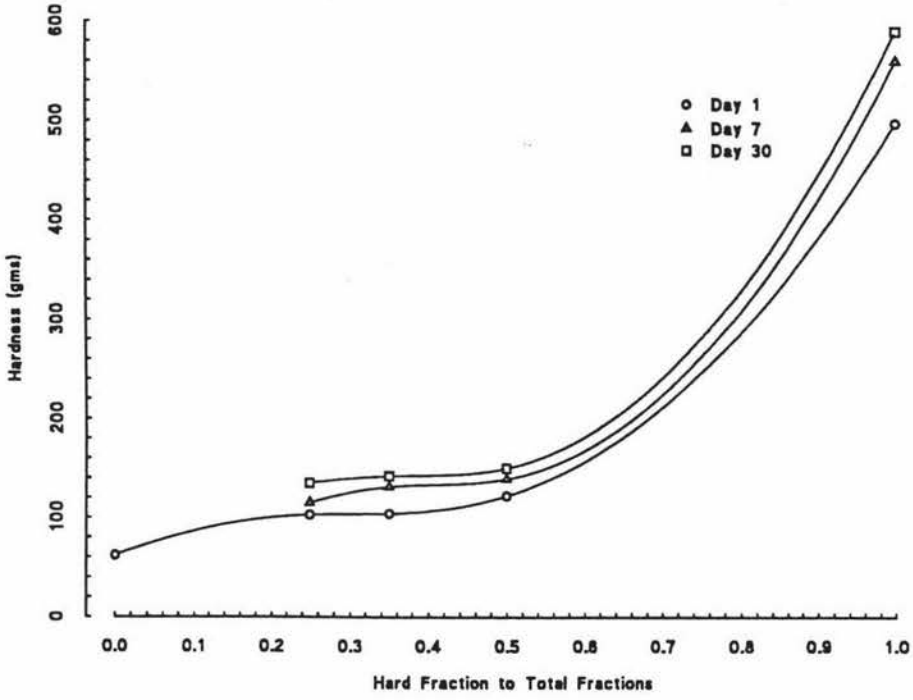


Fig 18 Butter Hardness versus % Hard Fat Fraction to Total Fat Fraction Used at Day 1, 7, and 30

Fig 18 shows the percentage of hard fat fraction to total fat fractions plotted against the butter hardness.

Table 4 shows the results of the Stand-Up and Oil-Off test of the butter samples. Earlier tests on commercial margarine under the same conditions showed it to be readily spreadable, having a hardness of approximately 70 gms (tested at 10°C), and no slumping or oiling off.

Table 4 Stand-Up and Oil-off Tests for Butter

Fat Frac Ratio* HF:SF	Hard Frac to Total Frac	Stand-Up	Oil-Off
25:75	0.25	91.67	71.01
35:65	0.35	70.83	79.58
50:50	0.50	33.33	95.46
75:25	0.75	0.00	100.00

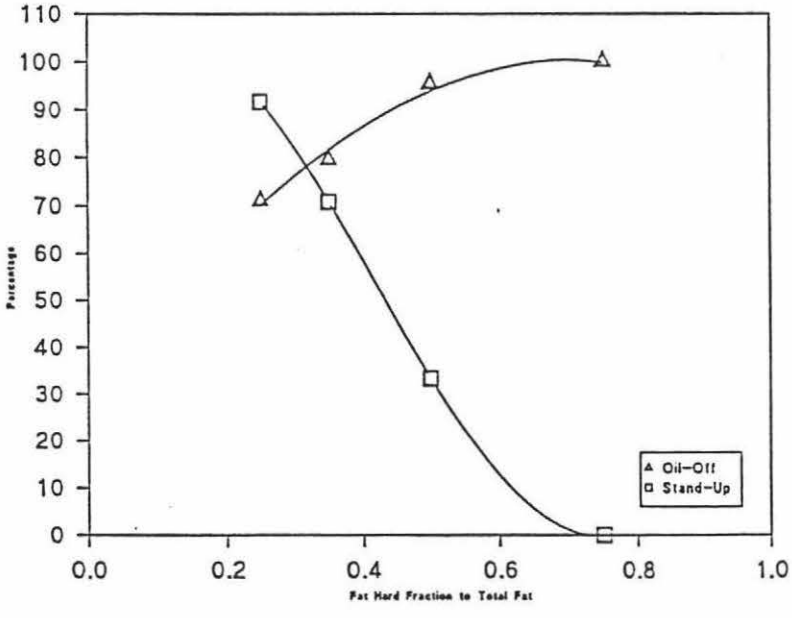


Fig 19 Stand-Up and Oil-Off of Butter

Where the percentage of hard fraction was low in the butter samples, severe slumping occurred and this was accompanied by the separation of free oil. However, the slumping and oiling off improved as the percentage of hard fraction increased in the butter composition. No slumping or oiling off occurred for fat hard and soft fraction ratio of

75:25. These findings suggested that for butter made from milkfat to be spreadable at the desired temperatures, a portion of the triglyceride fraction of milkfat which melts at these desired temperatures will need to be largely removed. Another possible option would be to combine fat spherulites with normal crystallised milkfat for butter making.

4.5.8 Buttermaking with High Fat Creamed Hard Fraction and Soft Fraction

Melted hard fraction was mixed with buttermilk powder in serum, heated to 55°C and homogenised at pressures ranging from 0.34/3.45 x10^3 kPas. The emulsion was then heated to 75-80°C and fed to an Alfa Laval cream separator (Model 103AE) to obtain a high fat cream.

The high fat cream was cooled to between 20-25°C and mixed well with the crystallised soft fraction. The mixture is kept overnight at 8-12°C and churned to make butter the next day. No intermediate fat fraction was used in these experiments.

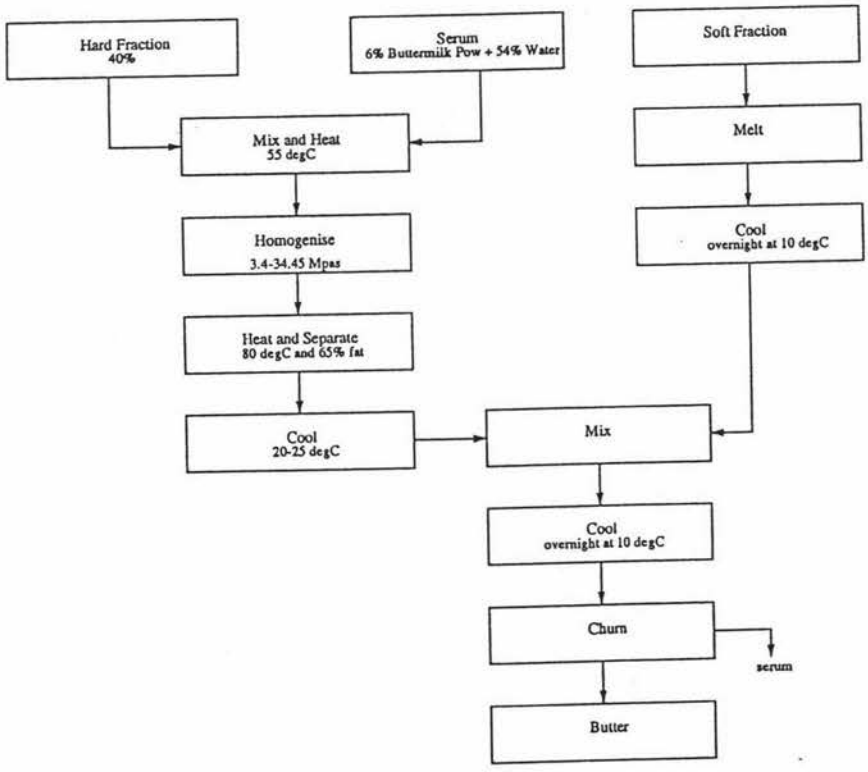


Fig 20 Buttermaking with High Fat Creamed Hard Fraction and Soft Fraction

4.5.8.1 Results and Discussion

Fat spherulites were produced when milkfat in serum was subjected to homogenisation. The use of emulsifiers helps to retain the stability of the fat spherulites in emulsion. Preliminary trials indicated that 40% fat in serum was the optimum for the Rannie homogeniser to prevent overloading. As a higher percentage was needed to avoid overmoist butter, the homogenised cream was further separated to increase the milkfat content. Initially, a Sorvall RC-5C Automatic Refrigerated Centrifuge was used to concentrate the homogenised cream. The cream produced was relatively solid in texture (due to low centrifuging temperature) and could not be blended consistently with the soft fraction. The butter made from this cream was generally lumpy. A smoother cream was obtained by using an Alfa Laval cream separator; the optimum fat content in the concentrated cream achieved was between 60-65%. It was necessary to heat the homogenised cream to 70-75°C to produce a good separation in the cream separator.
In churning butter from hard fraction cream and soft fraction, the temperature of the fat fractions prior to churning is important. Hard fraction cream of 65% fat content was quite viscous below 25°C and continued to harden on further cooling. When this hard fraction was mixed/churned with liquid soft fraction (at 6-8°C), the resulting mixture temperature was higher than the normal requirement for good churning. The butter produced in this manner was "leaky" in texture, and no phase inversion was observed during churning. The reason for this could be that the churning temperature of the cream hard fraction and soft fraction was in the range of 15 - 18°C. However, hardness of these butters have much lower values compared to that of butter made with the same compositions, but cooled overnight at 6-8°C before churning. The overnight cooling gave opportunity for further crystallization to occur.

Table 5 shows the butter hardness at Day 1 and 7 for the various hard fraction to total fat fraction. As expected, butter hardness is directly proportional to the hard fraction in total fat fraction.

Table 5 Sectility Hardness of Butter

Hard Frac to Total Frac	Hardness Day 1	Hardness Day 7	% Moisture
0.33	81	120	16.67
0.38	149	180	17.50
0.58	303	342	25.72
0.68	420	538	24.94
0.76	715	765	23.38
0.81	1413	1520	16.97

Table 6 Stand-Up and Oil-Off

Hard Frac to Total Frac	% Stand-Up	% Oil-Off
0.33	54.17	9.63
0.38	8.33	100.00
0.48	.00	100.00
0.65	.00	100.00

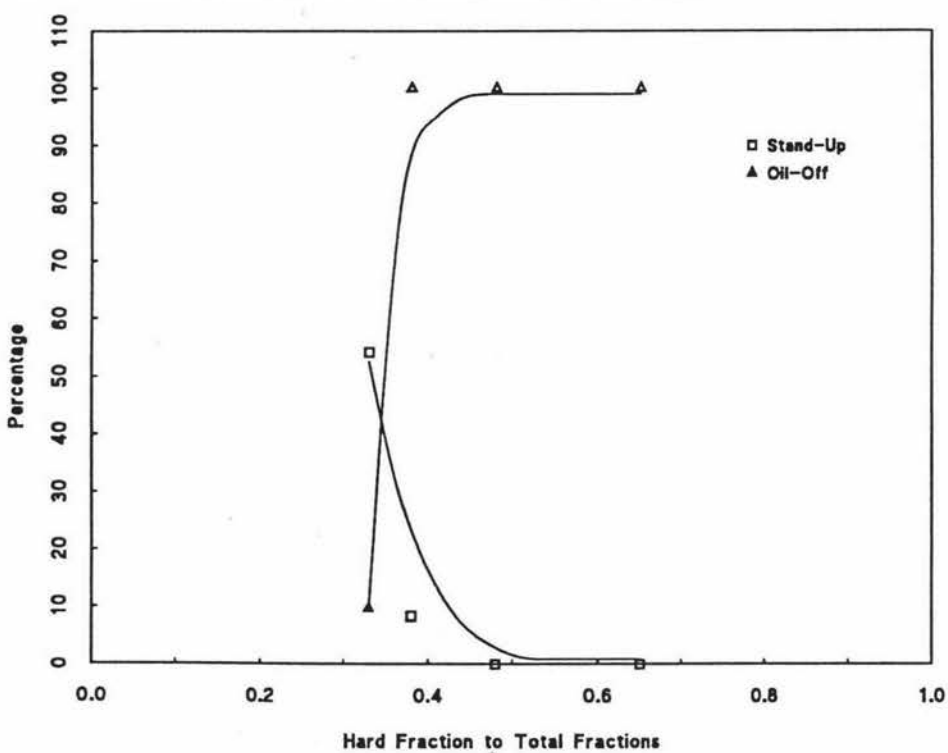


Fig 21 Stand-Up and Oil-Off Test on Butter Samples

The stand-up or slump test and Oil-Off tests were carried out on the butters, and the results are shown in Table 6. Fig 21 shows the graphical representation. The results indicated that the use of 50% hard in the total fat fraction would produced a butter with good stand-up and oil-off properties.

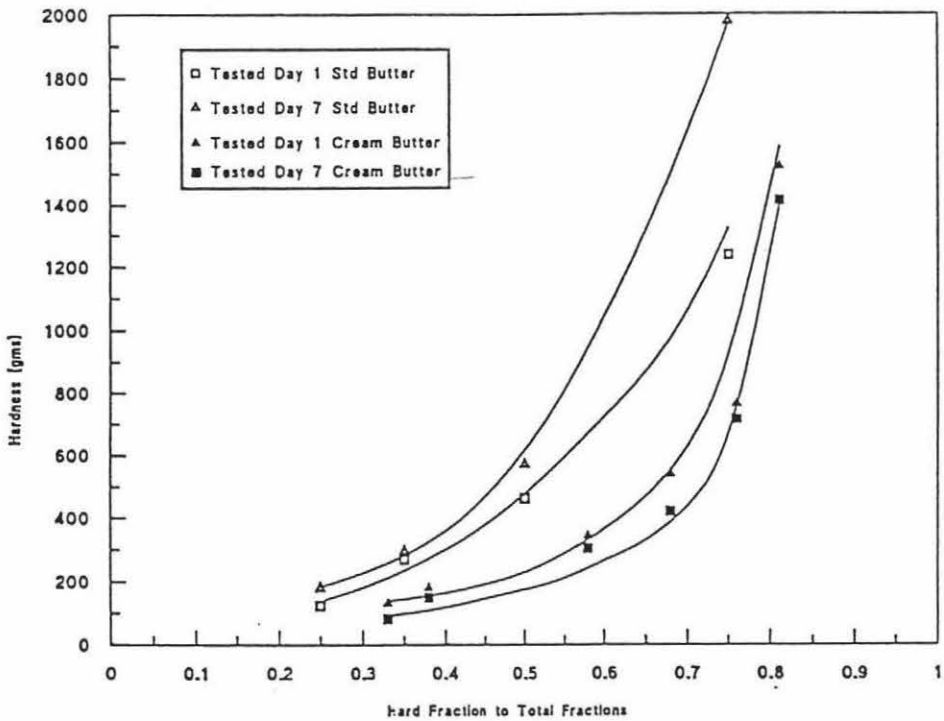


Fig 22 Comparison of Sectility Butter Hardness of Normal and Creamed High Fat Hard Fraction

Using the same fat fractions, Fig 10 shows that there were significant differences in butter hardness between the normal buttermaking and that of butter made by the high fat creamed hard fraction and soft fraction (as described in Section 3.4.8).

4.5.9 Buttermaking with Creamed Hard/Intermediate Fractions and Soft/Intermediate Fractions

The method used is similar to the Section 3.4.8 - Buttermaking with High Fat Creamed Hard Fraction and Soft Fraction, except that a proportions of intermediate fat fraction

were added to both the hard and soft fractions. The solid fat profile of the intermediate fraction is shown in Fig 7. In relation to the above, further trials were carried out the effect of butter hardness by using 100% creamed hard fat fraction mixed with the two intermediate fat fractions, namely, Intermediate I ("SH") and Intermediate II ("SSH").

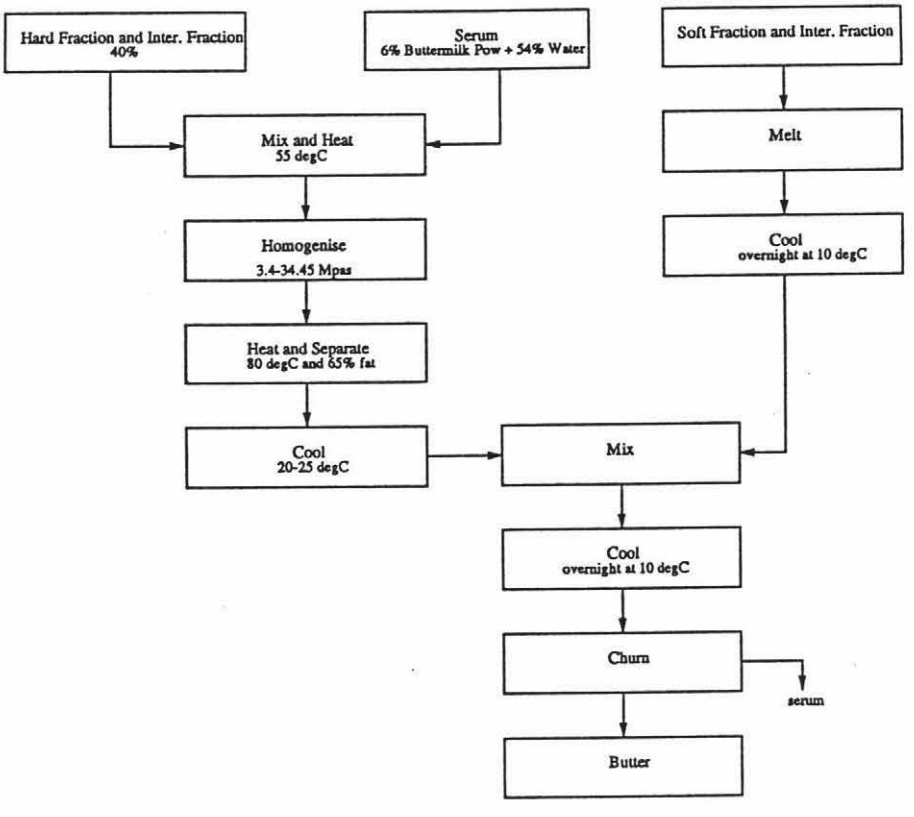


Fig 23 Buttermaking with High Fat Creamed Hard/Intermediate Fractions and Soft/Intermediate Fractions

4.5.9.1 Results and Discussion

Table 7 shows the results of the trials by varying and combining the various fat fractions. Sectility hardness tests were carried out at Day 1 and 7. Stand-up and Oil-off tests could not be determined for trial nos. 1,5 and 9 as the samples were too soft.

Table 7 Sectility Hardness of Butter

No	Composition of HF+Int:SF+Int	Fractions Ratio	Hardness(gms) Day1	Day 7	% Moist	% Stand-Up	% Oil-Off
1	100+0:100+0	37:63	30	57	19.66	-	-
2	100+0:90+100	37:63	140	217	19.89	0	100
3	100+0:75+25	37:63	218	327	18.84	0	100
4	100+0:50+50	37:63	593	800	17.40	0	100
5	90+10:100+0	35:65	58	88	17.11	-	-
6	90+10:90+10	35:65	108	163	17.47	0	100
7	90+10:72+25	35:65	204	278	16.90	0	100
8	90+10:50+50	35:65	421	550	16.51	0	100
9	75+25:100+0	36:64	30	39	20.05	-	-
10	75+25:90+10	36:64	128	207	17.52	0	100
11	75+25:75+25	36:64	295	460	17.22	0	100
12	75+25:50+50	36:64	527	684	14.58	0	100
13	50+50:100+0	36:64	35	71	18.43	45.83	1.09
14	50+50:90+10	36:64	77	153	18.24	12.50	18.24
15	50+50:75+25	36:64	178	320	15.65	8.33	15.65
16	50+50:50+50	36:64	455	644	15.10	4.17	100

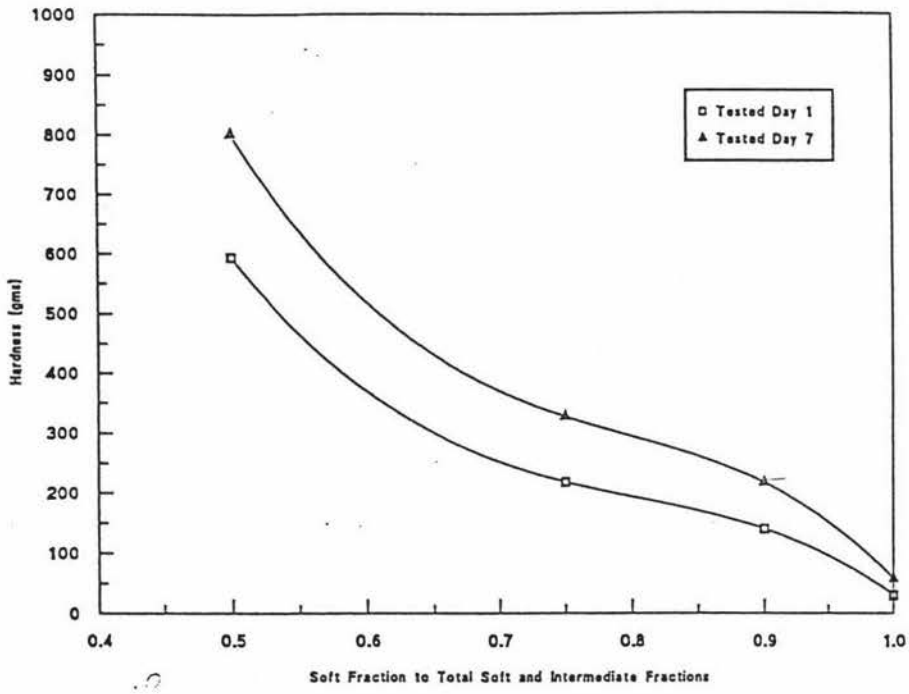


Fig 24 Sectility Hardness on Butter from Hard, Intermediate and Soft Fractions with HF + Int = 100 + 0 (37:63)

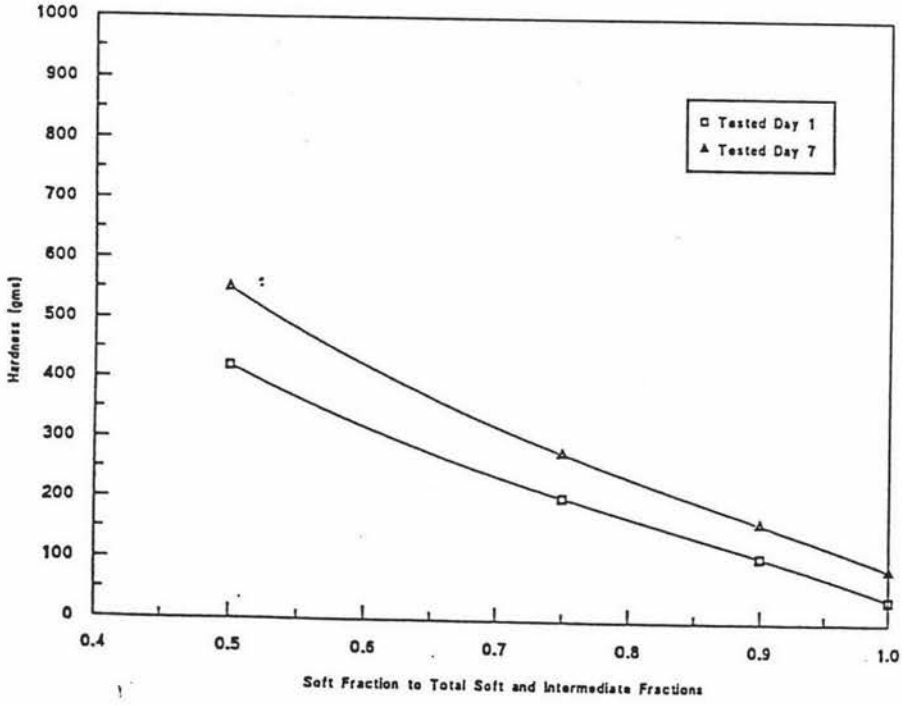


Fig 25 Sectility Hardness on Butter from Hard, Intermediate and Soft Fractions with HF + Int = 90 + 10 (38:62)

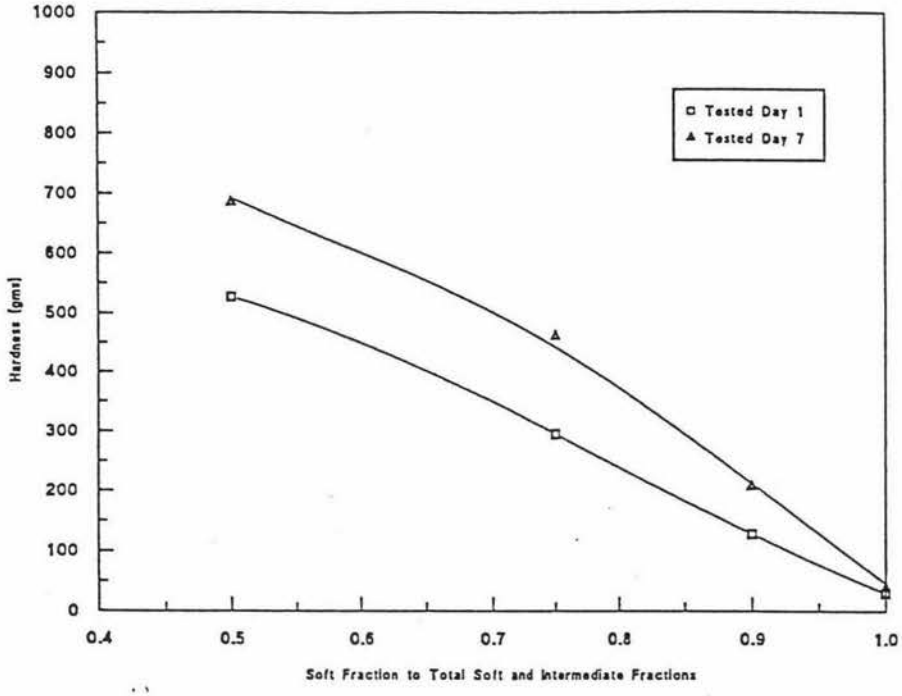


Fig 26 Sectility Hardness on Butter from Hard, Intermediate and Soft Fractions with HF + Int = 75 + 25 (36:64)

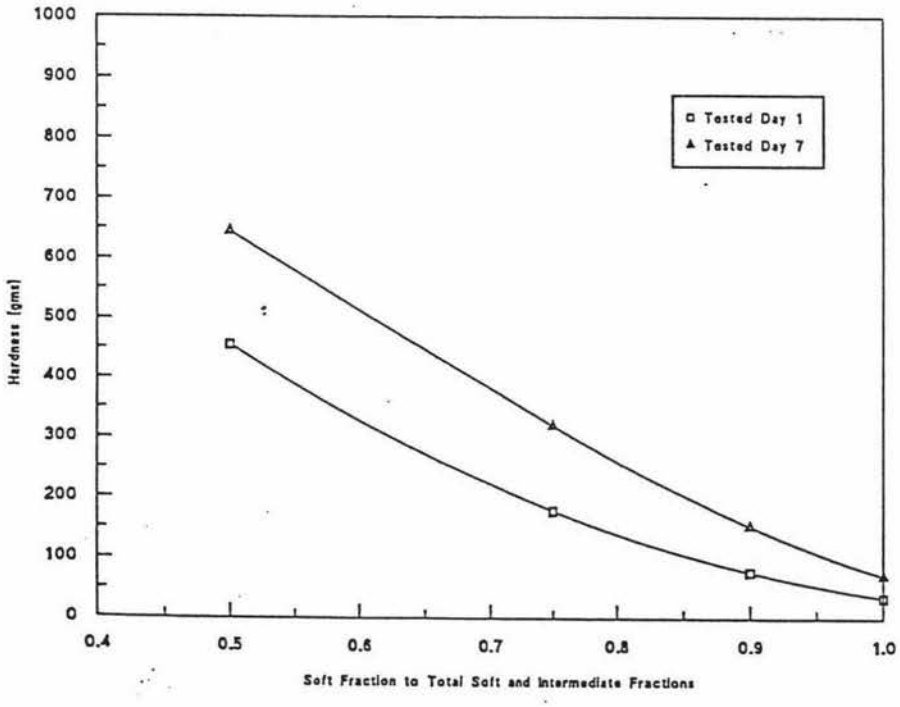


Fig 27 Sectility Hardness on Butter from Hard, Intermediate and Soft Fractions with HF + Int = 50 + 50 (35:65)

When intermediate fractions replaced a portion of the hard fraction there was slight little effect on the butter hardness, although at 50:50 soft:intermediate fractions there is some change in hardness with increasing proportion of hard fraction in the fat globules, the total of intermediate and hard fraction being the deciding factor (Fig 28). There is some scatter in the hardness values shown, but no significant difference in the slopes of the lines.

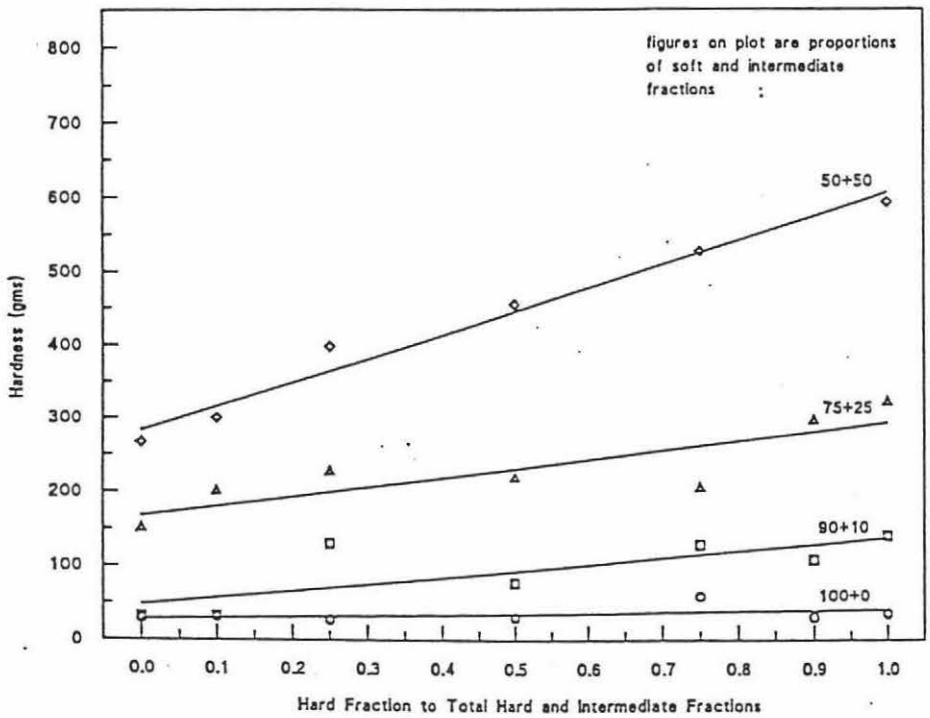


Fig 28 Effect of Hard and Intermediate Creamed Fat Fractions on Butter Hardness

However, the addition of intermediate fraction to the soft fraction caused the butter to be much harder. Mixed with the hard fraction the intermediate fraction did not appear to significantly weaken the globule strength, which is expected as at testing temperature most of the intermediate fraction would be solid. Equally important it appeared to be sufficiently "locked in" to the hard fraction globule that it played no part in setting, and the butter hardness did not change appreciably with time.

On the other hand, as the intermediate fraction is solid at the testing temperature, when added to the soft fraction it forms a crystalline matrix in the soft fraction and increases the butter hardness.

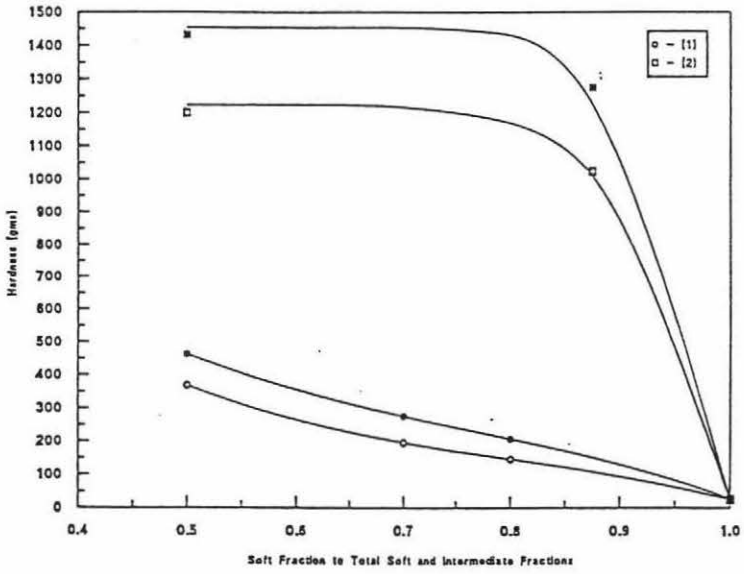


Fig 29 Sectility Hardness on Butter from Milkfat Fractions

HF + Int. = 100 + 0 Ratio (HF + Int.):(SF + Int.) - 40:60 (1)

Ratio (HF + Int.):(SF + Int.) - 43:57 (1)

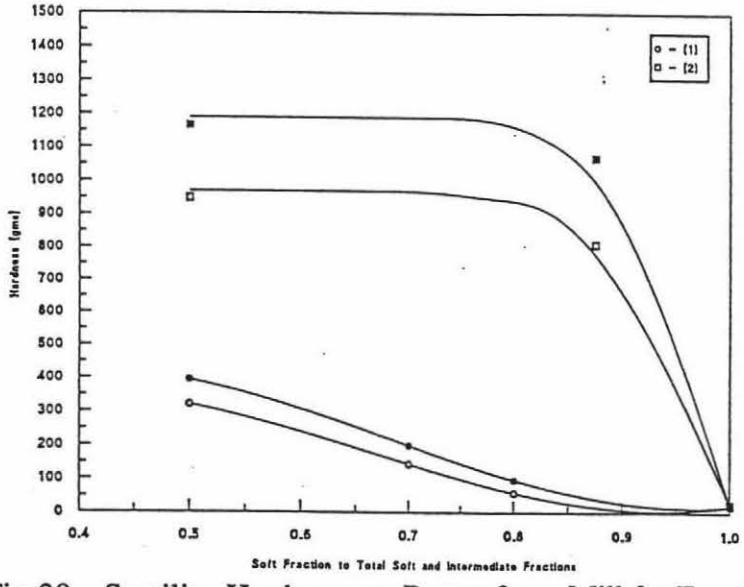


Fig 30 Sectility Hardness on Butter from Milkfat Fractions

HF + Int. = 100 + 0 Ratio (HF + Int.):(SF + Int.) - 40:60 (1)

Ratio (HF + Int.):(SF + Int.) - 43:57 (1)

Fig 29 and 30 shows the comparison of the variations in butter hardness using intermediate fractions('SSH' and 'SH') in mixed fat fractions. It is clear that there is significant difference in hardness when these intermediate fractions are used. This suggest that the amount of the intermediate fraction 'SH' that could be added to the soft fraction is limited if the butter made is to spreadable.

The Stand-up and Oil-off properties of the butter samples, shown in Table 4, generally showed good stand-up and oil-off properties when the hardness were above about 100gms. Poor stand-up and oil-off were expected and seen as the proportion of intermediate fraction in hard fraction was increased (i.e., expt 13-16).

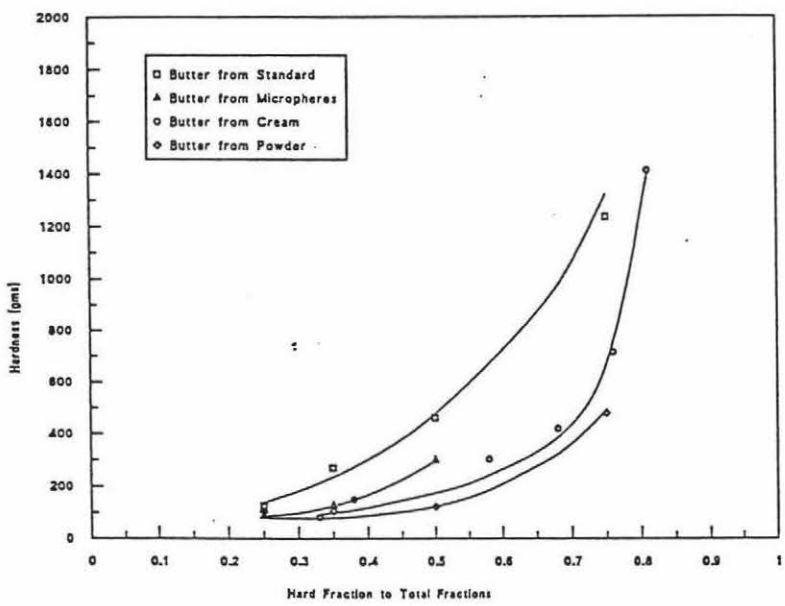


Fig 31 Overall Comparison of Butter Hardness for the Various Methods of Buttermaking.

Fig 31 shows an overall comparison of the butter hardness of four distinctive methods of buttermaking using similar or equivalent hard and soft fat fractions. It can be concluded that if a portion of the fat could be made into spherulitic form, the resulting "butters" would be much softer than one that have needle shaped crystalline matrix structures.

4.6 Statistical Analysis to determine the Fat Fraction Compositions in Relation to Butter Hardness

Table 5 Sectility Hardness of Butter

Expt No	Compositions of HF+Int:SF+Int	Fraction Ratio	Hardness(gms)	Hardness(gms)
1	50+50:90+10	36:64	120	234
2	90+10:90+10	35:65	114	242

Basic regression of the butter hardness at day 1 and 7 with hard fraction, intermediate fraction in hard fraction, and soft fraction gave the following regression equations,

Butter Hardness @ Day 1 = 336 + (17.1 x HF) + (16.4 x INT(HF)) - (14.4 x SF) .....(1)

s = 47.19 R-sq = 94.8% R-sq(adj) = 93.5%

Butter Hardness @ Day 1 = 961 - (2.07 x INT(HF)) - (14.4 x SF) .....(2)

s = 47.01 R-sq = 94.4% R-sq(adj) = 93.6%

Butter Hardness @ Day 7 = 1274 - (0.93 x INT(HF)) - (18.9 * SF) .....(3)

s = 62.55 R-sq = 94.2% R-sq(adj) = 93.3%

HF = Hard Fraction

INT(HF) = Proportion of Intermediate Fraction in Hard Fraction

SF = Soft Fraction

N.B. Unusual observations ie. points that did not fit the regression well, were noted for trial 4 and 12.

Regression analysis of the variables confirmed that either the hard or intermediate fractions are highly correlated with the other predictor variables (Equation (1) and (2) - for Day 1 hardness). Equation (3) predict Day 7 butter hardness.

The results suggested that the proportion of intermediate fraction in the hard fraction cream did not significantly affect the butter hardness. However, an increase in the proportion of intermediate fraction in the soft fraction gave a corresponding increase in the butter hardness. At the temperature of hardness measurement the intermediate fractions are solid, and could be expected to behave as hard fractions. Hence, the regression analysis has shown that the intermediate fraction in hard fraction has not significantly affected the butter hardness.

Note that samples containing a total of 40% intermediate fraction have a composition close to that of normal butter.

In the case of intermediate fraction in soft fraction, the mixing and subsequent cooling to 6°C would have resulted in the formation of fat crystals in needle shaped form.
By conditioning butter samples that have high (Table 5 - Expt No. 1) and low (Table 5 - Expt No. 2) intermediate fraction in the hard fraction cream at a temperature which is higher than the dropping point temperature of the intermediate fraction, an increase in hardness when intermediate fraction was present in the soft fraction, but not when it was present in the hard. The increase in hardness appeared to depend only on the presence of intermediate fraction, and not on the proportion.

4.7 Effects of Crystallising Temperature and High shear Mixing on Buttermaking

This experiment was carried out to determine the effects of crystallising temperatures of the lower melting point fat fractions i.e, soft and intermediate fractions; and, high shear mixing on butter hardness.

In this experimental, a quantity of hard and soft fraction from a single stage fractionation of anhydrous milk fat at 22°C was obtained from Dairy Research Institute. The proportion of the hard and soft fraction components were 24:76 respectively.
In the crystallisation process, soft fraction was placed in a container and the container put in a water bath with water temperature regulated by means of a heater and recirculation of refrigerated water in the waterbath for 16 hours.

Creamed hard fraction was produced by mixing the fraction with buttermilk serum, heated to 55°C and homogenised at 1.70/1.38 x 10^3 kPas. The compositions by weight were 45% combined milkfat, 5% buttermilk powder and 50% water. Note that these pressures are probably higher than optimum for this work, but are the lowest that can be consistently applied in our equipment. The homogenised mixture was then further heated to approx 75°C and separated to obtain a high fat cream. The cream was then cooled to approx 30°C, and blended in with the crystallised soft fraction. The mixture was further cooled to 10-12°C and churned the next day to make butter. Depending on the percentage of fat content in the high fat cream, the proportion blended with the soft fraction would be such that the butter milkfat composition is equal or close to the original anhydrous fat composition. After churning the butter was sampled and further worked in a Z-blade mixer. Hardness of the butters were determined after 1 and 7 days' storage at 10°C.

Table 9 Effects of Crystallising Temperatures on Butter Hardness

No	Ratio HF:SF	Crystallising Temperature °C	Hardness(Churned) gms Day 1	Hardness(Churned) gms Day 7	Hardness(Worked) gms Day 1	Hardness(Worked) gms Day 7	Moist %
1	30:70	13.5	841	957	461	544	11.40
2	30:70	16.5	970	1062	700	850	15.14
3	30:70	18.0	1369	1450	1009	1150	13.27

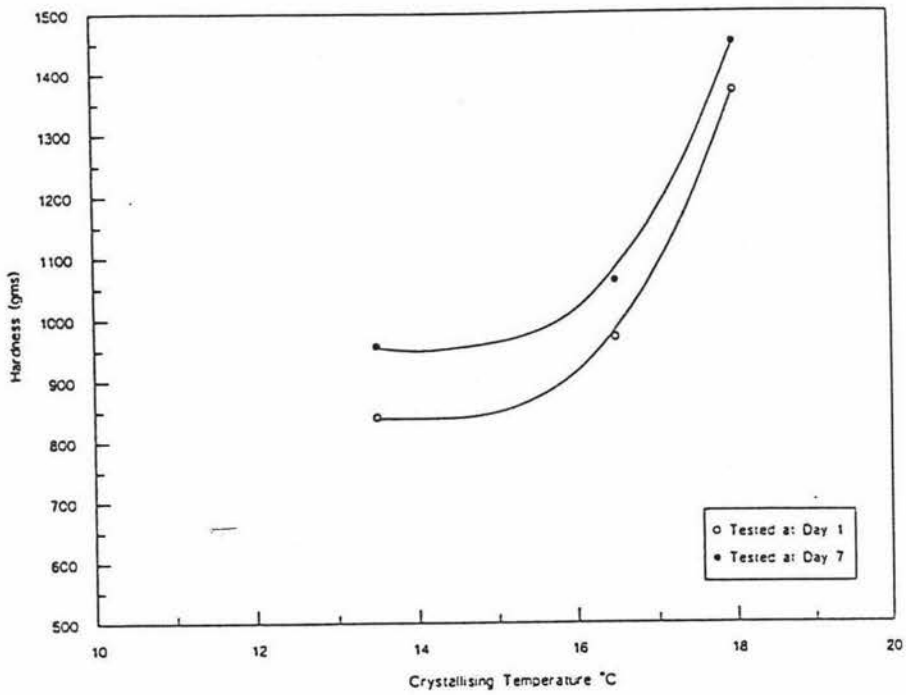


Fig 32 Effect of Butter Hardness in Relation to Soft Fraction Crystallising Temperature (churned)

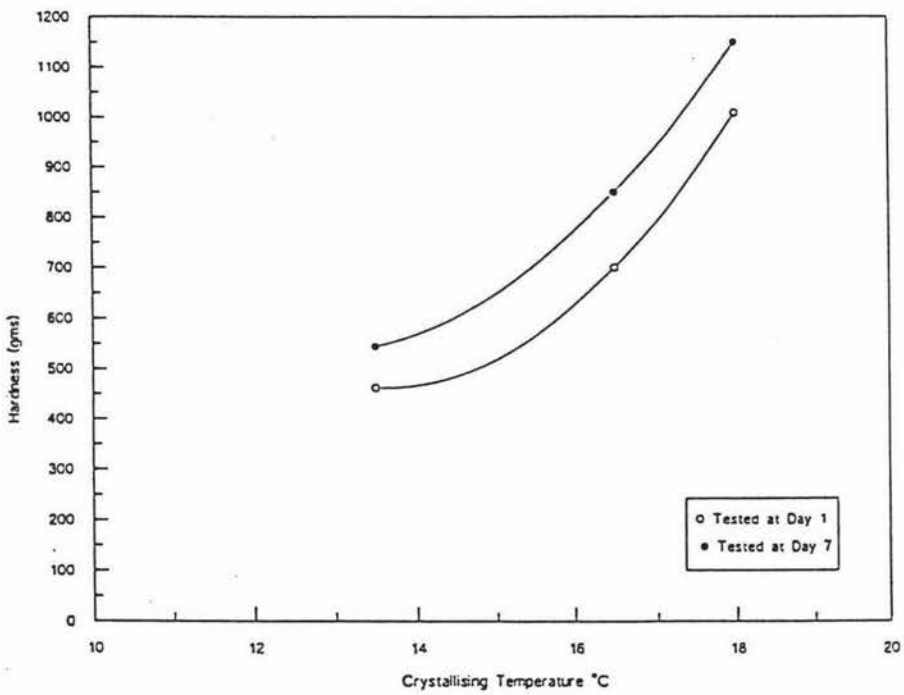


Fig 33 Effect of Butter Hardness in Relation to Soft Fraction Crystallising Temperature (worked)

4.7.1 Results and Discussion

Table 9 shows the experimental results. Although the single stage fractionation of AMF gave a ratio in proportion of hard and soft fractions of 24:76, the experimental proportions was 30:70 based on analysis and calculations. That is, more hard and proportionately less soft fraction were used; the butter produced would have a higher hardness value than if a 24:76 proportion of hard and soft fractions were used respectively.

Fig 32 and 33 shows that crystallisation temperatures is proportional to hardness and inversely to the graininess of the butter texture. It can be concluded that the graininess texture is from the soft fraction crystals which is still present in solid form after the churning process. It is suggested that using a colloid mill to grind the soft fraction crystals into finer crystal particles before blending with the hard fraction cream would help to reduce the graininess and at the same time maintain low butter hardness. The other very significant finding was that the same butter after churning would have a higher hardness (Fig 31) than the same churned butter being subjected to subsequent working in a Brabender S 300 H high shear mixer (Fig 32). Further working also greatly reduced the graininess of the butter texture.

Stand Up and Oil Off at 22±1°C of the churned and reworked butter samples were carried out and found to 100% standup and no oiling off.

Although the butter hardness is much lower than standard butter of similar fat compositions, it is still higher than what would have been preferred. This is attributed to the fact that single stage fractionation process only produced 24% of the hard or high melting point fat fraction that could be used to make the cream. It is expected that a higher proportion of cream in the butter making could reduced the hardness further. The can be done in the fractionation process by partial recycling of the soft or low melting fat fraction to the fractionation process and thus lower the crystallisation temperature.

4.8 Effects of Hardness on Reworked Butter

The butters used for this experiment were made from creamed high fat hard fraction method which had been stored for two months at -15°C. It was then taken out and conditioned at 10°C overnight. The butter was reworked in a Kenwood mixer with a hook attachment. The reworked butter was further conditioned at 10°C and tested for hardness at Day 1 and 7.

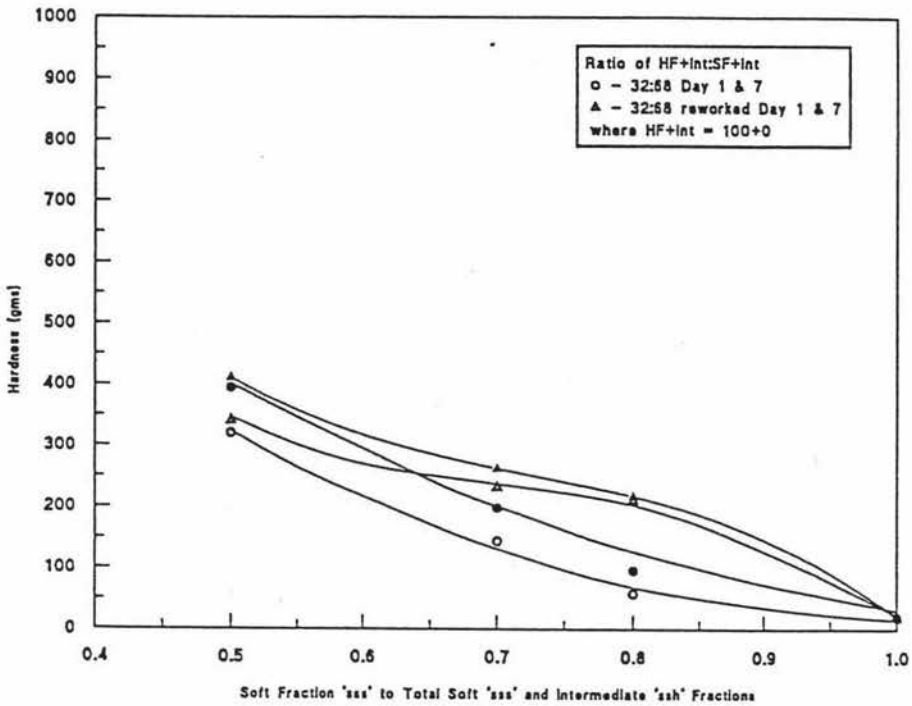


Fig 34 Comparison of Butter Hardness to that of same Butter after Storage for Two Months and Reworked at Day 1 and 7

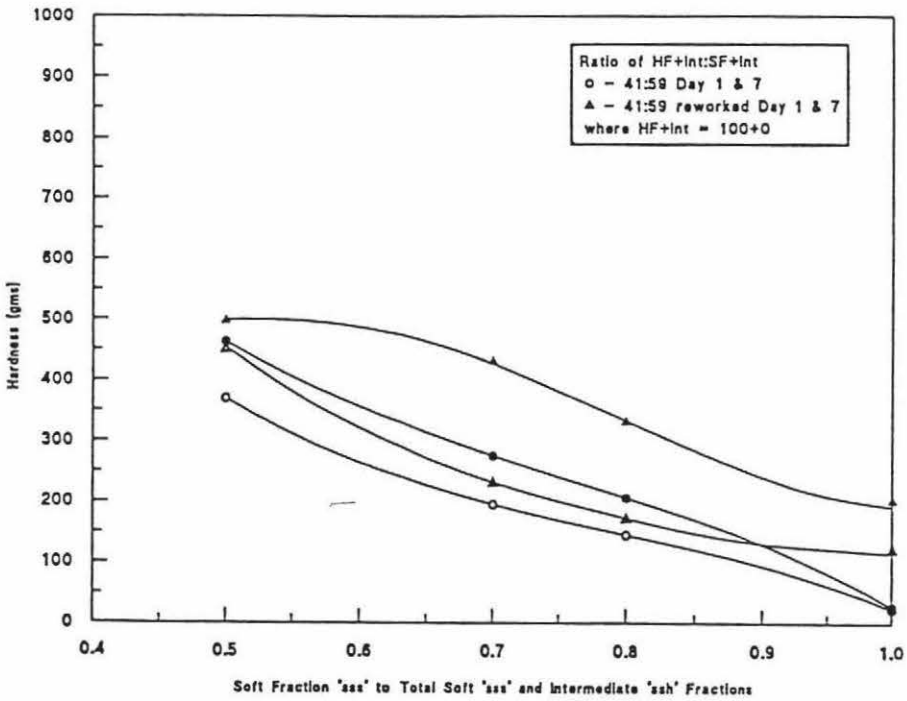


Fig 35 Comparison of Butter Hardness to that of same Butter after Storage for Two Months and Reworked at Day 1 and 7

4.8.1 Results and Discussion

Fig 34 and 35 shows the proportions of hard fraction cream mixed with soft and intermediate fractions in the ratio by weight of 32:68 and 41:59 respectively. The figures shows the comparison of hardness of the original butter to that of the same butter after storage for two months and reworked for Day 1 and 7. As expected, on reworking the butter behaves like normal butter with the release of moisture from the butter and this was reworked back into the butter. Using the Kenwood mixer with hook attachment took a longer time for reworking. In commercial reworked butter it is normal to see a decrease in the relative hardness. However, in both these cases, there was an increase in hardness of the reworked butter to that of the same butter without reworking. This confirms that reworking the butter has damaged or broken a proportion of the fat spherulites which revert the fat to its normal needle shaped matrix fat crystals resulting in an increase in butter hardness.

4.9 Effect of Butter Hardness from Crystallised Fat Fractions With and Without Mixing Arrangement

Crystallised fat fractions that were used in buttermaking had been crystallised without using a mixing arrangement during the crystallisation process. The formation of the fat crystals matrix compared to that with a mixing arrangement (as in a commercial process) would be different. In the experiment with the mixing arrangement, melted fat fractions was placed in a stainless steel container attached with a slow speed impeller mixer of 35-50rpm. The container was then put in a refrigerated waterbath. In the fat crystallisation without mixing arrangement, the melted fat fractions was placed in a container and kept in a cool temperature regulated room.

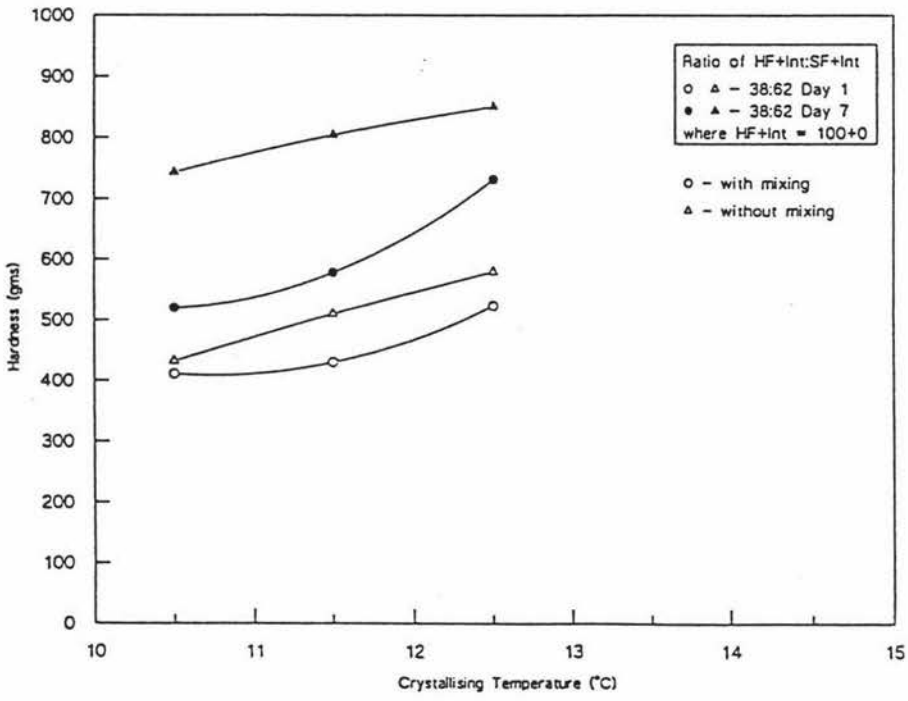


Fig 36 Effects on Butter Hardness of Crystallised Milkfat Fractions With and Without Mixing

4.9.1 Results and Discussion

Fig 36 shows the effects on butter hardness of crystallised milkfat fractions *with* and *without* a mixing arrangement at day 1 and 7. Butters having crystallised fat fractions with mixing had a much lower sectility hardness. Increase in crystallising temperature of the liquid fat fractions (soft and intermediate) gave a corresponding increase in the butter hardness. Although hardness at Day 1 was small, the corresponding Day 7 hardness were much higher with increasing crystallising temperatures. The reason for the increase could be due to the fact that when the creamed hard fraction was mixed with the crystallised fat, the resulting temperature in the range of 15-18°C was higher than the crystallising temperature of the crystallised fat. Depending on the percentage of the fat in the creamed hard fraction, the cream would become increasingly more viscous at temperature below 25°C and difficult to blend with the crystallised liquid fat. The resulting high mixing temperature would have "melted" a higher proportion of the crystallised fat of higher crystallising temperature. On conditioning at 10°C overnight, there is a higher tendency of the fat globules to "interlock" together than the crystallised fat which have a lower crystallising temperature. Hence, the increase in butter hardness. Although the hardness of butter made from liquid fat fractions with mixing arrangement was generally lower than that without mixing arrangement, it was not significantly different. However, the corresponding Day 7 hardness were quite significant. It was observed that butter made at lower crystallising temperatures has a more grainy texture in appearance and taste.

4.10 Effect of Butter Sectility Hardness of Varying the Proportion of Fat Fractions and Temperature to which the Fat Fractions were Cooled

Creamed high fat hard fraction was made in accordance with Section 3.4.8. The cream was then cooled to between 20-25°C and slowly blended with mixed fat fractions (soft and Intermediate fat fractions) which has been cooled overnight. The mixture is kept overnight at known temperature and then churned with a Kenwood mixer to make butter the next day. Immediate mixing and churning without cooling overnight will results in "leaky" butter being produced.

4.10.1 Results and Discussion

Three sets of experiment were carried out. Where the proportions of hard fraction cream was mixed with soft and intermediate fractions in the ratio by weight of 32:68 and 41:59, cooling of mixed fat fractions and churning temperature was carried out at 4-6°C. For fats mixed ratio of 37:63, the cooling and churning temperature was between 8-10°C. The mixed fat fractions cooling or fat crystallisation process was carried out without any mixing arrangement which could invariably affects the formation of the fat crystals matrix as compared to that in a commercial process. Beside cooling temperature the proportion of intermediate fat fraction in the mixed fat fractions governs the formation of the fat crystals matrix and its physical properties during the cooling/crystallisation period.

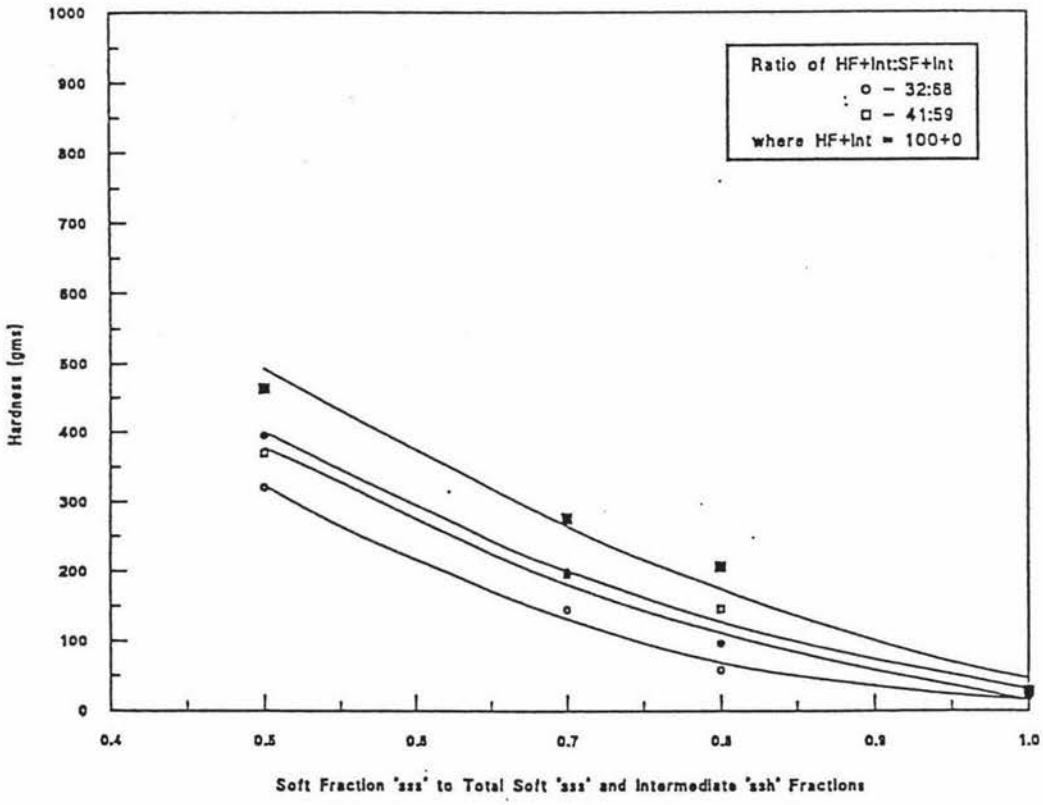


Fig 37 Sectility Hardness on Butter at Day 1 and 7

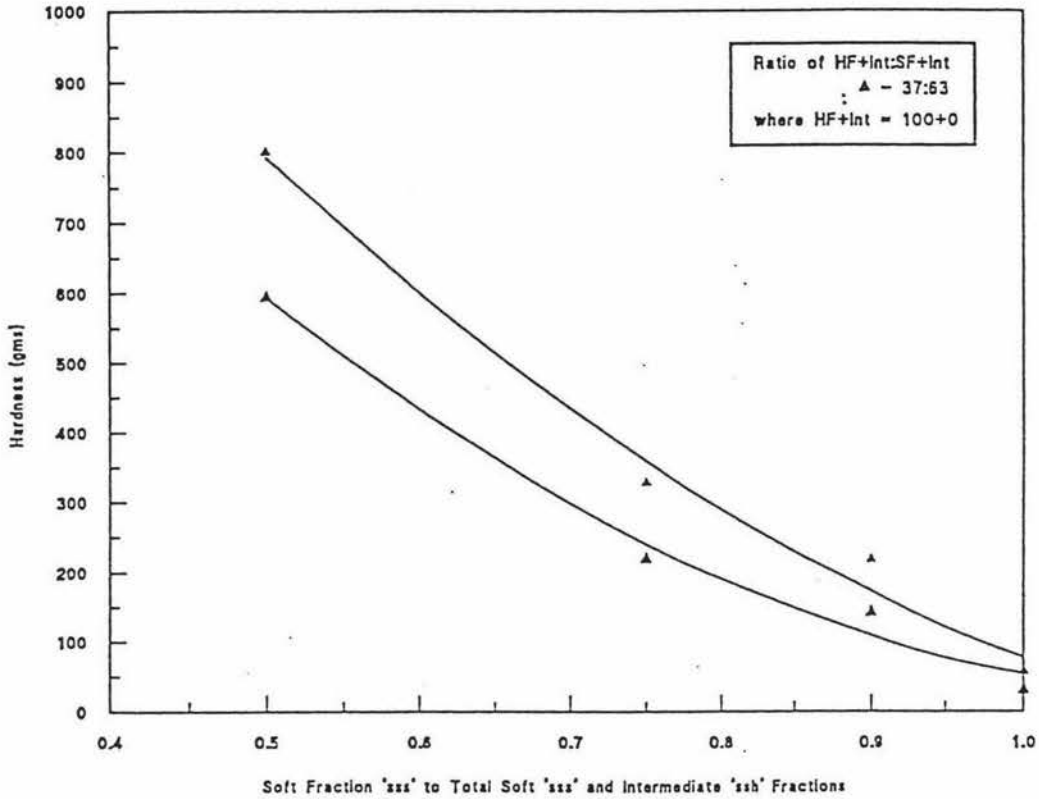


Fig 38 Sectility Hardness on Butter at Day 1 and 7

Fig 37 shows the sectility hardness of butter tested at Day 1 and 7 for various proportions of mixed fat fractions and varying the ratio of hard fraction cream to mixed fat fractions. As expected, the hardness increases with an increase in the proportion of intermediate fraction in the mixed fat fractions. The difference in hardness tested at day 1 and 7 are more pronounced with higher intermediate fraction in the mixed fat fractions. Fig 38 shows a similar pattern but at much higher butter hardness when the cooling and churning temperatures are higher although the ratio of the hard fraction cream and mixed fat fractions were between the other two sets of experiment. The butter produced at lower cooling and churning temperatures has an apparent graininess in texture compared to that of butter at higher cooling and churning temperatures. This is due to the formation of bigger fat crystals of the mixed fat fractions at lower cooling temperature prior to mixing and churning. As there was no mixing arrangement during the cooling/crystallisation period of the mixed fat fractions, it is envisaged that fat crystals size varies from the outside to the core of the sample. There is a correlation and limitation as to the proportion of intermediate fraction in the mixed fat fractions that could be used and that of cooling temperature for proper blending of the cream and cooled fat fractions.

4.11 Effects of Crystallised Mixed Fat Fractions of Varying Proportions and Crystallising Temperatures.

The Bohlin Rheometer System viscosity test was used to provide an indication of the properties on the formation of fat crystals of mixed fat fractions. The Bohlin determines directly the viscosity, shear rate and shear stress of a given sample at a predetermined constant temperature and a time frame. Soft 'SSS" and Intermediate 'SSH' fat fractions were used. Essentially, known proportion of melted soft and intermediate fat fractions was mixed and chilled in a refrigerated waterbath at a constant temperature for 20 hours. Mixing of the fat fractions was provided with a slow impeller mixer at approx 35-50 rpm. The conditions of cooling temperatures and proportion of fat fractions were chosen on the basis of the regimes that are useful.

4.11.1 Results and Discussion

Fig 39 shows the effect of crystallised milkfat fractions in relation to viscosity and time. It was assumed that the relative rate crystallisation of fat would have reached an equilibrium or optimum rate over a period of 20 hours.

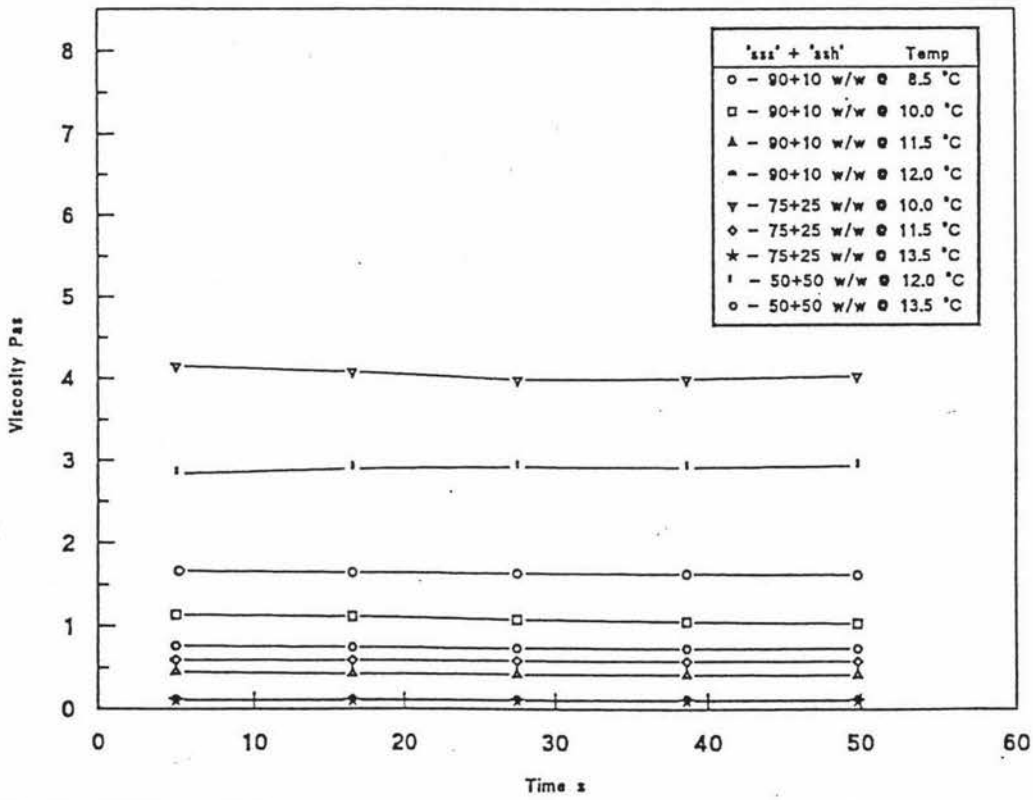


Fig 39 Effect of Crystallised Milkfat Fractions in Relation to Viscosity and Time

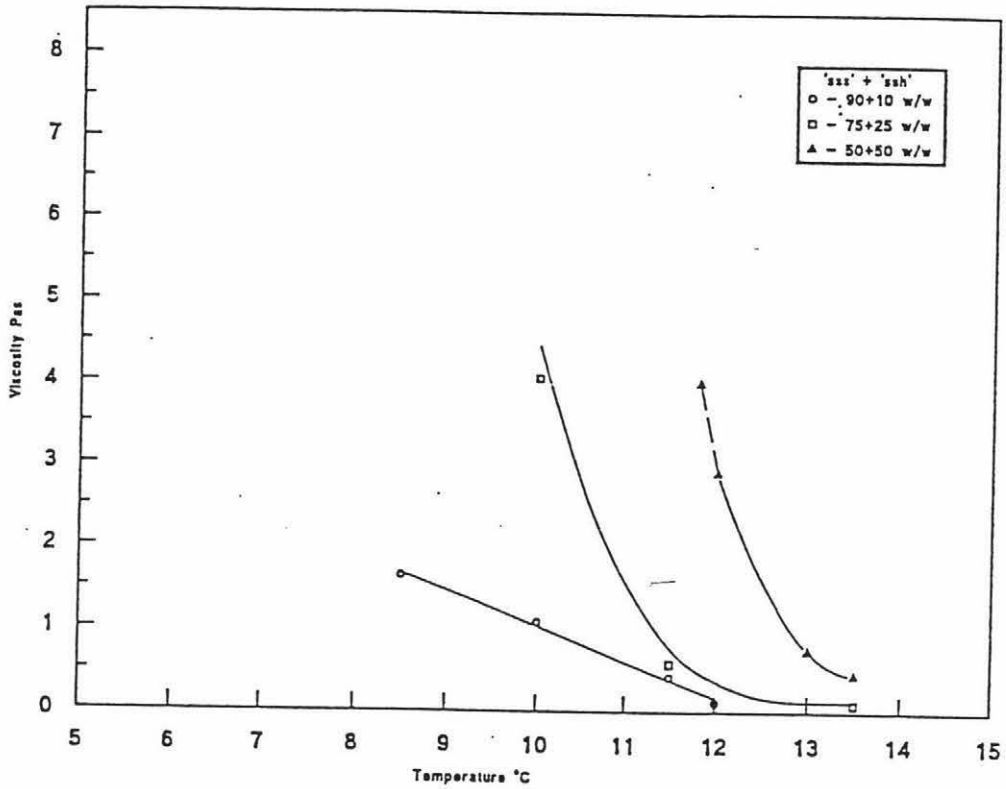


Fig 40 Effect of Crystallised Milkfat Fractions in Relation to Viscosity and Temperature

Fig 40 shows the variation of viscosity of milkfat fractions, crystallised at different temperatures and containing different proportions of intermediate fraction, in relation to the time of shearing.

It is assumed that the relative rate crystallisation of fat would have reached an equilibrium or optimum rate over a period of 20 hours. Fig 40 gives a presentation of the data that illustrates the viscosity against the cooling temperature of various mixed fat proportions. For a 90+10 w/w of soft 'SSS' and intermediate 'SSH' fat fractions, the cooling temperature is inversely proportional to the viscosity. As the proportion of intermediate 'SSH' fraction is increased, there is a steep rise in viscosity of the crystallised fat fractions. The temperature increases if the proportion of intermediate fraction is low and there is a linear relationship between the proportion and temperature. If the proportion is higher, as the temperature is increased, some melting of the intermediate fraction probably occurs, and the viscosity drops rapidly. From the solid fat profile of the fat fractions, it can be estimated that between 10-11.5°C, the percentage solid fat in the intermediate fraction decreases by about 5%. Although this figure is not

large when the proportion of liquid fat becomes small the viscosity rises very rapidly. We can conclude that the maximum viscosity range at which the crystallised fractions that could be blended with creamed high fat hard fraction in buttermaking is in the vicinity of 5-6 Pas.

4.12 Effects of Cycling Temperature on Butter

Butters made from the creamed high fat hard fractions method with the following fat compositions; ratio by weight of high fat cream HF+Int(SSH) = 50+50, mixed fat fractions SF(SSS)+Int(SSH) = 83+17, ratio of cream to that of mixed fat fractions by weight = 36:64, and moisture of 15.6%, were used in these trials after a storage period of 50 days at 3-5°C.

Sample rings were prepared and hold for two hours at temperatures of 10, 15, 20, and 28°C. The sample rings were then conditioned at 10°C overnight and taken for sectility hardness test using an Instron Testing Instrument Series 4502.

4.12.1 Results and Discussion

Fig 41 shows the variation in butter hardness over a range of temperature cycles. There was a rapid increase in the butter hardness at cycling temperatures above 16°C, the rate of increase being greatest in the temperature cycle ranges between 14-24°C. This suggest that a certain amount of the fat spherulites from the cream fractions of lower melting point entrained in the oil-in-water phase has "melted" and when cooled its crystals structure changed to that of normal platelet or needle shaped fat crystals. Therefore, butter made by this method could rapidly increase in hardness if exposed to temperature higher than the melting point of the fat spherulites.

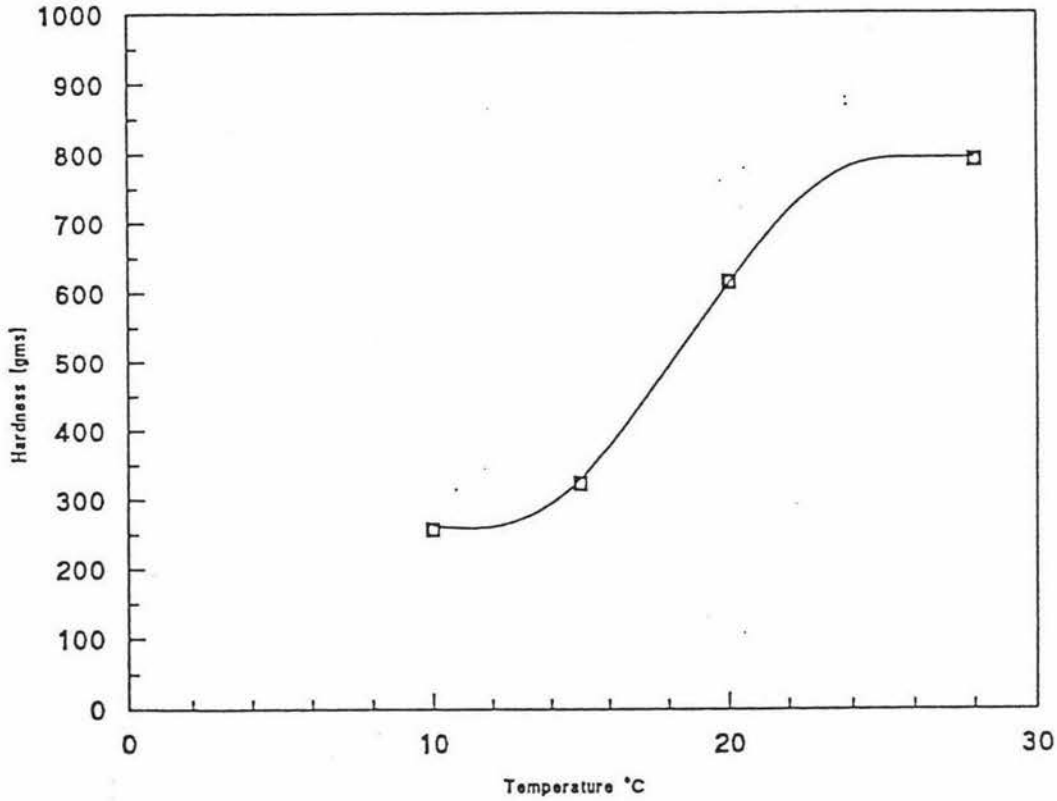


Fig 41 Effect of Temperature Cycling on Butter Hardness

4.13 Preliminary Work on a Larger Scale of Buttermaking using the Creamed High Fat Hard Fraction Method

This part of the work was carried out by a group of Dairy Diploma students as part of their course project. A single stage fractionation of milkfat at 22°C was conducted, to produce a hard and a soft fraction. A "Control - Standard Method" and an "Experimental - Cream Method" trial was carried using these fractions.

For the Control, each milkfat fraction was mixed with buttermilk powder in serum in the appropriate proportions. The two products were then blended together and homogenised to produce a cream containing 40% fat. The homogenised cream was conditioned at 12°C overnight and churned the next day in a batch churn. The appropriate amount of salt was added during churning.

In the case of Experimental, the hard fraction was mixed with buttermilk powder in serum and salt to give a composition of 48% fat and 0.8% salt(in butter). The blend was heated to 60°C through a plate heat exchanger and homogenised through a single stage

homogenising valve at 400kPa. The crystallised soft fraction (at 12°C overnight) was then mixed with the cream in the batch churn by turning the churn a few times. The final mix contained 82% fat (ratio HF:SF of 24:76), 15.8% water and the remainder buttermilk and salt. The churn was maintained at approx 7°C overnight by running chilled water. The cream was churned the next day to produce butter. Samples were taken at different stages of the process.

Of interest would be the microphotographs of the "control" and "experimental" butter as shown in Fig 42 and 43 respectively.

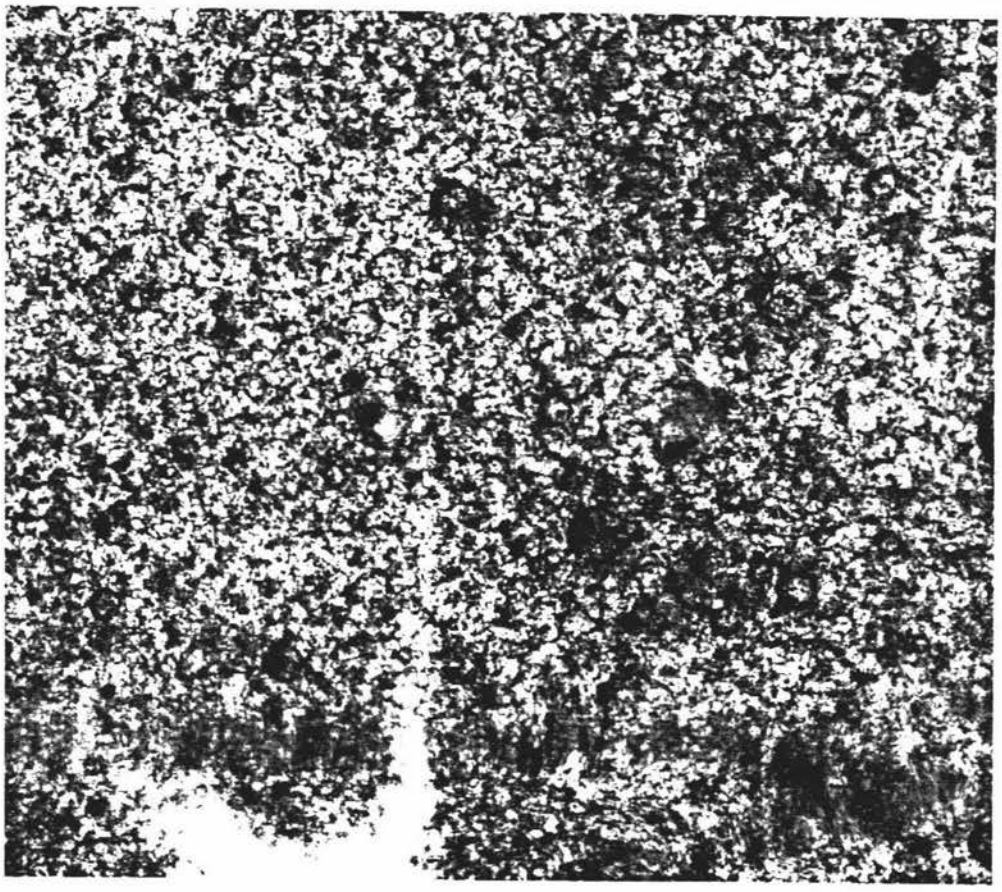


Fig 42 Microphotograph of "Control" Butter

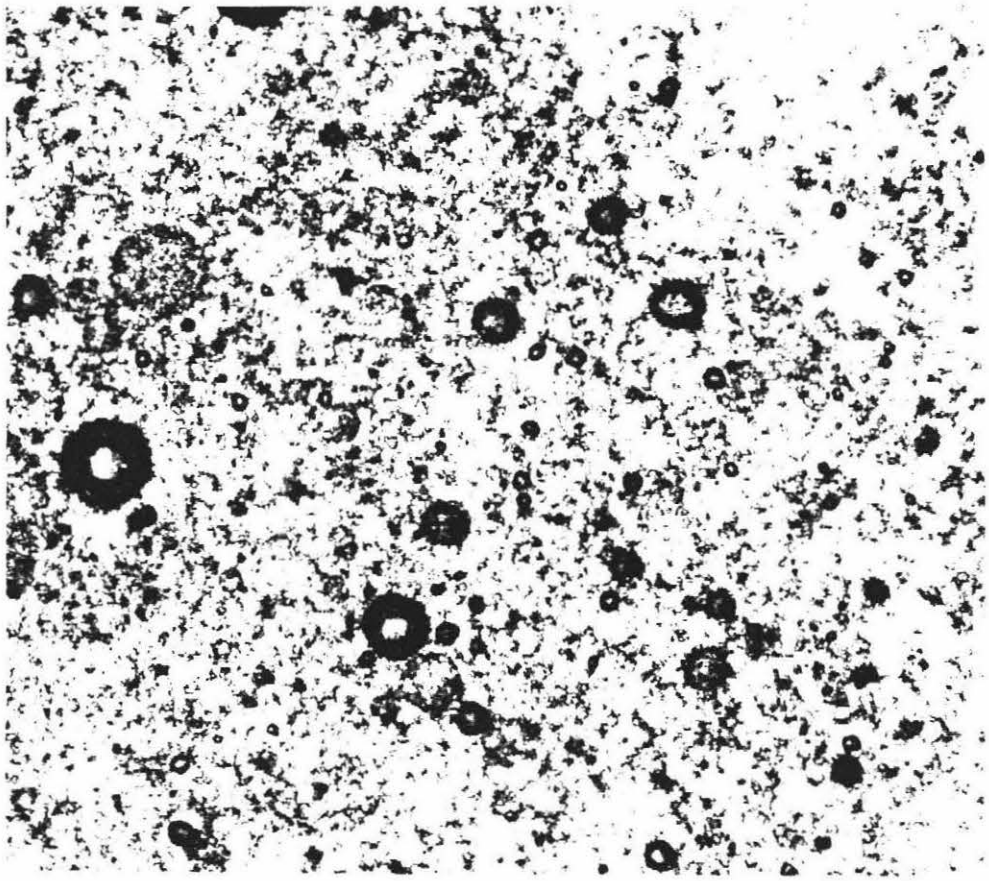


Fig 43 Microphotograph of "Experimental" Butter

4.13.1 Results and Discussion

Although the trials, in particular, the "Experimental Method" confirmed that butter could be made which was as much as 50% softer than the "Control Method", there were some shortcomings in the steps and conditions used in butter making. As a result, the butter produced was grainy, had poor moisture distribution and poor standup properties. It was suggested that the homogenising pressure used to cream the hard fraction was inadequate to produce fat spherulites sufficiently small enough as not to contribute to the overall graininess of the butter. However, from earlier trials in the determination of fat spherulitic sizes on homogenised cream, it is unlikely that these sizes (of the order of less than 10μm) would contribute to the butter graininess. A more likely explanation is

that the cooling conditions produced large crystals from the soft fraction. The poor standup properties would most likely be attributed to the fact that the crystallised soft fraction was not properly blended in to the hard fraction cream. As such, there were a high proportion of "free" crystallised soft fraction in the mix which could not be sufficiently "locked in" to the hard fraction cream. In effect, the "free" fat was unable to complete an water-to-oil phase inversion during the churning process. Such butter would also taste somewhat oily in texture. It highlights the importance of the need to have good control and proper tailored conditions depending on the fat fractions used, to produce an acceptable butter.

Comparing Fig 42 and 43 of the Control and Experimental butter, it can be seen that the experimental butter contain a much higher proportion of relatively large soft fraction crystals. It is these crystals that contributed to the graininess of the butter.

4.14 CONCLUSION

Different methods of buttermaking using milkfat fractions were investigated and comparisons of butter hardness made. Of significant interest is the comparison of butter hardness made by the standard method (Section 3.4.1) and that of the creamed fat fraction method (Section 3.4.8). Using only hard and soft milkfat fractions of similar compositions, it was noted that butter hardness made by using high fat creamed hard fraction method were significantly lower than that of standard buttermaking method. This is illustrated in Fig 10. In standard buttermaking, the cooling of the fat fractions prior to churning may cause the formation of fat crystals in a needle shaped structure, whereas in using creamed fat fraction, fat crystals in spherulitic form is more prominent in the fat fractions. Fig 11 show a comparison of butter hardness for the various methods of buttermaking.

Having confirmed that the creamed high fat fractions did produced butter which were much softer than the standard buttermaking method, the experimentation were extended to determine whether a complete composite of milkfat could be incorporated using this method to achieve butter which are spreadable at refrigerated temperatures.

Chapter 5

Section 5.1 REFERENCES

DARLING, D.F., BIRKETT, R. J., *Food Emulsion and Foams*, E. Dickson, ed., Royal Society of Chemistry, Lond., 1987

DIXON, B.D., *Aust. J. Dairy Technol.*, March 1974, 15-20

DIXON, B. D., *XVIII Int. Dairy Congress.*, **1E**, 1970, 205

DOLBY, R. M., *Aust. J. Dai. Technology.*, **14**, 1959, 103

ESDALE, J.D., WILSON, B. W., *XIX Int Dairy Congress.*, Vol **1E**, 1970, 240

FECHNER, E. J., : *U.S. Patent. 2,065,675/2,065,676* cited by Hansen, P.M.T., 1963

FISKER, A.N., JANSEN, K., *XVI Int. Dairy Congr.*, **B**, 1962, 65

FJAERVOLL, A., *XVIII Int. Dairy Congress.*, Vol **1E A4.2**, 239.

FOX, P. F., ed, *Developments in Dairy Chemistry*, Vol 1 - 4, Applied Science, New York.

GARRETT, O. F., *12th Inter. Dairy Congr.*, Stockholm, **3**(Sec. iii), 1949, 49

HANSEN, P.M.T., *Aust. J. Dairy Tech.* **18**, 1963, 79

HAUQUE, Z., KINSELLA, J.E., *J. of Food Sci.*, Vol 54, **5**, 1989, 1341-1344

JEBSON, R. S., *XIX Int Dairy Congress.*, Vol **IIE** , 1970, 240

JEBSON, R.S., *N.Z. J. Dairy Sci. and Technol.*, Vol **9**, 1974, 50

JEBSON, R.S., MUNRO, D.S., BISSELL, T.G., NORRIS, R., TAYLOR, M.W., *International Dairy Congress*, **E**, 1978, 862

JEBSON R.S and HARTEL R.W., Research Proposal "A New Technology for Milkfat Products" 1989

JENNESS, R., *Composition of milk, in Fundamentals of Dairy Chemistry*, 3rd Ed., Van Nostrand Reinhold, New York, 1988, 1-38

JENSEN, R. G., *J Dairy Sci.*, 1967, **50**, 119

KERKER, M., *"The Scattering of Light and Other Electromagnetic Radiation"*, Academic Press, Lond., 1969

KROG N., and others, *Dispersion Sci. and Technol.*, **10**, 1989, 483-504

McDOWALL, F. H. The Buttermaker's Manual, NZ University Press, Wellington, 1953, 474-479

MacGIBBON, A.K.H., McLENNAN, W.D., *N.Z. J. Dairy Sci. and Technol.*, 1987, **22**, 143-156

MORTENSEN, B. K., DANMARK, H., Milchwissenschaft, **37**, 1982, 193

MULDER, H., WALSTRA, P., *The Milkfat globule. Pudoc, Wageningen, Netherlands, and Commonwealth Agricultural Bureaux, Farnham Royal, Bucks, UK.*, 1974

MULDER, H., WALSTRA, P., The milkfat Globule : Emulsion Science as applied to milk products and comparable foods. Wageningen. CAP/PUDOC

MUNRO, D.S., *International Dairy Federation*, 1982, **142**, 137

MUNRO, D. S., BISSELL, T.G., JEBSON, R. S., NORRIS, R., TAYLOR, M. W.., *XX Int Dairy Congress.*, Vol **E**, 1978, 862

NORRIS, R., *N.Z. Patent No. 172 101.*, 1973

NORRIS, R., *U.S.A. Patent No. 4 005 228*

PATEL, A.A., FREDE, E., BUCHHEIM, W., KIELER *Milchwirtschaftliche Forschungsberichte.*, **39**(4), 1987, 191-202

PEARCE, K. N., KINSELLA, J. E., *J. Agric. Food Chem*, Vol. 26, **3**, 1978, 716-722

PRENTICE, J.H., An instrument for estimating the spreadability buttter. Lab Practice., **3**, 1954, 186-189

PYENSON, H., TRACY P. H., *J. Dairy Sci.*, **29**, 1946, 511

RIEL, R., *Dairy Science and Technology, La Fondation De Technologie Laitiere Du Quebec, Inc.*, Chapter I, 1985, 10-14

SNOW, N.S., el at., *Aust. J. Dairy Tech*nol., Sept 1967, 122-125

SAMUELSSON, E., K. I. PETTERSON., Arsskr. Alnarps, lantbruksmejeri otradgardsmst. 1937

SCHMIDT, D.G., *Development in Dairy Chemistry.*, Vol 1, Applied Science Publishers, Essex, England., 1982

SHERBON, J. W., *J. Am. Oil Chem. Soc.*, **51**, **22**, 1974

TAYLOR, M.W., DOLBY, R.M., RUSSELL, R.W., *N.Z. J. Dairy Sci. and Technol*, The reworking of Butter, 1972, 172-176

TAYLOR, M. W., DOLBY, R. M.., RUSSELL, R. W., The effect of manufacturing conditions of setting rate of butter. *The Journal of Dairy Research*, **140** 1973, 393-402

TAYLOR, M. W., JEBSON, R.S., *XIX Int. Dairy Congr.*, **1E**, 1974, 673

TAYLOR, M.W., NORRIS, R., The physical properties of dairy spreads. *N.Z. J. Dairy Sci. and Technol.*, **12**, 1977, 166-170

TIRTIAU, A., *J.O.A.C.S 60.*, 1983, 452A

TOWNSEND, F.R., el at., *Aust. J. Dairy Technol.,* June 1968, 85-89

TRACY P. H., *Canad. Dairy Ice J.*, **27** (11), 1948, 52

Van Dam, W., Versl. Landbouwk. Onderz., **32**, 1927, 234

WALSTRA, P., *Neth. Milk Dairy J.* **19**, 1965,93

WALSTRA, P., E.C.H. van BERESTEYN., *Neth. Milk Dairy J.*, 29, 1975, 35-65

WALSTRA, P., JENNESS, R., *Milkfat Globules - Dairy Chemistry and Physics, John Wiley and sons, NY.*, 1984, 237

WALSTRA, P., JENNESS, R., *Outline of Milk Composition and Structure - Dairy Chemistry and Physics, John Wiley and sons, NY.*, 1984, 2

WILSON, B. W., *Aust. J. Dairy Technology.*, **30**, **10**, 1975

WOOD, F. W., DOLBY, R.M., *J. Dairy Res.*, **32**, 1965, 269