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Arunee Srichantra

STUDIES OF UHT-PLANT FOULING BY FRESH, RECOMBINED AND RECONSTITUTED WHOLE MILK

EFFECTS OF PREHEAT TREATMENTS

**A THESIS PRESENTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS
FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN FOOD ENGINEERING**

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ARUNEE SRICHANTRA

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Abstract

The objective of this study was to investigate the effects of preheat treatments on fouling by fresh whole milk (FWM), recombined whole milk (RCB) and reconstituted whole milk (Recon) in the high-temperature heater of indirect UHT plants.

Various preheat treatments prior to evaporation during milk powder manufacture were applied to skim milk powder (SMP, 75 °C 2 s, 85 °C, 155 s and 95 °C, 155 s) and whole milk powder (WMP, 95 °C, 33 s). These preheat treatments were so-called “evaporator preheat treatments”. Skim milk powder (SMP) and whole milk powder (WMP) were derived from the same original batch of pasteurised FWM to remove the effects of the variation in milk composition between different milk batches. These SMPs were recombined with anhydrous milk fat and water to prepare RCB, and WMPs were reconstituted with water to prepare Recon. Then, (homogenized) FWM, RCB and Recon were subjected to various preheat treatments (75 °C, 11 s, 85 °C, 147 s and 95 °C, 147 s) prior to UHT processing. These preheat treatments were so-called “UHT preheat treatments”. Temperature difference (hot water inlet temperature – milk outlet temperature) was taken as a measure of the extent of fouling in the high-temperature heater. The slope of the linear regression of temperature difference versus time (for two hours of UHT processing) was taken as fouling rate (°C/h).

Increasing both evaporator and UHT preheat treatments resulted in increasing fouling rate and total deposit weight for all three whole milk types for several milk batches. In the case of FWM, there was no reduction in fouling rate with increasing UHT preheat treatment whether FWM was homogenized then preheated, preheated then homogenized or not homogenized at all. These findings, which are wholly consistent and well replicated, are in apparent conflict with the results of most previous comparable studies. Possible reasons for this are explained.

Further investigations of the effects of homogenization relating to the role of whey protein on the surface of the fat globules showed that whey protein associated with the membrane covering the surface of fat globules for homogenized then preheated FWM, RCB and Recon and that association increased with increasing heating process stage. The increasing association of whey protein with the milk fat globules membrane with increasing severity

of heating process stage became faster when preheat treatment was more severe: the association of whey protein plateaued on intermediate temperature heating when the milks were preheated at 75°C, 11 s and on preheating when the milks were preheated at 95°C, 147 s.

In the case of FWM, the thickness of the membrane covering the surface of fat globules for homogenized then preheated FWM, which increased with the severity of heating process stage, was greater than the thickness of the membrane in preheated then homogenized FWM. Preheating then homogenization resulted in the greater interfacial spreading of small molecules on the surface of fat globules, i.e. whey protein or small molecules from the disintegration of casein micelles during preheating.

Possible basic mechanisms for UHT fouling in the high-temperature heater include: the reduction in the solubility of calcium phosphate and the deposition of protein as fat-bound protein and non-fat-bound protein. When non-fat-bound protein in milk plasma deposited, it could be a carrier for the deposition of mineral, such as, the precipitate of calcium phosphate in the casein micelles or the deposition of complexes between whey protein and casein micelles.

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Chapter 1

Project overview

There is a conflict in the literature as to the effect of evaporator preheat treatment (prior to evaporation) and/or UHT preheat treatment (prior to UHT processing) on the fouling that occurs in the high-temperature heat exchanger of indirect ultra high temperature (UHT) milk sterilization plants. Bell & Sanders (1944), Burton (1968), Lalande *et al.* (1984), Patil & Reuter (1986a, 1988) and Mottar & Moermans (1988) found that preheating raw fresh whole milk prior to UHT treatment reduced UHT plant fouling. These reports have led to preheaters being added to some UHT plants, particularly by APV (SPX). However, this effect is opposite to the results of Newstead *et al.* (1999) and D F Newstead (personal communication, Fonterra, 2003), who reported that the UHT fouling rates of recombined whole milk and reconstituted whole milk increased with increasing evaporator preheat treatment (applied during milk powder manufacture) when the recombined whole milk used in successive trials was derived from the same batch of skim milk powder and reconstituted whole milk used in successive trials was derived from the same batch of whole milk powder.

Subsequent investigations of this phenomenon by Fonterra only resulted in further compounding of the conflict when it was found that increased preheat treatment of the recombined whole milk prior to UHT processing also increased, not decreased, fouling.

A search for possible reasons for this apparent discrepancy revealed the following variations in conditions: milk composition between batches, homogenization before or after UHT preheat treatment for fresh milk and the composition of the membrane of fat globules among fresh whole milk (FWM), recombined whole milk (RCB) and reconstituted whole milk (Recon).

Thus, this project was set up to clarify the effects of preheating on UHT fouling. The objectives of the project were:

- To investigate the effect of evaporator and/or UHT preheat treatments on the fouling rate and deposit formation in the high-temperature heat exchanger of a UHT plant, for FWM, RCB and Recon when the effects of batch-to-batch variation of the composition of milk was removed.
- To investigate heat-induced changes in the membrane of fat globules for FWM, RCB and Recon.
- To investigate the effect of homogenization before or after UHT preheat treatment on UHT fouling for FWM.

Chapter 2

Literature review

2.1 Introduction

Fouling on the heated surface during heating of milk is a complex process of deposit formation involving proteins, fat and minerals. The presence of a deposit decreases the efficiency of heat transfer from the heating medium to the milk and limits the run time for the process because of the gradual blockage of the milk passes in the heat exchanger (Burton, 1968). The effect of fouling in the heat exchanger are: an increase in total deposit weight (Lalande *et al.*, 1984; Mottar & Moermans, 1988; Patil & Reuter, 1988; Grandison, 1988b; Foster *et al.*, 1989), an increase in heat transfer resistance (Kastanas *et al.*, 1995a; 1995b; Newstead *et al.*, 1999; Lewis & Heppell, 2000) and an progressive increase in pressure loss on the product side (Bell & Sanders, 1944; Burton, 1966a, 1968; Grandison, 1988b; Kastanas *et al.*, 1995b).

This project focused on the effects of fouling during UHT milk processing, specifically concentrating on fouling of the high-temperature heating section of the plant, and the factors that affect the level of fouling in that region.

Thus, the objectives of this literature review are:
1) To review the effect of UHT preheat treatment on the fouling rate, total deposit weight and deposit composition for fresh whole milk (FWM), recombined whole milk (RCB) and reconstituted whole milk (Recon) in the high-temperature heater.
2) To review the effect of evaporator preheat treatment on these same responses.
3) To review relationships between heat-induced changes in fresh whole milk, RCB and Recon, and UHT fouling.

2.2 Processing of fresh whole milk

After raw fresh whole milk is collected from the farm, it is unloaded from road tankers to silos at the manufacturing site. The steps of subsequent milk processing are described in the following.

2.2.1 Milk reception

In general, only small distances between farms and manufacturing sites allow a small change of the milk temperature before arriving the factory. Raw milk can be kept for at most 2 days if it is cooled to < 6 °C at the milk reception site at the dairy plant (Walstra *et al.*, 1999).

2.2.2 Milk separation

Cold raw milk from the silo is filtered before it is separated into cream and skim milk by centrifugal separators. The skim milk and cream are then pasteurized. Alternatively, raw fresh whole milk can be pasteurized prior to separation. Cream is used to standardize the fat content of whole milk to the required level.

2.2.3 Milk pasteurisation

Pasteurisation kills bacterial pathogens and causes only small chemical and physical changes in the milk (Walstra & Jenness, 1984). Milk can be pasteurised at 63 °C for 30 min or equivalent processes, such as 72 °C for 15 s or 89 °C for 1 s.

2.2.4 Standardization

Fat standardization may be required depending on the desired composition of the final product. Standardization may also involve standardizing the solids not fat, the fat: solids not fat ratio, and the protein: solids not fat ratio.

2.3 Milk powder manufacture

The steps for standard milk powder manufacture are described below.

2.3.1 Evaporator preheating

Preheating prior to evaporation denatures whey proteins, forms complexes between β-lg and κ-casein, changes the form of soluble calcium phosphate to the insoluble colloidal calcium phosphate, modifies the casein micelles structure, decreases the milk pH and causes Maillard browning between proteins and lactose in milk (Singh & Newstead, 1989). Evaporator preheating of skim milk during standard skim milk powder manufacture has a relatively large effect with respect to whey protein denaturation (Singh & Creamer, 1991a; Oldfield *et al.*, 2005a). For non-homogenized fresh whole milk, there is an increase in the association of casein micelles and whey proteins with the MFGM (Ye *et al.*, 2005).

2.3.2 Evaporation

Evaporation is the process that removes water from the milk, increasing the concentration of milk prior to homogenization and drying. Evaporation is normally carried out in the temperature range 50-70 °C. Evaporation causes the denaturation of whey proteins, an increase in the level of colloidal calcium phosphate, an increase in the size of casein micelles and a further decrease in milk pH (Singh & Newstead, 1989; Singh & Creamer, 1991a). For whole milk powder manufacture, there was association of casein micelles and whey protein with the fat globules during evaporation (Ye *et al.*, 2005).

2.3.3 Homogenization

Homogenization is applied to whole milk only, not skim milk. Homogenization of milk concentrate during whole milk powder manufacture is the step that increases the number of fat globules and the extent of adsorption of casein (Singh & Newstead, 1989; Ye *et al.*, 2005). However, whey protein and some natural membrane are still present in the membrane covering the fat globules of whole milk concentrate (Singh & Newstead, 1989).

2.3.4 Drying

Drying is a step that removes most of the remaining water and transforms the milk concentrate into milk powder. Drying has a minor effect on casein, whey protein and salts (Singh & Newstead, 1989). Drying of whole milk was reported by Ye *et al.* (2007) to not cause any further association of whey protein with the surface of fat globules, in the case of fresh whole milk.

2.4 Recombining processes

Recombining is the process used to prepare RCB and Recon from skim milk powder and whole milk powder, respectively, prior to UHT processing. The general process for the preparation of RCB and Recon is shown in Figure 2.1.

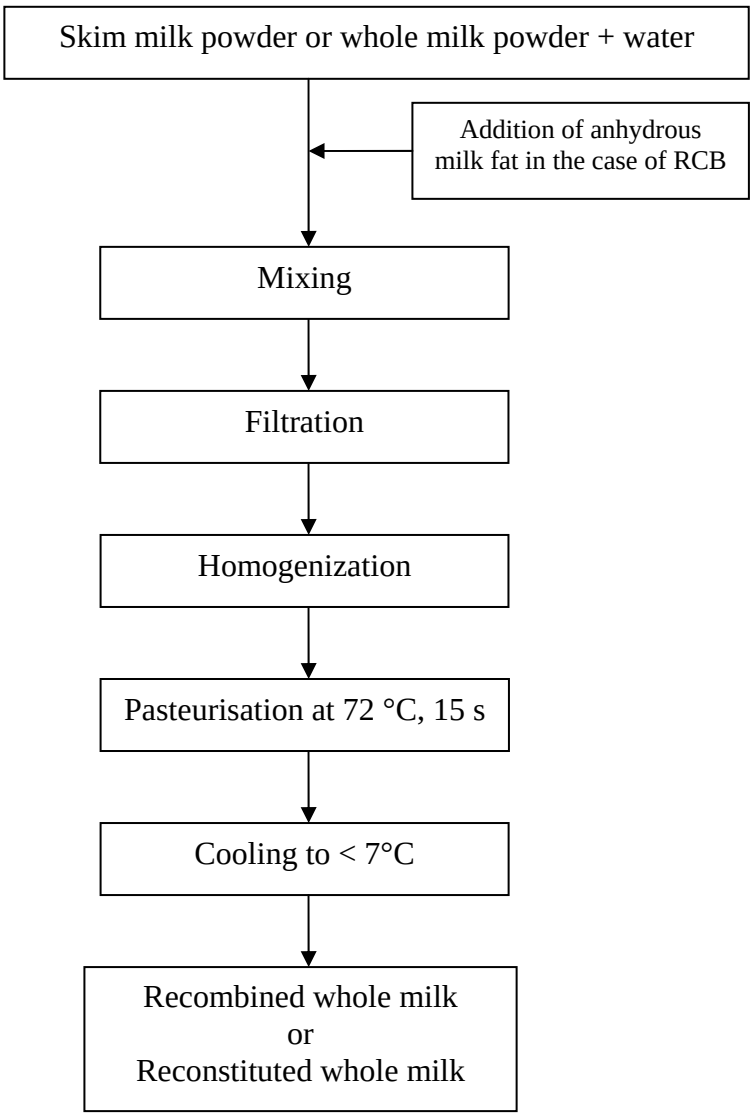


Figure 2.1 Recombining process (from a private Fonterra document).

Skim milk powder is mixed with water. Anhydrous milk fat is added to the skim milk before the milk is filtered to remove undissolved powder and then homogenized. After homogenization, RCB is pasteurised at 72 °C for 15 s before cooling. For Recon, the whole milk powder is mixed with water, filtered, homogenized, pasteurized, cooled down to < 7 °C and stored at 4 °C.

2.5 Heat-induced changes in fresh, recombined and reconstituted whole milks

When fresh whole milk, RCB and Recon are heated, there are changes in casein and whey protein in the membrane covering the fat globules and in the milk plasma. RCB and Recon

are subjected to more heating steps than fresh whole milk due to the various heating steps performed during milk powder manufacture that they are based on.

Denatured whey protein (β-lg and α-la) associates with casein micelles in milk plasma during heating and the resulting whey protein-casein complexes increase the size of the casein micelles (Anema, 2007). Furthermore, heating results in the dissociation of κ-casein from the casein micelles, which has been found to be dependent on pH (Singh & Fox, 1985; Singh, 1993b). It also results in the dephosphorylation of the casein (Singh & Fox, 1989) and the Maillard reaction between the carbonyl group of lactose and lysine (Singh & Fox, 1989). Furthermore, there is a reduction in the solubility of calcium phosphate and its conversion to colloidal calcium phosphate during heating (Singh & Creamer, 1989; Singh, 1993b).

In general, when fresh whole milk is not heated and not homogenized, the fat globules are covered with natural milk fat globule membrane (MFGM), which consists of protein, phospholipids, enzymes and minerals (Walstra & Jenness, 1984). Proteins in the natural MFGM are derived from the cellular membrane of the secretary cells in the mammary gland (Walstra & Jenness, 1984). When raw unhomogenized fresh whole milk is heated at temperatures greater than 60 °C, whey protein in the milk plasma denatures and some whey protein associates with the membrane of the fat globules (Dalgleish & Banks, 1991; Houlihan *et al.*, 1992a; Sharma & Dalgleish, 1993; Sharma & Dalgleish, 1994).

Three possible associations of whey protein with the heated natural MFGM of fresh whole milk were suggested by Dalgleish & Banks (1991): the association of whey protein with the casein micelles in the MFGM, the association of whey protein with already adsorbed denatured whey proteins on the surface of fat globules and the association of whey protein aggregates with proteins in the natural MFGM. The third mechanism was suggested to be the most likely one for the interaction between the protein in the MFGM and β-lg. Furthermore, it was also reported that the association of denatured α-la was slower than that of β-lg and resulted in a lower level of α-la in the MFGM than that of β-lg.

When raw fresh whole milk is homogenized, homogenization causes a reduction in the size of the fat globules, decrease the proportion of the native MFGM to 25 % of the original amount of natural MFGM (Walstra & Jenness, 1984) and causes the absorption of

surface-active material (Van Boekel & Walstra, 1995). The major component of the milk fat globule membrane of homogenized raw fresh whole milk was reported by McPherson *et al.* (1984b), Sharma & Dalgleish (1993) and Van Boekel & Walstra (1995) to be casein, which was present in greater proportion than whey protein. κ-casein was the binder between the casein micelles and serum proteins in the membrane of fat globules of homogenized raw fresh whole milk (Sharma & Dalgleish, 1993; Sharma & Dalgleish, 1994; Van Boekel & Walstra, 1995). When heating was applied to homogenized raw fresh whole milk, there were association between whey protein and casein micelles (Walstra & Jenness, 1984; Kessler, 2002). This association took place in the milk plasma (Anema, 2007) or in the membrane covering the fat globules for homogenized then preheated raw fresh whole milk (Van Boekel & Walstra, 1995). Thus, the composition of the membrane covering the fat globules of homogenized and preheated raw fresh whole milk consisted of whey protein and casein.

When raw fresh whole milk was preheated then homogenized, heating caused the denaturation of whey protein which formed complexes with the casein (Sharma & Dalgleish, 1994; Van Boekel & Walstra, 1995). After homogenization, these complexes formed part of the MFGM together with other milk proteins (Van Boekel & Walstra, 1989; Sharma & Dalgleish, 1994; Van Boekel & Walstra, 1995; Lee & Sherbon, 2002). Thus, the compositions of the membrane which covered the fat globules for preheated then homogenized fresh whole milk included complexes between casein-whey protein as well as some whey protein.

There was a limitation for the association of whey protein with the heated MFGM for fresh whole milk when the milk was heated in the temperature range 80 °C to 90 °C because there was a limitation in binding sites for κ-casein on the membrane of fat globules for the association of whey protein (Sharma & Dalgleish, 1994). When fresh whole milk was subjected to homogenization before UHT preheat treatment, its membrane was thicker than the membrane of preheated and then homogenized fresh whole milk (Sharma & Dalgleish, 1994).

The heating and processing steps during whole milk powder and skim milk powder manufacture affected the composition of the MFGM of RCB and Recon. For skim milk, preheating prior to evaporation, and evaporation itself, caused the association between

whey protein and casein micelles, but drying did not (Singh & Newstead, 1989; Singh & Creamer, 1991a; Oldfield *et al.*, 2005a; Singh, 2007). For whole milk, preheating prior to evaporation increased the association of both casein and whey protein with the membrane of the fat globules (Ye *et al.*, 2005). Drying caused a further increase in the concentration of casein micelles on the surface of fat globules, but not of whey protein (Ye *et al.*, 2007). When SMP and WMP were recombined, the composition of the MFGM of RCB and Recon consisted of casein and whey protein. However, the proportion of casein was greater than that of whey protein in the case of RCB (Walstra & Oortwijn, 1982; Sharma *et al.*, 1996a).

When RCB was heated above 90 °C, Singh *et al*. (1996) and Sharma *et al.* (1996b) reported that whey protein associated with the casein micelles on the surface of fat globules and this association slightly increased with increasing heat treatment. There have been no studies of changes in the MFGM of Recon when it was heated above 90 °C.

2.6 Fouling by fresh, recombined and reconstituted whole milk in the high-temperature heating section of heat exchangers

Fouling by milk causes the blockage of passages in heat exchangers. This results in a short run time and the reduction of heat transfer. Thus, it is important to understand how fouling can be measured and the factors that may affect fouling in the high-temperature heater. These topics are reviewed in sections 2.6.1 and 2.6.2. A correlation between the measurement of fouling in the pilot plant and in the industrial milk plant is reviewed in section 2.6.3.

2.6.1 Fouling measurement

Fouling in heat exchangers can be measured by a range of experimental methods as follows.

Deposit weight

The deposit formation in a high-temperature heater can be weighed to determine the severity of fouling by whole milk (Lalande *et al.*, 1984; Mottar & Moermans, 1988; Patil & Reuter, 1988; Grandison, 1988b; Foster *et al.*, 1989). A higher weight of deposit collected in a given time indicates more severe fouling.

Pressure loss

Increase in pressure loss on the product side with time in the high-temperature heat exchanger was used to monitor the severity of fouling in a milk plant (Bell & Sanders, 1944; Burton, 1966a, 1968; Grandison, 1988b; Kastanas *et al.*, 1995b).

Heat transfer coefficient

The measurement of rate of decrease in the overall decreasing heat transfer coefficient was used by Kastanas *et al.* (1995a,b) and Lewis & Heppell (2000) to measure rate of fouling in a heat exchanger.

Temperature difference

Newstead *et al.* (1999) used increase in temperature difference across the heat exchanger surface to measure fouling in the high-temperature heater. Under constant flows of heating medium and product, and controlled product inlet and outlet temperatures, as the surface of the heat exchanger fouled the heat-transfer capability of the heat exchanger decreased. The product temperature was maintained by increasing the temperature of the heating medium, thus increasing temperature difference across the heating surface.

2.6.2 Factors affected UHT fouling

When fouling occurs during milk processing, it can be influenced by various factors. These factors are reviewed in the following.

Induction period

An induction period is the time for the initial layer of fouling to deposit on the surface of the stainless steel (Lewis & Heppell, 2000). In general, an induction period was suggested to depend upon the condition of the surface: roughness, material and cleanliness (Lund & Bixby, 1975). Kastanas *et al.* (1995b) reported that an induction period was shorter for aged raw fresh whole milk, compared with the initial raw fresh whole milk, when the milk was sterilized at 140 °C, 2 s. In some cases, there was no induction period for fouling of whole milk at 100 °C (Foster *et al.*, 1989).

The mechanism for the induction period was thought to involve the adsorption of protein and calcium phosphate onto the stainless steel (Delsing & Hindink, 1983). The role of fat during the induction period was not reported in the literature.

Milk temperature

The milk temperature at different heating stages affected the composition of the deposit (Burton, 1988). The proportion of protein in the deposit in the preheater was higher than in the deposit in the high-temperature heater, but the proportion of mineral deposit in the high-temperature heater was higher than that in the preheater (Burton, 1968; Lalande *et al.*, 1984; Patil & Reuter, 1988). However, only small proportion of fat in the deposit in the high-temperature heater of fresh whole milk was also reported.

Preheating

Preheating affected UHT fouling by whole milk (Bell & Sanders, 1944; Burton, 1968; Lalande *et al.*, 1984; Patil & Reuter, 1986a; Mottar & Moermans, 1988; Newstead *et al.*, 1999). These effects are discussed in the section 2.9.

Calcium concentration

The composition of fresh whole milk varies from batch to batch. Calcium is essential for the growth of the fouling layer from skim milk and whey (Delsing & Hindink, 1983; Hiddink *et al.*, 1986). A high calcium concentration in raw fresh whole milk gave more protein in the deposit in the plate heat exchanger at 85 °C, 15 s than low or normal calcium concentrations (Jeurnink, 1995b).

Milk pH

The reduction in milk pH from the natural fermentation of lactose to lactic acid or the addition of acid increased deposit formation whereas an increase in milk pH slightly decreased deposit formation at 140 °C (Skudder *et al.*, 1986; Kastanas *et al.*, 1995a). Burton (1965, 1968) noted that fresh whole milk with a low pH tended to give more deposit than milk with a high pH. However, the effect of variation in the natural milk pH on the deposit formation at 100 °C was small (Burton, 1965).

Stage of lactation, time of year and year

The amount of deposit varied with different milk seasons throughout the year, the stage of lactation, year, or time of year (Burton, 1967, 1968). The variation in the concentration of phospholipid may have been the cause of the variation in the amount of deposit formation on a hot platinum wire with different milk seasons with time of year and year (Burton,

1967, 1968). This result agreed with Grandison (1988b), who reported that in UHT fouling trials the amount of deposit from the same sources of milk varied with time of year.

β-lg variant

Hill *et al.* (1997a) and McKenna & Hill (1997) reported that the fouling rates for RCB and Recon varied with the genetic variants of β-lg, A, AB and B. The milks with variant A fouled 10 times faster than those with variant B. The fouling rates for milk with variant AB, equal amounts of variant A and B mixed together, are in between the fouling rates from variant A and those from variant B. In general, most cows in NZ are Friesian or Jersey, and commercial fresh whole milk is ~50 % variant A of β-lg and ~50 % variant B of β-lg (David Newstead, personal communication, 2003). The general proportions of variants A and B of β-lg in fresh whole milk in other countries (Australia, Italy and Canada) was in the range of 35-41 % variant A of β-lg and 61-65 % variant B of β-lg (Hill, 1993).

Flow characteristic

The greater flow velocity of milk can reduce the rate of deposition (Burton, 1968). This agreed with Lyster (1965), who reported that the rate of high-protein deposit formation in the high-temperature heater depends more on velocity gradients and turbulence in the milk than on the temperature gradients at the wall. However, the effect of flow velocity of milk on the deposit formation was less with increasing processing temperature and at 90 °C, there was no effect of velocity on the deposit formation (Burton, 1968). The effect of stirring rate on the deposit formation showed that the deposit mass at the heated surface of 100 °C decreased 20 % when the stirring rate in the model heat exchange apparatus was doubled (Foster *et al.*, 1989).

Milk aging

The amount of deposit decreased when the milk was aged (Burton, 1965). On the other hand, Jeurnink (1991), De Jong (1993) and Truong *et al.* (1996) reported that aging of fresh whole milk increased fouling. However, Kastanas *et al.* (1995b) suggested that the aged raw fresh whole milk is suitable for processing up to 12 days (if kept at 2 °C) before there was a significant increase fouling by raw fresh whole milk.

Types of UHT plant

Patil & Reuter (1986b, 1988) reported that the protein and ash contents in the deposit were not affected by the type of UHT plant (direct or indirect) and there was a reduction in deposit formation with increasing intensity of preheat treatment for both direct and indirect UHT plants.

Bubbles of air

If the pressure in the heat exchanger is low, bubbles form on the hot heat transfer surface and this can accelerate fouling. Jeurnink (1995a) reported that entrained air encouraged the formation of bubbles on the heating surface at 85 °C. These bubbles acted as nuclei for the accumulation of caseins and denatured serum proteins.

Type of surface material and coatings on the heating surface

The type of surface material showed no effect on fouling when Teflon, stainless steel, titanium, polysiloxane, and electropolished stainless steel were used to cover the plate heat exchanger surface (Yoon & Lund, 1994). This result agreed with Britten *et al.* (1988) who found that the interfacial properties of various polymer-coated surfaces did not influence the formation of deposit on stainless steel discs when the aged and degassed fresh whole milk was heated at 60 °C.

2.7 Correlation of fouling behaviour between a pilot plant and an industrial plant

The comparison of fouling rates between two commercial plants (one plate heat exchanger-based plant and one tubular heat exchanger-based plant) and a pilot plant was made using RCB prepared by the same procedure from the same milk powders (David Newstead, personal communication, 2003). Fouling rates from a commercial UHT plant (Steritherm, plate heat exchanger type) were measured in Saudi Arabia (Saudi Irish Dairy Co Ltd (Al Rabie), Riyadh, courtesy of the GM, P.M. Delahunty, 10-12 November 1990) with the flow rate of milk at 8,000 litre/h. The Stork “Sterideal” plant was a tubular heat exchanger plant, courtesy of Saudi Danish Dairy Co (Saudia), Jeddah, KSA, October 1995-1996 with the flow rate of 12,000 L/h. The NZDRI pilot plant was PHE (Alfa-Laval Type D P20-HB, 1979, Lund, Sweden, NZDRI Report MP91R16) with the flow rate of

milk at 120 kg/h., in which the plate heat exchanger was reconfigured to match a commercial "Steritherm" plant.

To investigate the correlation between the commercial plants and the pilot plant, fouling rates from the commercial plant were plotted against fouling rates from the pilot plant for a number of RCBs made with different skim milk powders. These powders were made using different process conditions and evaporator preheat treatments. The results are shown in Figures 2.2 and 2.3.

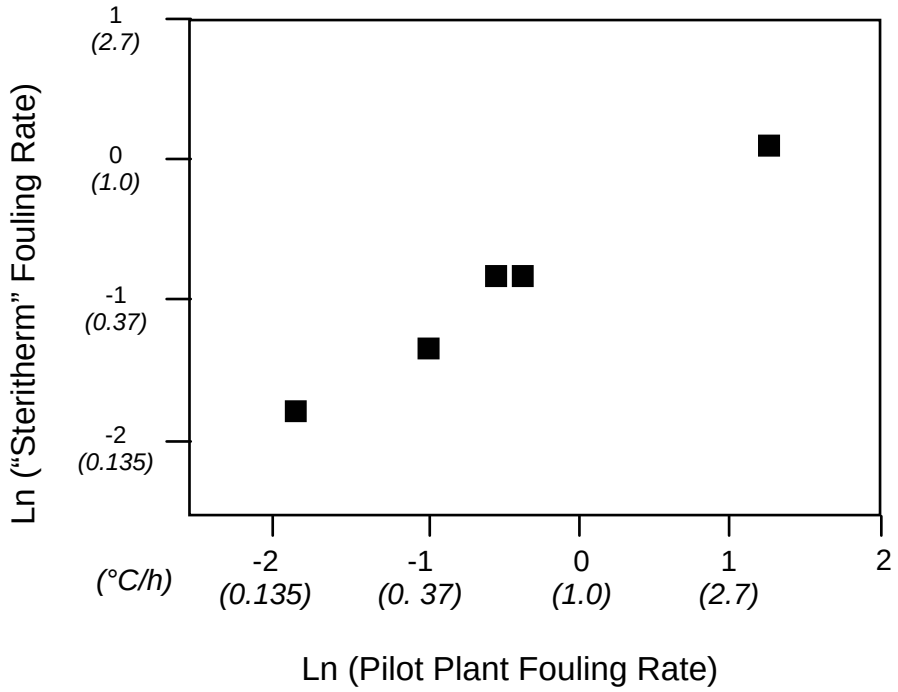


Figure 2.2 Fouling rates of RCB from NZDRI (the New Zealand Dairy Research Institute, the former name of Fonterra, Palmerston North)'s pilot plant versus a commercial plate heat exchanger based plant (David Newstead, personal communication, 2003).

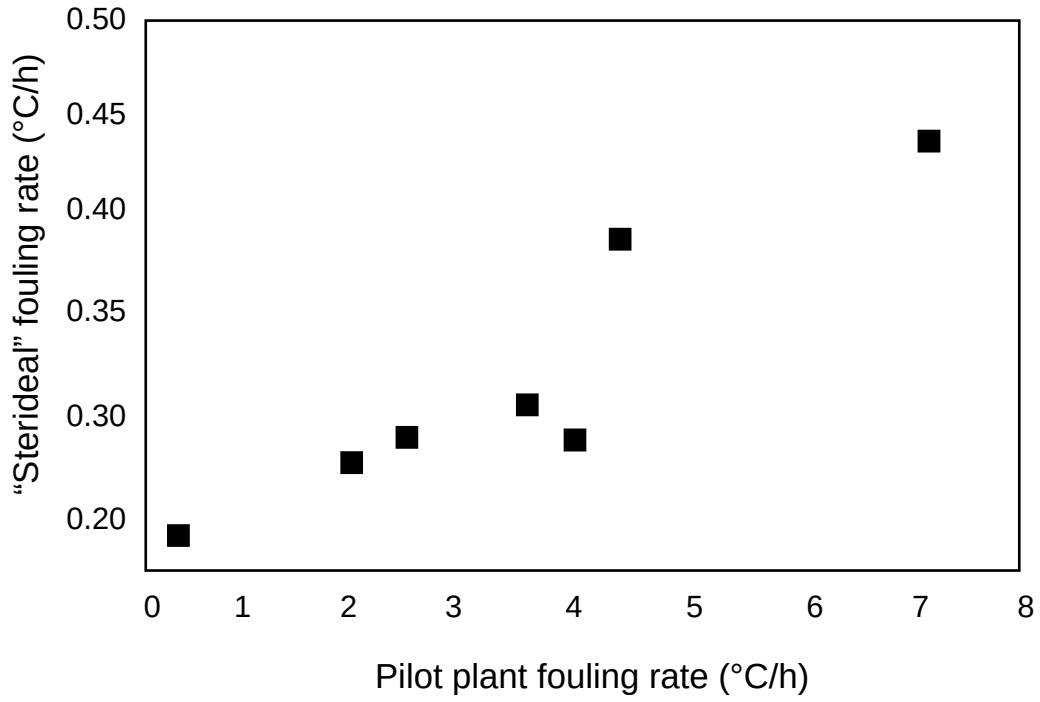


Figure 2.3 Fouling rates of RCB from NZDRI (the New Zealand Dairy Research Institute, the former name of Fonterra, Palmerston North)'s pilot plant versus a tubular heat exchanger based plant (David Newstead, personal communication, 2003).

The correlation between the fouling rates from the pilot plant and the commercial plants showed that fouling rates from the pilot plant adequately reflected the general behaviour of the commercial plants when tested with a variety of commercial milk preparations (r^2 = 0.91, 3 df for Figure 2.2 and r^2 = 0.85, 5 df for Figure 2.3, NZDRI Report FS97R14).

2.8 Deposit composition

Burton (1968) and Lyster (1965) reported two types of milk deposit. The first type of deposit, type A, formed at temperatures above 75 °C, but lower than 120 °C, was soft, white and voluminous and consisted of 50-70 % protein, 30-40 % minerals and 4-8 % fat. The second type of deposit, type B, formed at temperatures above 120 °C, was grey in colour, brittle and gritty. Type B deposit consisted of 70-80 % minerals, 10-20 % protein and 4-8 % fat. It is noted that type B deposit was the deposit of interest in the present study.

For fresh whole milk, the maximum amount of deposit for initially raw fresh whole milk was found in the high-temperature heater and the deposit was type B deposit (Lyster, 1965; Lalande *et al.*, 1984; Tissier *et al.*, 1984; Patil & Reuter, 1986a, 1988). This result differed from the deposit from RCB and Recon. Newstead *et al.* (1999) reported the composition of the deposits from RCB (3 milk batches) and Recon (2 milk batches). The deposit composition of RCB consisted of fat 40-64 % w/w dry matter (DM), protein 16-18 % w/w DM and minerals 8-18 % w/w DM. The composition of the deposit for Recon consisted of fat 6-30 % w/w DM, protein 34-38 % w/w DM and minerals 15-40 % w/w DM.

2.9 The effect of preheat treatments on UHT fouling by fresh, recombined and reconstituted whole milk

There are differences in the number of process heat treatments and in the composition of the MFGM among fresh whole milk, RCB and Recon. Thus, the effect of UHT preheat treatment on UHT fouling by fresh whole milk, RCB and Recon and the effect of evaporator preheat treatment on UHT fouling by RCB and Recon are reviewed in sections 2.7.1 and 2.7.2, respectively.

2.9.1 Effect of UHT preheat treatment on UHT fouling by fresh, recombined and reconstituted whole milk

Fresh whole milk

Bell & Sanders (1944), Burton (1968), Patil & Reuter (1986a, 1988) and Mottar & Moermans (1988) reported that more severe UHT preheat treatment reduced the deposit formation by raw fresh whole milk in the high-temperature heater. This trend differs from the results of Newstead *et al.* (1999) and Newstead (personal communication, 2003).

Lalande *et al.* (1984) did not, in fact, truly investigate the effect of preheating on UHT fouling. They merely found that the rate of fouling by pasteurized fresh whole milk in the heating sections of their UHT plant was lower than the rate of fouling by raw fresh whole milk in their pasteurizer (which they referred as a preheater). They implied that, because less fouling occurred under the relatively severe conditions in their UHT plant than in their pasteurizer, the heat treatment in the pasteurizer must have had an anti-fouling effect. However, they conducted no control experiments to check this.

There were differences in the trends of the effect of preheat treatment on fouling by fresh whole milk in the high-temperature heater between Newstead *et al.* (1999) and Newstead (personal communication, 2003), and Bell & Sanders (1944), Burton (1968), Patil & Reuter (1986a, 1988), and Mottar & Moermans (1988).

- Firstly, there are differences in the method of fouling measurement. Newstead *et al.* (1999) and Newstead (personal communication, 2003) used temperature difference to measure the fouling rates in the high-temperature heater, but the measurement of total deposit weight in the high-temperature heater was used by Mottar & Moermans (1988) and Patil & Reuter (1986a, 1988) reported that the percentage (w/w dry matter) of protein and fat in the deposit from raw fresh whole milk decreased with increasing UHT preheat treatment whereas the percentage (w/w dry matter) of ash, calcium and phosphorus in the deposit increased with increasing UHT preheat treatment.
- Secondly, there were differences in the processing steps, the order of homogenization and preheat treatment or homogenization itself. Newstead *et al.* (1999) and Newstead (personal communication, 2003) reported that homogenization plays a key role in UHT fouling. In one trial out of three trials the fouling rate of preheated and then homogenized fresh whole milk increased with increasing UHT preheat treatment. When fresh whole milk was not homogenized at all, in two trials out of three, fouling rate increased with the higher UHT preheat treatment. Mottar & Moermans (1988) reported that the total deposit weight of preheated and homogenized raw fresh whole milk in the high-temperature heater at 140 °C decreased when the milk was preheated from 70 °C to 90 °C: this result was duplicated.
- Thirdly, there were differences in milk pasteurization treatment. Newstead *et al.* (1999) used pasteurized fresh whole milk as the initial milk for UHT processing, but raw fresh whole milk was used by Patil & Reuter (1986a, 1988), Mottar & Moermans (1988) as the initial milk for UHT processing.
- Fourthly, only one replicate of raw fresh whole milk appears to have been used in some of these experiments (Bell & Sanders, 1944; Burton, 1968; Patil & Reuter, 1986a, 1988). It was not clear whether control treatments and higher preheat treatment trials were carried out on the same batch of fresh whole milk. Duplicates

of raw fresh whole milk were used by Mottar & Moermans (1988) whereas 3 or more batches of milk were used in each trial by Newstead *et al.* (1999) and Newstead (personal communication, 2003).

Recombined whole milk

Newstead *et al.* (1999) and Newstead (personal communication, 2003), found that in four out of five fouling trials with RCB (prepared from different batches of SMP), the fouling rate increased with the higher UHT preheat treatment (increased from 75 °C, 3 s to 90 °C, 120s). They speculated that heating and processing steps during skim milk powder manufacture influenced the fouling rate.

Newstead *et al.* (1999) also reported that the fouling rate of reconstituted skim milk was lower than the fouling rate of RCB when the same SMP was used. The degree of this reduction was dependent on milk batch. However, the fouling rate of these reconstituted skim milks also increased with the intensity of UHT preheat treatment for all five fouling trials.

Reconstituted whole milk

Newstead *et al.* (1999) and Newstead (personal communication, 2003) reported that the fouling rate in two out of three fouling trials with Recon increased with the intensity of UHT preheat treatment and in the third fouling trial at low preheat treatment the fouling rate was equivalent to that in the high preheat treatment trial. They suggested that heating and processing steps during whole milk powder manufacture resulted in an increase of fouling rate with increasing UHT preheat treatment.

2.9.2 Effect of evaporator preheat treatment on UHT fouling by recombined and reconstituted whole milk

The studies on the effect of evaporator preheat treatment on UHT fouling by RCB were reported by Smith (1992) and Harnett *et al.* (1997) and by Recon were reported by Lean *et al.* (1996), Hill *et al.* (1997a) and Armstrong *et al.* (1998). They reported that the greater preheat treatment prior to evaporation resulted in the greater fouling rate of RCB and Recon. The fouling rate for RCB and Recon from β-lg variant AB was greater than the fouling rate from β-lg variant B, but lower than the fouling rate from β-lg variant A. In

some cases, the fouling rates for RCB and Recon from β-lg variant B were about 1/10 of the fouling rate from β-lg variant A.

2.10 Mechanisms for the deposition of fat, protein and ash for fresh, recombined and reconstituted whole milks at the high-temperature heater

Mechanisms for the deposition of fat, protein and ash in the high-temperature heater appear to relate to heat-induced changes in the fat, protein and minerals during heating upstream of the high-temperature heater and in that heater itself. In this section, attempts are made of deriving mechanism broadly reviewed in terms of chemistry and physical effects. General fouling mechanisms of milk are summarised below.

Adsorption of whey protein aggregates on to the heated surface of the heat exchanger

It was suggested that denatured β-lg and α-la played a key role during the growth of the foulant layer on the heated surface when the milk was heated above 65 °C (Lyster, 1965; Tissier *et al.*, 1984; Lalande *et al.*, 1985; Maas *et al.*, 1985; Burton, 1988; Visser *et al.*, 1997; Visser & Jeurnink, 1997). During heating, these denatured whey proteins may associate with casein micelles or whey protein in the MFGM or with milk protein in the milk plasma (Smits & Vanbrouwershaven, 1980; Singh & Creamer, 1991a; Oldfield *et al.*, 1998b). The adsorption of the whey protein-casein micelle complexes to the heating surface at 65 °C to 85 °C was due to the deposition of whey protein (Itoh *et al.*, 1995). The adsorption of β-lg and α-la on the heated surface of stainless steel increased with temperature in the range of 50-140 °C (Hegg *et al.*, 1985).

Involvement of fat and protein

Fat was reported to play only a minor role in fouling (Visser *et al.*, 1997; Visser & Jeurnink, 1997). Fat was coincidently entrapped in the protein network of the foulant layer during the fouling process (Burton, 1968; Lalande *et al.*, 1984; Visser & Jeurnink, 1997).

Decrease in the solubility of calcium phosphate

There were two steps of mineral fouling in the high-temperature heater (Burton, 1968). The first stage was the reduction in the solubility of the milk salts at high temperature and

the second stage involved the slow formation of crystal nuclei and the growth of the nuclei into crystals which deposited in the high-temperature heater. The rate of mineral deposition was influenced by the deposition of protein at the heated surface (Lalande *et al.*, 1984).

The solubility of calcium phosphate salts decreased with increasing milk temperature and present in milk as a precipitate (Visser *et al.*, 1997; Visser & Jeurnink, 1997). The precipitation of calcium phosphate played a key role in the fouling process when the temperatures were greater than 120 °C (Burdett & Burton, 1974; Delsing & Hiddink, 1982; Sandu & Lund, 1985; Hiddink *et al.*, 1986; Skudder *et al.*, 1986; Tissier & Lalande, 1986).

Visser *et al.* (1997) suggested that calcium phosphate in milk can precipitate as such or deposit onto the surface of casein micelles and/or β-lg in the milk serum. The composition of this precipitate was suggested by Visser *et al.* (1997) to be a mixture of calcium phosphate dehydrate ($CaHPO_4.2H_2O$) and Octacalcium phosphate ($Ca_8H_2(PO_4)_6.5H_2O$). This result was different from Burton (1968) and Lyster (1965), who suggested that the form of mineral in the deposit of fresh whole milk was in the form of β-$Ca_3(PO_4)_2$, in which the ratio of Ca/P is 1.5 which was transformed into hydroxyapatite ($Ca_5OH(PO_4)_3$), giving a ratio of Ca/P of 1.6 when the precipitate was subjected to prolonged heating.

The forms of calcium phosphate in the deposit of whole milk, brushite ($CaHPO_4.2H_2O$), monetite ($CaHPO_4$); octacalcium phosphate ($Ca_8H_2(PO_4)_6.5H_2O$, whitlockite (β-$Ca_3(PO_4)_2$ and hydroxyapatite ($Ca_5OH(PO_4)_3$) and brushite and octacalcium phosphate are the forms that precipitate most easily (Sandu & Lund, 1985). A mixture of hydroxyapatite and amorphous calcium phosphate are commonly found in the micellar colloidal calcium phosphate of fresh whole milk (Sandu & Lund, 1985).

In addition to the deposition of calcium phosphate in the heat exchangers, calcium citrate is also involved in the deposition (Jeurnink *et al.*, 1996b; Visser & Jeurnink, 1997). There was no report in the literature for the deposition of calcium citrate by whole milks.

2.11 UHT plant fouling mechanisms as related to the effect of preheating on fresh, recombined and reconstituted whole milks

Bell & Sanders (1944), Burton (1968), Patil & Reuter (1986a, 1988), and Mottar & Moermans (1988) reported the reduction of UHT fouling with increasing UHT preheat treatment and that fouling resulted in type B deposit in the high-temperature heater. The mechanism of the effect on UHT fouling of preheating for raw fresh whole milk was still unclear. Patil & Reuter (1988) reported that the greater UHT preheat treatment resulted in a decrease of the percentages of fat and protein and an increase of the percentage of ash in the deposit of fresh whole milk in the high-temperature heater. They suggested that there was a reduction in the solubility of minerals with increasing milk temperature and that led to the deposition of minerals in the high-temperature heater. The higher preheat treatment resulted in the lower denaturation of whey protein and the lower deposition of protein in the high-temperature heater. Fat was trapped in the network of deposited protein.

Newstead *et al.* (1999) and Newstead (personal communication, 2003) pointed that there are differences between the MFGMs of fresh whole milk, RCB and Recon and these may relate to the different responses in fouling rate for those three whole milks.

Chapter 3
Materials and methods

3.1 Milk

Raw fresh whole milk for use in trials was collected by milk tanker from dairy farms in the Manawatu collection area of the Fonterra Co-operative Group, New Zealand.

3.2 Pasteurisation

All milk was pasteurised at 72 °C for 15 s on reception at the Fonterra pilot plant, Palmerston North. Then, the pasteurised milk was kept at 4 °C, but not held for more than 48 hours as the fouling trials progressed. Zero hour was taken as 5 pm on the date that the milk was received. Four fouling trials with fresh milk was the maximum that could be carried out with a single batch of fresh milk within 48 hours.

3.3 Standardisation

3.3.1 Fresh skim milk

After the fresh milk was pasteurised, it was separated into cream and skim milk. Part of fresh skim milk was standardised to 8.81 % total solids and sent for drying into skim milk powder (SMP) at the Fonterra milk powder pilot plant, Palmerston North.

3.3.2 Fresh whole milk

Standardised FWM with a composition of 3.50 % milk fat and 8.50 % non-fat milk solids (12.00 % TS) was prepared by recombining cream and skim milk after separation (plus a little water may require to bring the final TS to 12.00 %) (Figure 3.1). Homogenization by a nozzle homogenizer was applied to the feed FWM prior to UHT processing.

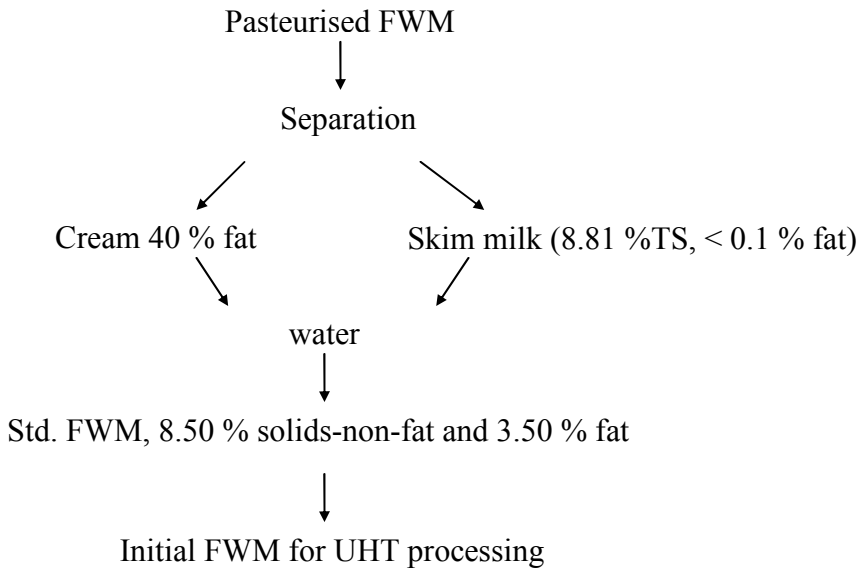


Figure 3.1 Process diagram for standardisation. FWM = pasteurized, standardized and homogenized whole milk for fouling trials.

3.4 Milk powder manufacture

Standardised fresh skim milk or fresh whole milk was sent to the milk powder pilot plant for drying into skim milk powder (SMP) or whole milk powder (WMP). The drying process is shown in Figure 3.2.

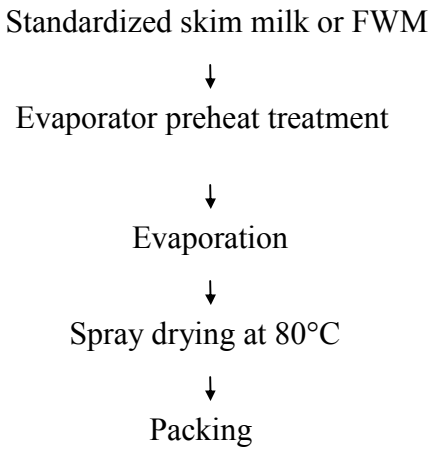


Figure 3.2 Process diagram for milk powder production.

Three different evaporator preheat treatments were applied to skim milk: 75 °C, 2 s, 85 °C, 155 s or 95 °C, 155 s and one to FWM: 95°C, 33 s. The resulting skim milk powders were called SMP1, SMP2 and SMP3. Then, the milk was concentrated to ~50 % TS on a Wiegand three-effect falling-film evaporator and spray dried using pressure nozzle

atomization. The final moisture content of the skim and whole milk powders was 3-4 % w/w. Then, milk powder was filled in 25 kg bags and kept in a dry store at 22 °C to 25 °C.

3.5 Recombination and reconstitution

Skim milk powder was reconstituted with water and recombined with milk fat to make recombined milk (RCB), and whole milk powder was reconstituted in water to make reconstituted milk (Recon) as shown in Figure 3.3.

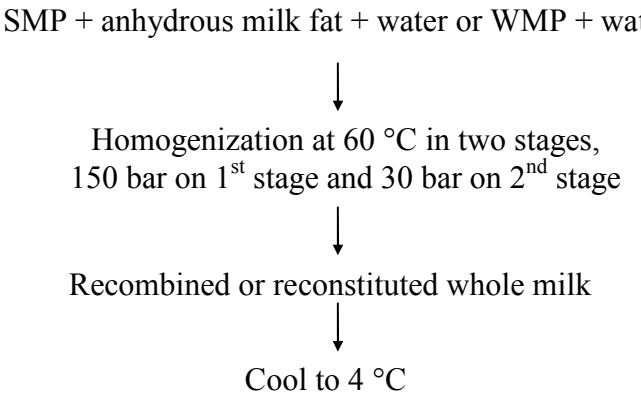


Figure 3.3 Process diagram for recombination and reconstitution of milk powders.

RCB and Recon were subjected to homogenization during recombination and cooled to 4°C before UHT processing.

3.6 UHT pilot plant 1

An indirect UHT pilot plant, Alfa-Laval Type D P20-HB, 1979, Lund, Sweden, was used to measure fouling rate for Milk no.1. It is named "UHT plant 1". The milk flow rate was controlled at 120 L/h. A schematic plant diagram is shown in Figure 3.4. The milk outlet temperature from the preheater was manually controlled at the desired temperature, 75, 85, or 95 °C, and the milk was held for the required holding time in the preheating holding tube. Then, the milk was heated up in the regeneration section before the milk entered the high temperature heating section. The milk outlet temperature from this section was automatically controlled at 140 °C and the milk was held for 8 s in the UHT holding tube prior to cooling. T2, T4, T5 and T6 (Figure 3.5) were temperature sensors (platinum 100/RTD-2, fast response temperature probes) connected to a data logger.

Flows in the heat exchanger were counter-current. The heat exchanger was equipped with temperature sensors (platinum resistance 100/RTD-2, fast response temperature probes) to measure the temperature of milk and hot water. Details of flows in the plate heat exchanger are shown in Figure 3.5.

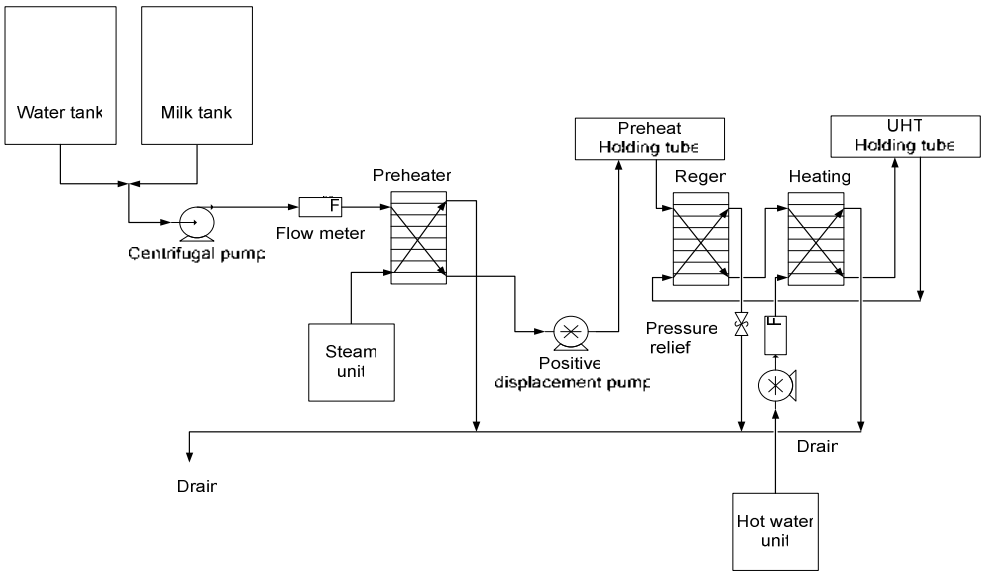


Figure 3.4 Simplified UHT plant1 diagram (Product contact surfaces were of stainless steel).

Before the plant was started up, the required holding tubes for preheating and sterilization were connected to the UHT plant. The plant was run with water until product side outlet temperatures reached steady state and were close to the set points. Then, the milk outlet temperatures from the preheater, intermediate heater and high-temperature heater were finely adjusted to 75 or 95 °C, 126 °C and 140 °C. The control system for the high-temperature heater increased the hot water inlet temperature to control the temperature of hot milk outlet temperature at 140 °C. When the plant reached steady state, milk processing started for two processing hours. After two processing hours, the plant was rinsed with hot water until the intermediate heater and high-temperature heater product side outlet temperature were 85 °C.

The plant was cleaned by adding 1,300 ml NaOH and 500 ml Stabicip ZN (a commercial acid solution) to the balance tank of hot water (83-85 °C) to give a concentration of 1.5 ± 0.2 %. The product side flow rate was increased to 400 L/h and the cleaning solution circulated for at least 25 min. Then, the plant was flushed with hot water for 5 min. before

adding 1,000 ml Nitric acid to the balance tank of hot water (60-65 °C) to give a concentration of 1.0 ± 0.2 %. The acid solution was circulated at 400 L/h for 15 min. Finally, the plant was flushed with hot water (circulated for 15 min.) before the plant was shut down.

When the plant was properly cleaned, temperature difference (T4 - T2) in the high-temperature heating section (Figure 3.5) was below 0.5 °C. No fouling trial was carried out if (T4 - T2) was greater than 0.5 °C.

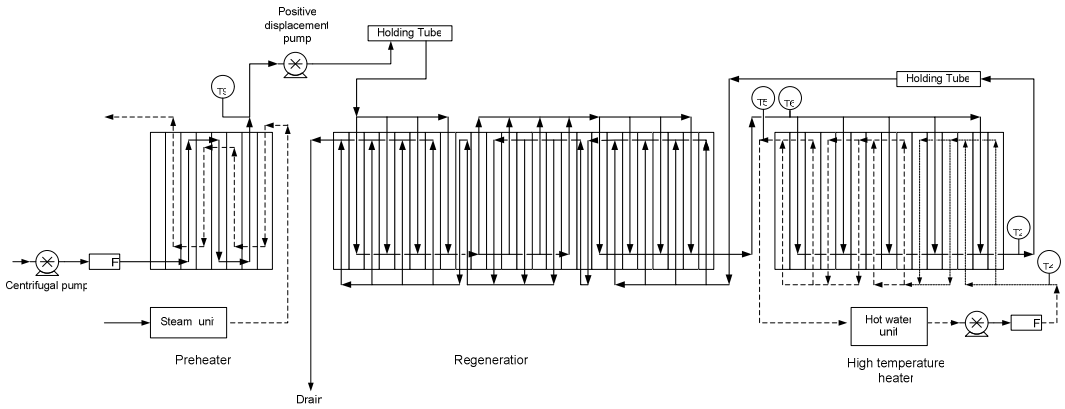


Figure 3.5 Flow patterns in the first three sections of the plate heat exchanger in UHT plant 1 (Product contact surfaces were of stainless steel).

T2, T4, T5, T6 and T9 were logged by a data logger. The slope of the plot of temperature difference (hot water inlet temperature (T4) - milk outlet temperature (T2)) and in the high-temperature heating section against time was taken as the fouling rate (°C/h). (See section 3.8 and Chapter 4).

3.7 UHT pilot plant 2

The second UHT pilot plant, used for most of the experimental work, was purpose-built for sub-commercial sample production, and had the following characteristics: flow rate 120 L/h was measured with an in-line electromagnetic flow meter, working temperature up to 150 °C, with temperature independently controllable in all three heating sections, variable holding tubes for preheating and for sterilisation, and fully instrumented, with data logging. The stainless steel spiral tubes are used as temperature-controlled holding tubes. The tank consisted of different inlet positions in order to be able to vary the heating holding time. This holding tube was designed to be able to hold the milk temperature for

up to 3 minutes. The heat exchanger (Alfa-Laval type Clip3 R, TetraPak, Sweden) comprised five sections: preheater, intermediate-temperature heater, high-temperature heater, precooler and final cooler. Each plate heat exchanger was 0.1 m wide and 0.54 m long. The plant is named "UHT plant 2".

A schematic flow diagram is shown in Figure 3.6. In the first four sections, milk outlet temperatures were under automatic control. The final cooling section was supplied with chilled water. A detailed flow diagram of the first three sections is shown in Figure 3.7. Flow within the plate heat exchanger was mainly counter-current. Each section was equipped with temperature sensors to measure the inlet and outlet temperatures of the milk and water.

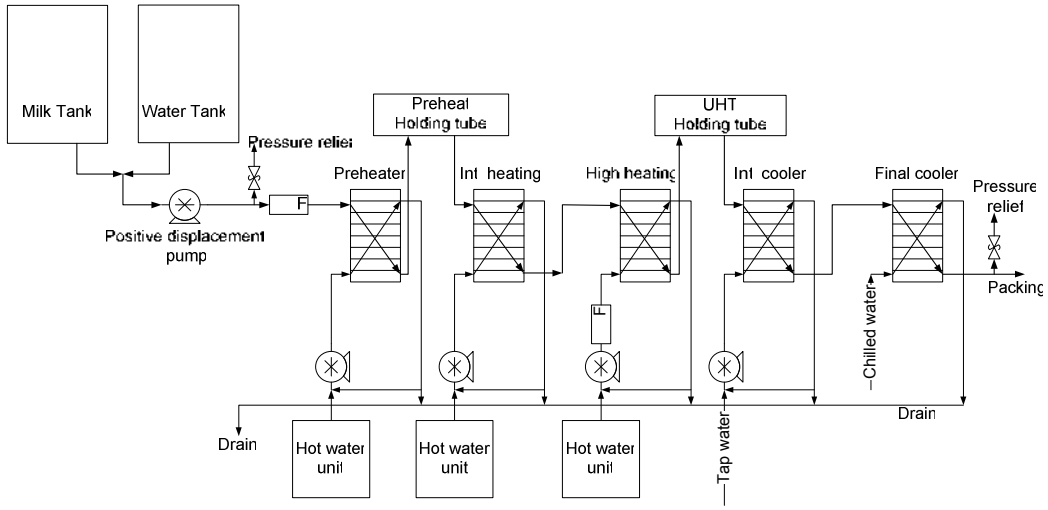


Figure 3.6 Simplified UHT plant 2 diagram (F = flow meter).

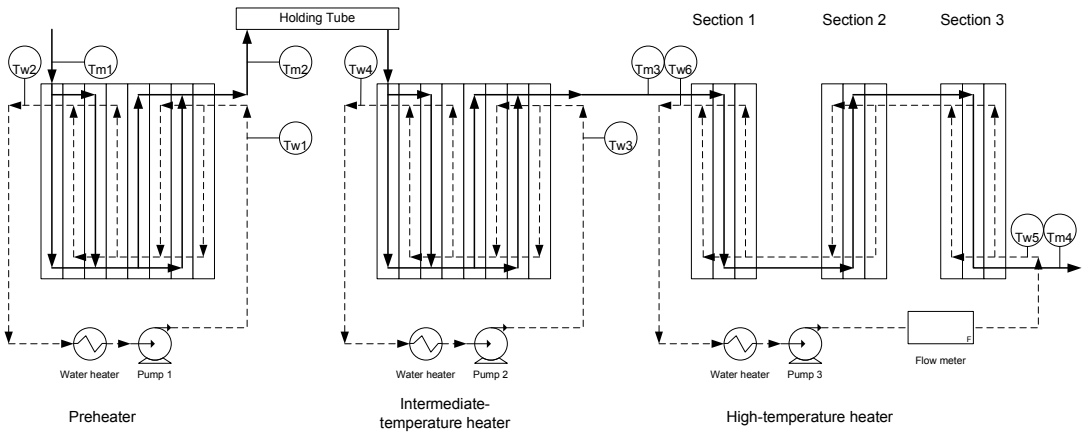


Figure 3.7 Flow patterns in the first three sections of the plate heat exchanger of UHT plant 2 (------water; —— milk).

The plant was started on water, and switched to milk once temperature control had been established. The start of the fouling run was taken as the time at which the milk, displacing the water, reached full concentration in the high-temperature heater. Temperature and flow-rate data were logged at 5 s intervals. The preheat treatment under study was applied in the first, preheating section, followed by the preheating holding tube. The milk temperature was then raised to 126 °C by the intermediate heater, and fouling measured in the high-temperature heater, in which the temperature was raised from 126 °C to the final sterilisation temperature of 140 °C. Holding at sterilisation temperature was for 8 s prior to cooling in the final two sections. Fouling rate in the high-temperature heater was determined as the rate of change in the heat-transfer characteristics of the high-temperature heater as indicated by the temperature differential (ΔT) at the product outlet (ΔT = hot water inlet temperature (Tw_5, Figure 3.7) minus the milk outlet temperature (Tm_4, Figure 3.7). The milk and hot water flow rates, and the milk inlet and outlet temperatures were controlled to constant values.

A CIP baseline trial was done to check the cleanliness of the high-temperature heating section before and after a fouling trial. It was found that the initial temperature difference in the high-temperature heating section (Tw5 - Tm4, Figure 3.7) in the range 0.4-0.5 °C represented a clean plate heat exchanger. If not, a CIP was applied to decrease the temperature difference until it reached 0.4-0.5 °C before another fouling trial was started.

3.8 Measurement of fouling rate

Fouling rate was evaluated as the rate of increase in ΔT, as defined above in section 3.7. The fouling rate, FR, for a run was determined as the slope of the linear regression of ΔT against time (Figure 3.8) over the period of the run as follows:

$$\Delta T = T_{w5} - T_{m4} \qquad \text{Eq. 3.1}$$

where $T_{w5} = \textit{hot water inlet temperature} \left(°C\right)$

$T_{m4} = \textit{milk outlet temperature} \left(°C\right)$

Fouling rate was calculated from the regression equation.

$$y^* = mt + C_3 \qquad \text{Eq. 3.2}$$

where $y^* = \textit{predicted } \Delta T$

$m = \textit{fouling rate } (°C/h)$

$t = time\ from\ start\ of\ run\ (h)$

$C_3 = Best - fit\ \Delta T\ at\ start\ of\ run$

The basis of calculating fouling rate in this way is explained in Chapter 4. The example of fouling rate measurement is presented in Figure 3.8.

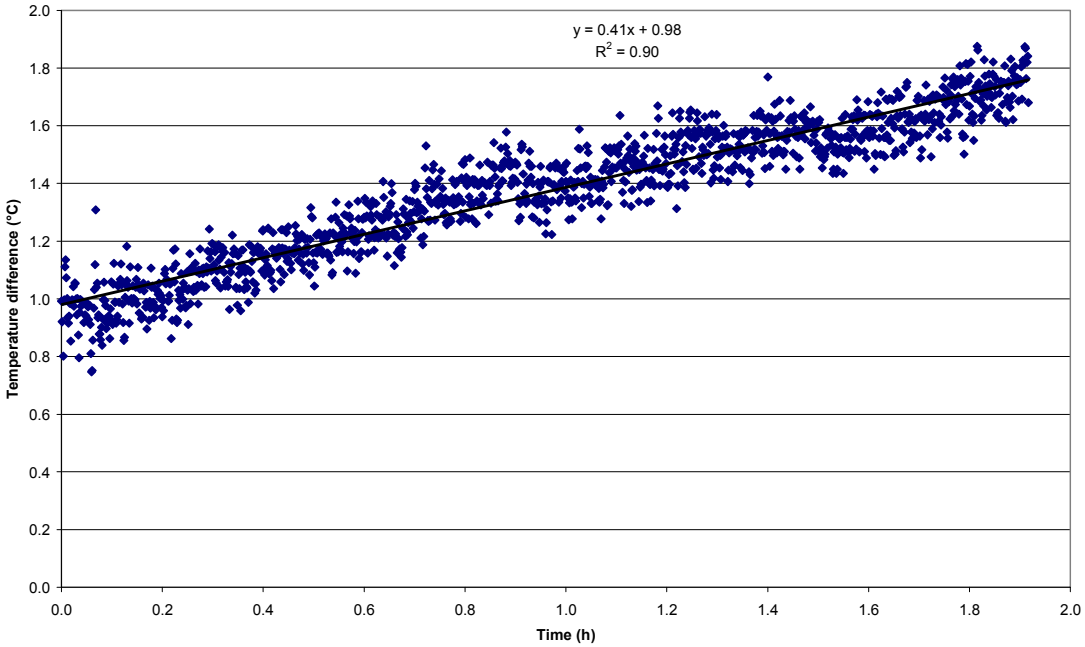


Figure 3.8 Plot of temperature difference with time from the high-temperature heater of RCB which preheated at 95 °C, 147 s (Milk No. 8). Fouling rate is 0.41 °C/h.

The data in Figure 3.8 yielded a fouling rate of 0.41 °C/h. These calculations were applied to all data obtained in fouling trials with UHT plant 2.

ΔT in the Eqs. 3.1 and 3.2 can be calculated from;

$$\phi = UA\Delta\theta_{lm} = (mc\Delta\theta)_{milk} = (mc\Delta\theta)_{water} \qquad \text{Eq. 3.3}$$

where $$U = \frac{1}{\frac{1}{h_{milk}} + \frac{x}{\lambda} + \frac{1}{h_{water}} + R} = overall\ heat\ transfer\ coefficient$$

where $\Delta\theta_{lm} = Temperature\ driving\ force\ for\ heat\ transfer\ (°C)$

$\Delta\theta = Temperature\ change\ (°C)$

$h_{milk} = convective\ heat\ transfer\ coefficient\ of\ milk\ (W/(m^2.K))$

$h_{water} = convective\ heat\ transfer\ coefficient\ of\ water\ (W/(m^2.K))$

$R = fouling\ factor\ (m^2.K/W)$

$x = wall\ thickness\ (m)$

$\lambda = thermal\ conductivity\ (W/(m.K))$

$$\Delta\theta_{lm} = \frac{\Delta\theta_1 - \Delta\theta_2}{\ln\left(\frac{\Delta\theta_1}{\Delta\theta_2}\right)} = log\ mean\ temperature\ difference \qquad \text{Eq. 3.4}$$

where $\Delta\theta_1 = \theta_{water\ in} - \theta_{milk\ out} = \Delta T$

$\Delta\theta_2 = \theta_{water\ out} - \theta_{milk\ in}$

The milk inlet and outlet temperatures and the milk and water flow rates in the high-temperature heater were automatically controlled. These equations show that as the extent of fouling increases U decreases, and the milk outlet temperature control system will keep increasing $\Delta\theta_{lm}$ (by increasing Tm4 ; Figure 3.7) to maintain ϕ to keep the milk outlet temperature (Tm5 ; Figure 3.7) constant at 140 °C. Thus the rate of increase in $\Delta\theta_{lm}$ could have been taken as the fouling rate. However, $\Delta\theta_{lm}$ is the average temperature driving force across the high-temperature heater whereas most of the fouling deposit accumulated near the milk outlet of this heat exchanger. For this reason, the rate of change of (Tw4 – Tm5) was taken as the fouling rate. This approach is fast and adequate, and can easily be used in an industrial UHT plant.

The uncertainty of fouling rate is demonstrated using FWM at the 95°C, 147 s preheat treatment (Table A1.1, Milk No. 2).

$$Uncertainty = Arithmetic\ mean \pm (2 * standard\ deviation)$$

$$= \left(\frac{0.21 + 0.26}{2}\right) \pm (2 * 0.04)$$

$$= 0.24 \pm 0.08\ \text{°C/h}$$

3.8.1 Fouling deposit recovery

The milk was processed with the UHT plant at a steady state. The process run time was 2 h. The plant was then switched to water, the flow rate was reduced from 120 L/h to 50-60 L/h and the plant rinsed for 8 min when the plant was fitted with the 75 °C preheat treatment configuration and for 10 min when the plant was fitted with the 95 °C preheat treatment configuration. The plate heat exchanger was dismantled. The high-temperature heating section of the heat exchanger was dismantled; the plates left at room temperature

(18-20 °C) for 5 min to drain off the water and the plates weighed. Then, the plates were dried at 55 °C in an oven for 15 min. The plates were weighed again. Then, the deposit was scraped off for mass measurement and composition analysis. The process diagram is shown in Figure 3.9.

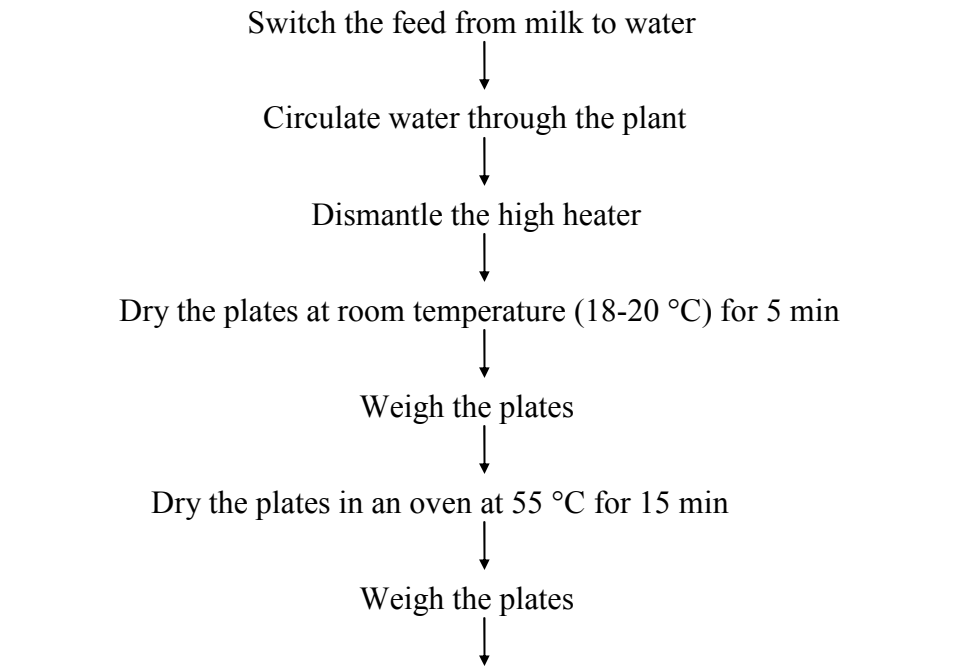


Figure 3.9 Regime of deposit sampling.

3.9 Experimental design

Twelve milk batches (milk numbers) were used in this work. They were used for different experimental purposes. Seven experimental designs were used in this work.

3.9.1 Milk Nos. 1, 2 and 3

After the pasteurized fresh whole milk (Milk Nos. 1, 2 and 3) was standardised to 3.5 % fat and 12.0 % solids-not-fat, it was used as the initial milk for UHT processing or for drying. The experiment design in Figure 3.10 consisted of three parts. The first concerned the effects of heat treatments (i.e. preheating) fresh milk prior to UHT processing. The second concerned the effects of preheating reconstituted WMP prior to UHT processing. The third concerned the effects of both pre-evaporation heat treatment (evaporator preheating) during the manufacture of SMP, and preheating the recombined milk derived from it prior to UHT processing.

A milk number was a batch of raw fresh whole milk as delivered from which sub-batches (after pasteurization and standardization) were used in fouling trials directly, or were used to make SMP and WMP. More milk numbers were treated as replicates or more correctly, repeated measures. They were not true replicates, but using them as replicates was realistic because batch to batch variation in the composition of milk is a reality. True replicates were not possible as large quantities of milk would have had to have been stored, and because of the limited storage life of pasteurized milk.

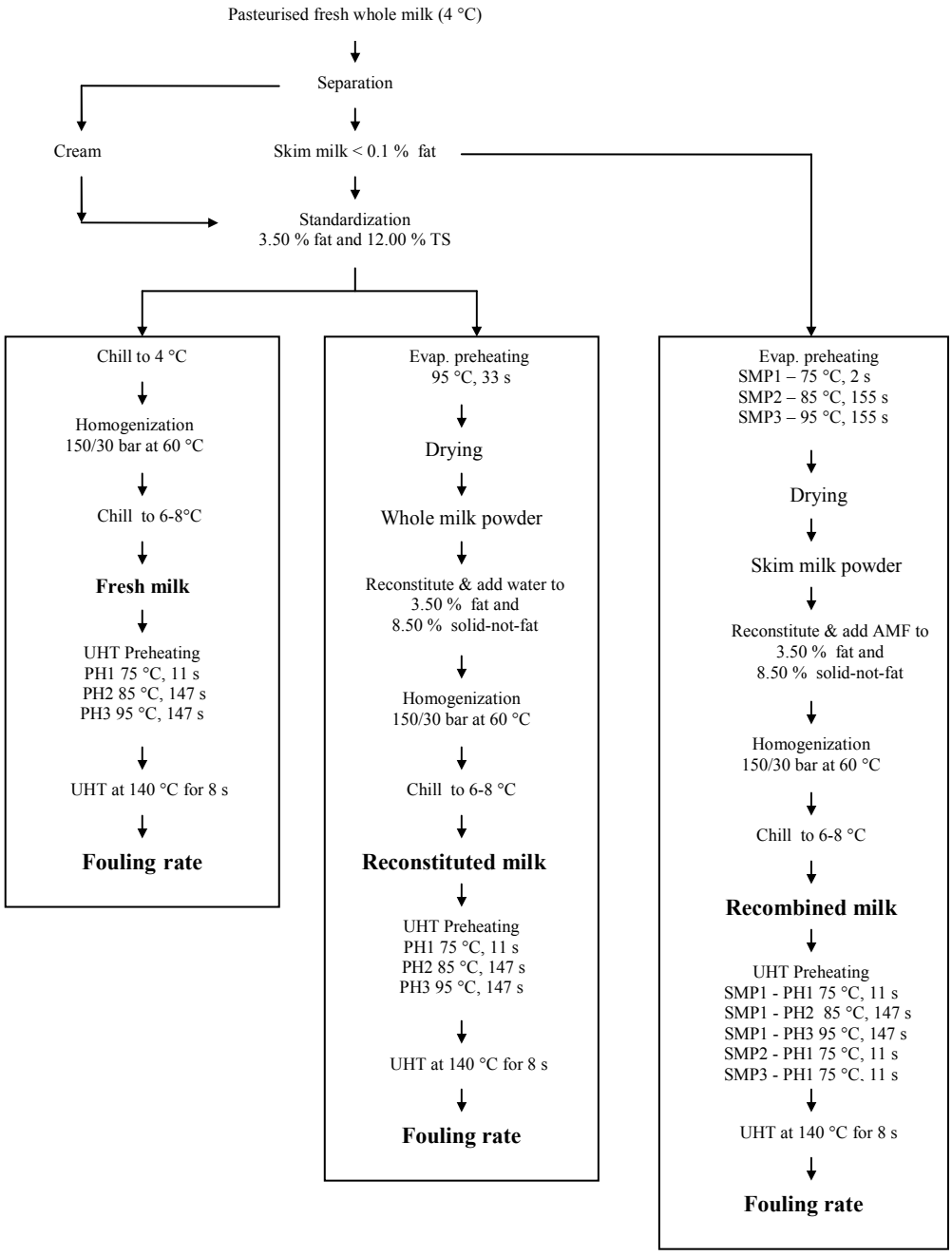


Figure 3.10 Experimental design for fresh whole milk (FWM), recombined milk (RCB) and reconstituted milk (Recon) (Milk Nos. 1-3).

This experimental design was applied to three milk numbers; milk No.1 at UHT plant 1 and milk Nos. 2 and 3 at UHT plant 2. In each experiment, FWM, RCB and Recon were all derived from the same batch of fresh whole milk. Skim milk was preheated using three different evaporator preheat treatments to give SMP1, 75 °C, 2 s; SMP2, 85 °C, 155 s; SMP3, 95 °C, 155 s. Recon was evaporator preheated at 95 °C, 33 s.

The rest of the batch of standardized fresh whole milk, was not used for whole milk powder drying, was used to determine UHT-plant fouling rates for each of the three UHT preheating conditions given in Figure 3.10. Two fouling trials were carried out the same day, and two on the following day after overnight storage of the milk (at 4 °C). On the second day, one of the preheating conditions (95 °C, 147 s UHT preheat treatment) was a repeat of one of the conditions used on the first day.

In this thesis the acronym FWM (fresh whole milk) is used to identify the pasteurized, standardized and homogenized whole milk subjected to UHT preheating and the subsequent stages of UHT processing, in order to distinguish it from recombined whole milk (RCB) and reconstituted whole milk (Recon) subjected to the same processing steps.

3.9.2 Milk Nos. 4 and 5

Pasteurised fresh whole milk (Milk Nos. 4 and 5) was dried to make whole milk powder using the process shown in Figure 3.11.

Pasteurised fresh whole milk was dried to make whole milk powder at two evaporator preheat treatments, called low-heat (LH) WMP, 75 °C, 2 s and high-heat (HH) WMP, 95 °C, 155 s.

Reconstituted LH WMP and HH WMP were each subjected to two UHT preheat treatments, 75 °C, 11 s and 95 °C, 147 s, during UHT processing.

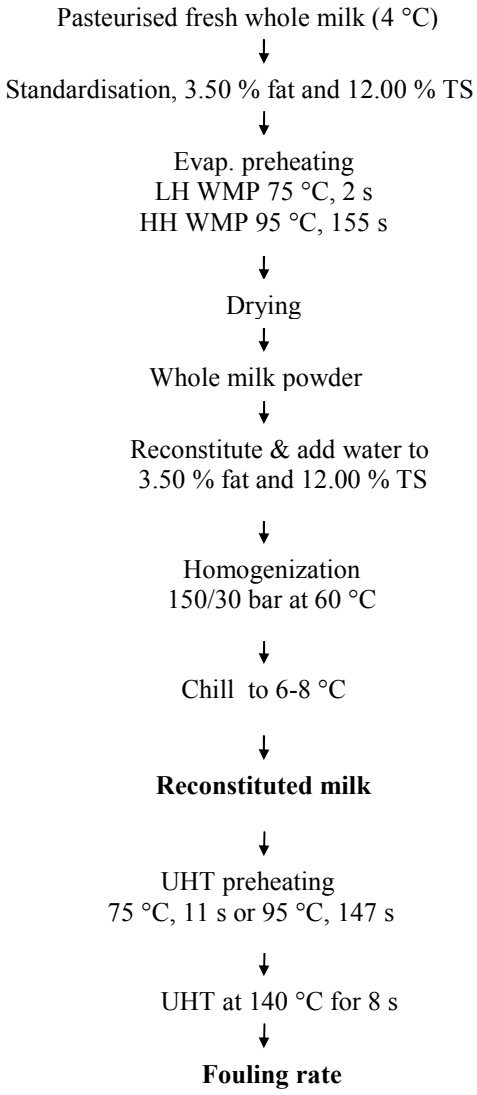


Figure 3.11 Experimental design for investigation of the effect of evaporator preheating on fouling by Recon (Milk Nos. 4 and 5).

3.9.3 Milk Nos. 6 and 7

Standardised, pasteurised fresh whole milk (Milk Nos. 6 and 7) was used to investigate the effect of the order of homogenization and UHT preheat treatment on fouling rate. The process was as shown in Figure 3.12.

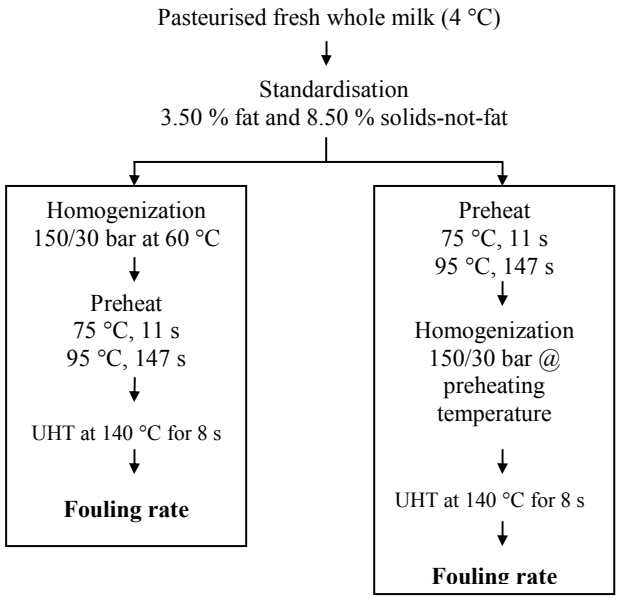


Figure 3.12 Experimental design for FWM with homogenization before or after preheat treatment (Milk Nos. 6 and 7).

Pasteurised fresh whole milk was subjected to preheat treatment before or after homogenization. Two UHT preheat treatments at 75 °C, 11 s and 95 °C, 147 s were used before or after homogenization prior to UHT processing. Fouling rate was measured for each combination of process order and UHT preheat treatment.

3.9.4 Milk No. 8

Milk No. 8 was used for trials similar to those shown in Figure 3.10, except that only two UHT preheat treatments were used, and RCB were made only from SMP1. The experimental design is shown in Figure 3.13.

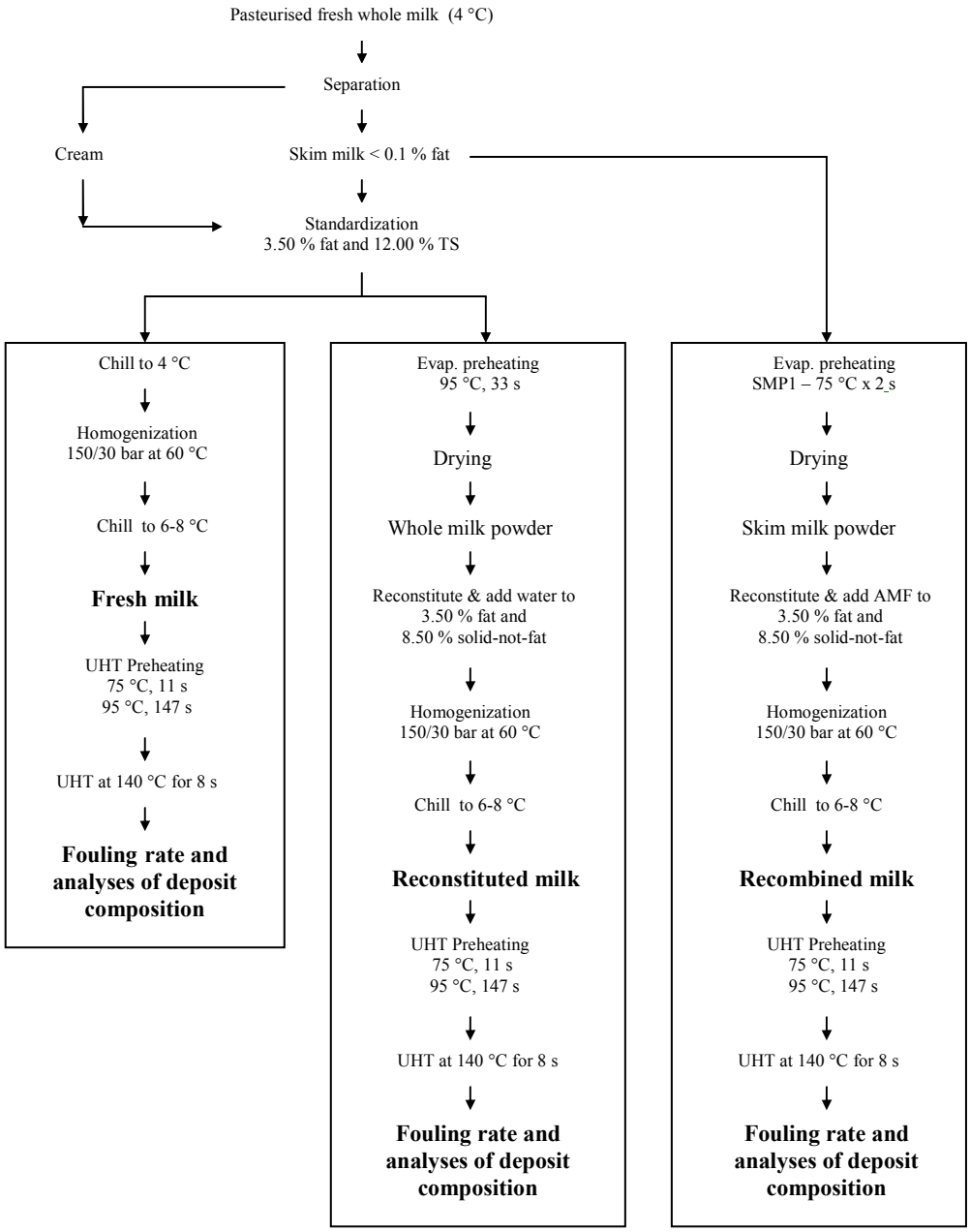


Figure 3.13 Experimental design for fresh whole milk (FWM), recombined whole milk (RCB) and reconstituted whole milk (Recon) (Milk No. 8)

In this experiment, skim milk was evaporator preheated at 75 °C, 2 s and whole milk was evaporator preheated at 95 °C, 33 s prior to drying. FWM, RCB and Recon were subjected to two UHT preheat treatments, 75 °C, 11 s and 95 °C, 147 s, prior to UHT processing. Then, the fouling rates were measured as described in section 3.8. The high temperature heater was dismantled for deposit collection after 2 h running time after the plant had been rinsed (see section 3.7).

3.9.5 Milk No. 9

Pasteurised and standardized fresh whole milk (Milk No. 9) was used to study the effect of order of homogenization and UHT preheat treatment on the amount and composition of the fouling deposit in the high-temperature heater. The process was as shown in Figure 3.14.

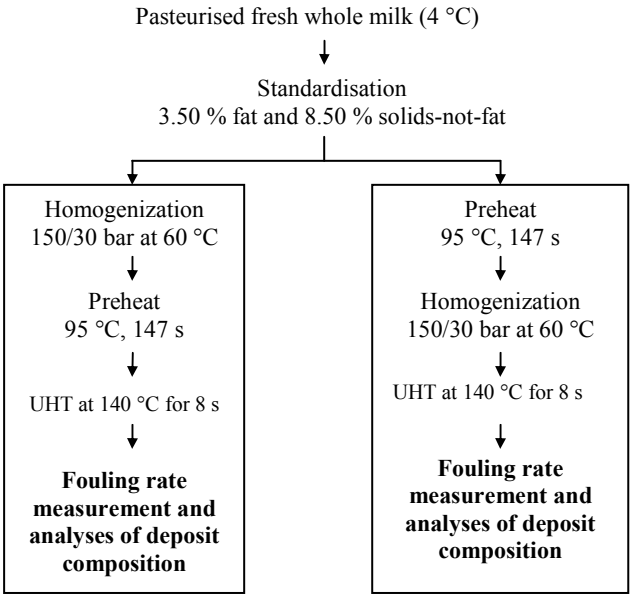


Figure 3.14 Experimental design for FWM with preheat treatment before and after homogenization (Milk No. 9).

Pasteurised fresh whole milk was standardised and then was preheated before or after homogenization prior to UHT processing.

3.9.6 Milk Nos. 10 and 11

Two batches (Milk Nos. 10 and 11) of pasteurised fresh whole milk were processed as shown in Figure 3.15 to investigate the effect of homogenization on fouling rate, and on the amount and composition of fouling deposit in the high-temperature heater.

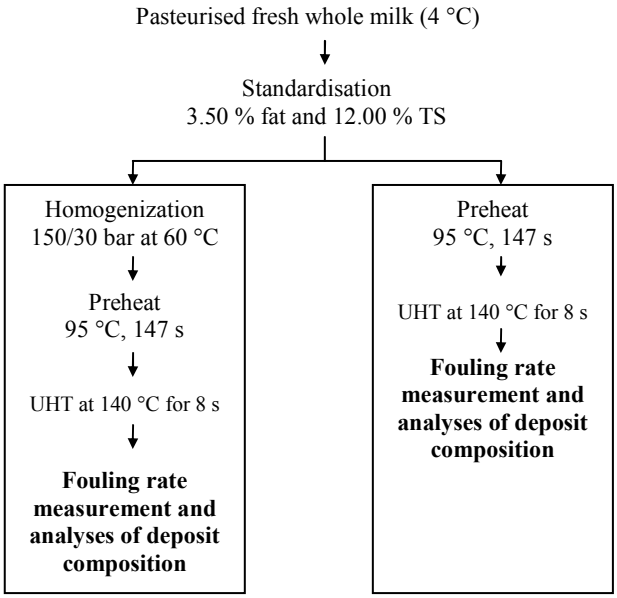


Figure 3.15 Experimental design for fresh whole milk with homogenization and no homogenization (Milk Nos. 10 and 11).

Pasteurised and standardised fresh whole milk was subjected to homogenization and no homogenization prior to UHT processing. The fouling rates were measured and the amount and composition of the deposit from the high-temperature heater measured.

3.9.7 Milk No. 2b

SMP1, 2 and 3 and WMP from Milk No. 2 were recombined to RCBs and Recon after they had been stored at 14-16 °C for 2 years in order to investigate the effect of aging on fouling rates of RCB and Recon. The experimental design is shown in Figures 3.16 and 3.17.

RCB and Recon were preheated at the different UHT preheat treatments as shown in Figures 3.16 and 3.17 prior to UHT processing. Fouling rates were measured and the amount and composition of deposits were measured.

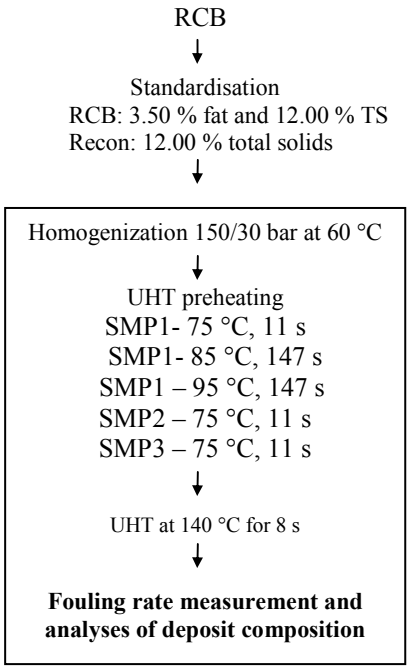


Figure 3.16 Experimental design for RCB (Milk No. 2b).

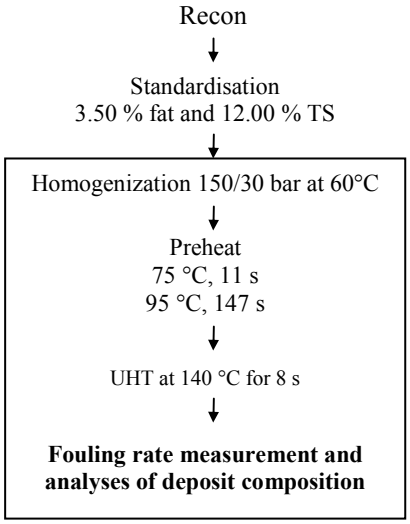


Figure 3.17 Experimental design for Recon (Milk No. 2b).

3.10 Summary of all experimental designs

Every batch of raw whole milk was pasteurised before entering the milk pilot plant.

Table 3.1 summarises all milk batches used in this work, the preheating conditions applied to them and the objectives of fouling rate measurement.

Table 3.1 Description of all experiments.

Milk no.	FWM		RCB			Recon			UHT plant	Purpose of study
	Milk month	UHT preheat treatment	Milk powder manufacture	Evap. preheat treatment	UHT preheat treatment	Milk powder manufacture	Evap. preheat treatment	UHT preheat treatment		
1	Dec'03	75 °C, 1 s 75 °C, 123 s 95 °C, 123 s	Dec'03	SMP1 -75 °C, 2 s SMP2 -75 °C, 155 s SMP3 -95 °C, 155 s	75 °C, 1 s 75 °C, 123 s 95 °C, 123 s	Dec'03	95 °C, 33 s	75 °C, 1 s 75 °C, 123 s 95 °C, 123 s	Plant 1	Effect of preheat treatments of FWM, RCB and Recon on UHT fouling rate
2	Oct'04	75 °C, 11 s 85 °C, 147 s 95 °C, 147 s	Oct'04	SMP1 -75 °C, 2 s SMP2 -85 °C, 155 s SMP3 -95 °C, 155 s	75 °C, 11 s 85 °C, 147 s 95 °C, 147 s	Oct'04	95 °C, 33 s	75 °C, 11 s 85 °C, 147 s 95 °C, 147 s	Plant 2	
3	Nov'04	75 °C, 11 s 85 °C, 147 s 95 °C, 147 s	Nov'04	SMP1 -75 °C, 2 s SMP2 -85 °C, 155 s SMP3 -95 °C, 155 s	75 °C, 11 s 85 °C, 147 s 95 °C, 147 s	Nov'04	95 °C, 33 s	75 °C, 11 s 85 °C, 147 s 95 °C, 147 s	Plant 2	
2a			Oct 04	SMP1 -75 °C, 2 s	75 °C, 11 s 95 °C, 147 s					Effect of homogenization on fouling rate of reconstituted skim milk
3a			Nov'04	SMP1 -75 °C, 2 s	75 °C, 11 s 95 °C, 147 s					
4						June'05	WMP1 - 75 °C, 2 s WMP2 - 95 °C, 155 s	75 °C, 11 s 95 °C, 147 s	Plant 2	Effect of low and high evaporator preheat treatment on fouling rate of Recon
5						July'05	WMP1 - 75 °C, 2 s WMP2 - 95 °C, 155 s	75 °C, 11 s 95 °C, 147 s	Plant 2	
6	Aug'05A	75 °C, 11 s 95 °C, 147 s							Plant 2	Effect of homogenization and UHT preheating order on fouling rate of FWM
7	Aug'05B	75 °C, 11 s 95 °C, 147 s							Plant 2	
8	Feb'06	75 °C, 11 s 95 °C, 147s	Feb'06	SMP1 -75 °C, 2 s	75 °C, 11 s 95 °C, 147 s	Feb'06	95 °C, 33 s	75 °C, 11 s 95 °C, 147 s	Plant 2	Effect of preheat treatments on the deposit composition of FWM, RCB and Recon in the high temperature heating section of the heat exchanger
9	June'06	95 °C, 147 s							Plant 2	Effect of homogenization and UHT preheating order of FWM on the deposit composition
10	Oct'06	75 °C, 11 s 95 °C, 147 s							Plant 2	Effect of homogenization on fouling rate of FWM
11	Apr'07	75 °C, 11 s 95 °C, 147 s							Plant 2	Effect of homogenization on fouling rate of FWM
2b			Oct'04	SMP1 -75 °C, 2 s SMP2 -85 °C, 155 s SMP3 -95 °C, 155 s	75 °C, 11 s 95 °C, 147 s	Oct'04	95 °C, 33 s	75 °C, 11 s 95°C, 147 s	Plant 2	Effect of ageing of skim milk powder and whole milk powder on UHT fouling and deposit composition

Note: -Evaporator preheat treatment is preheat treatment before evaporation in milk powder manufacture.

3.11 Chemical analyses of liquid milks and fouling deposits

Chemical analyses were carried out by the Analytical Services Group of Fonterra, Palmerston North. Similar analyses were applied to both deposits and liquid milks. Repeatability is the variation in measurements obtained when a person does multiple measurements using the same instruments and techniques on the samples from the same source. Reproducibility is the variation in measurements obtained when two people or more measure the same samples using the same measuring technique but perhaps different instruments. Both repeatability and reproducibility are measured and reported for milk liquid only. There are no repeatability and reproducibility figures for the analyses of the deposit because of inadequate amount of the deposit for duplicate measurements and because the recorded repeatabilities and reproducibilities for liquid milk do not necessarily apply to deposits.

Fat

Fat was analysed by the Roese-Gottlieb method (Fonterra, 2005). Milk fat is extracted from an ammoniacal ethanolic solution of the liquid, diluted or dissolved test portion with diethyl and petroleum ethers. The solvents are evaporated and the residue is dried. The weight of substances extracted is determined. This method is based on IDF Provisional Standard 1D:1996, Milk, Determination of Fat Content, Gravimetric Method (Reference Method), International Dairy Federation, Brussels. The accuracy of this test is shown in Table 3.2.

Table 3.2 Accuracy of fat determination (Fonterra, 2005).

Samples	Repeatability	Reproducibility
Raw and processed liquid milk	± 0.02 %	± 0.04 %
Cream	± 0.50 %	± 1.00 %

Ash

A weighed portion of the sample is dried and then combusted to constant weight (Fonterra, 2003). The residue is expressed as a mass percentage of the original sample. This method is based on BS 1741:1988, Chemical Analysis of Liquid Milk and Cream, Part 9,

Determination of ash from liquid milk. The accuracy of this determination is reported in Table 3.3.

Table 3.3 Accuracy of ash determination (Fonterra, 2003).

Samples	Repeatability	Reproducibility
Liquid milks	± 0.03 %	± 0.07 %

Moisture content

The sample is dried in an oven at 102 ± 2 °C to constant mass and weighed to determine the loss of moisture (Fonterra, 2004b). Moisture content is expressed as g/100g of product. The method is based on IDF Provisional Standard 26A;1993, Dried Milk and Dried Cream, Determination of Water Content, International Dairy Federation, Brussels. The accuracy of the moisture content test is shown in Table 3.4.

Table 3.4 Accuracy of moisture content determination (Fonterra, 2004b).

Samples	Repeatability	Reproducibility
Liquid milks	± 0.2 g of moisture per 100 g of product	± 0.4 g of moisture per 100 g of product

Total solids

Samples are evaporated to dryness on a boiling water bath prior to complete removal of free moisture in a drying oven at 102 °C. Total solids is the mass of residue remaining after the completion of the drying process as a percentage of the mass of the test portion. Solids-not-fat may be calculated from total solids by substracting the percentage of fat determined by the Roese-Gottlieb procedure (Fonterra, 2000a). The method is based on IDF Standard 21B:1987, Milk, Cream and Evaporated Milk, Determination of Total Solids Content (Reference Method), International Dairy Federation, Brussels. The accuracy of the total solids test is reported in Table 3.5.

Table 3.5 Accuracy of total solids determination (Fonterra, 2000a).

Samples	Repeatability	Reproducibility
Liquid milks	± 0.10 %	± 0.20 %
Cream	± 0.20 %	± 0.35 %

Total nitrogen

This method is used to determine the total nitrogen content by the Kjeldahl method and to calculate the crude protein content of milk samples (Fonterra, 2004a). After the sample is weighed, it is catalytically digested with sulphuric acid, converting the total organic nitrogen into ammoniacal nitrogen. The ammonia is released by the addition of sodium hydroxide, distilled and absorbed in boric acid, and then titrated. The percentage nitrogen content is multiplied by the factor 6.38 to convert to percentage protein. Because milk contains nitrogen-bearing compounds which are not protein the protein content thus calculated is referred to as the crude protein content. The accuracy of the total nitrogen test is reported in Table 3.6.

Table 3.6 Accuracy of total nitrogen determination (Fonterra, 2004a).

Samples	Repeatability	Reproducibility
Liquid milks	± 0.04 % protein	± 0.05 % protein

Non-protein-nitrogen

True proteins in the sample are precipitated with trichloroacetic acid and filtered off. Non-protein nitrogen (NPN) in the filtrate is determined by the Kjeldahl method (Fonterra, 2000c). This method is based on IDF Provisional Standard 20B:1993, Milk, Determination of Nitrogen Content. Part 4: Determination of Non-Protein Nitrogen Content. International Dairy Federation, Brussels. The accuracy of the non-protein-nitrogen test is reported in Table 3.7.

Table 3.7 Accuracy of non-protein-nitrogen determination (Fonterra, 2000c).

Samples	Repeatability	Reproducibility
Liquid milks	± 0.003 %	± 0.006 %

Casein nitrogen

The casein in the milk sample is then precipitated from a further test portion with acetic acid-acetate buffer and filtered off. The nitrogen content of the filtrate (non-casein nitrogen) is determined. Casein nitrogen is calculated by substracting non-casein nitrogen from total nitrogen (Fonterra, 2000b). The accuracy of this test is shown in Table 3.8.

Table 3.8 Accuracy of casein nitrogen determination (Fonterra, 2000b).

Samples	Repeatability	Reproducibility
Liquid milks	± 0.04 %	± 0.06 %

Phosphate and lactose

The Auto Analyser (AA) instrument is used to determine the level of phosphate, lactose calcium and phosphorus in the sample.

Phosphate

The milk sample is mixed with citrate buffer and air. The mixture is passed through a dialysis block. A consistent proportion of the phosphate ions get through to the other side of the membrane of the dialysis block. These phosphate ions are picked up by water and air stream and then, nitric acid is added to lower the pH of the stream. The nitric acid is necessary for the colour reaction to take place. The solution is mixed with a mixing coil. The phosphate colour reagent, containing ammonium molybdate and ammonium metavanadate, are added. The phosphate in the sample will react with these reagents and form a coloured complex on heating. The solution is passed through a mixing coil and heated to 35-40 °C. The absorption of the coloured complex is measured by the colorimeter at 405 nm. This method is based on Hoffman, W.S., 1937, Technicon AutoAnalyser II, Industrial Method No 120-71A. May 1973. J.Biol.Chem., 120, 51-55.

Lactose

Sample, citrate buffer and air mixture passes through a dialysis block, allowing a consistent proportion of lactose molecules to pass through to the other side of the membrane. These lactose molecules are picked up by a stream of water.

Lactose Colour Reagent which contains potassium ferricyanide and Sodium Carbonate is added. The stream is mixed via a mixing coil and passed through a heated oil bath at 65-70 °C, where the dialysed lactose reduces the alkaline ferricyanide (yellow) to ferricyanide (colourless).

The stream then passes through a condenser, which cools the liquid, prior to it entering the colorimeter. The absorption of the remaining ferricyanide (yellow colour) is measured at 420 nm by the colorimeter. The amount of lactose in the sample is inversely proportional to the absorbance.

The accuracy of the phosphate and lactose test is shown in Table 3.9.

Table 3.9 Accuracy of phosphate and lactose determination.

Determination	Repeatability	Reproducibility
Phosphate	± 1.30 %	n/a
Lactose	± 0.40 %	n/a

Phosphorus and calcium

The method of acid digestion and inductively coupled plasma optical emission spectrophotometry is used to determine the calcium and phosphorus content in milk samples and fouling deposit. Samples are digested with nitric and hydrochloric acids using a commercial microwave system or enclosed polycarbonate or polypropylene vials, then analysed by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-OES). High-pressure microwave digestion is essential for the digestion and breakdown of fat products prior to ICP analysis but the low-pressure digestion at elevated temperatures is

adequate or the digestion of all other product types. The accuracy of the phosphorus and calcium test is shown in Table 3.10.

Table 3.10 Accuracy of calcium and phosphorus determination (Fonterra, 2004).

Determination	Repeatability	Reproducibility
Calcium	± 2.70 %	± 12.00 %
Phosphorus	± 8.80 %	± 15.50 %

3.12 Analysis of fat-bound-protein in the cream layer

The fat (fat globules) to be analysed was prepared by centrifugation of whole milk (homogenized or not homogenized). The milk was warmed to 40 °C to ensure the fat was in the liquid state at the outset. 36 ml aliquots were added to each of 8 prewarmed 50 ml silicone screw capped centrifuge tubes. They were placed in a warmed centrifuge rotor head (Sorvall, F-28/36) at 40 °C in order to liquidify the fat in the samples. Then, the milk samples were centrifuged in a precooled refrigerated high speed centrifuge so that the head and samples were cooled to 5 °C during the run at 15,000 rpm for 30 min (g-force = 20,800 g). The centrifuged samples were taken to a controlled temperature room (4 °C) to recover the cream layer, the "top-serum", the underlying serum and sediment fractions. The "top-serum" fraction, the 2.5 ml immediately underlying the cream plug, was used as a "control" for the estimation of the amount of non-fat-bound protein in the trapped serum portion of the cream plug. This top-serum was withdrawn first from under the cream plug, using a 2.5 ml syringe, and weighed. The cream layer was then removed, placed in a petri dish and weighed. The serum was drained off all the centrifuge tubes and weighed. The sediment was scraped out from each of the tubes and weighed. All weights of fat layer, top-serum, serum and sediment were recorded. The cream layer, top-serum, serum, and sediment fractions after centrifugation are illustrated in Figure 3.18.

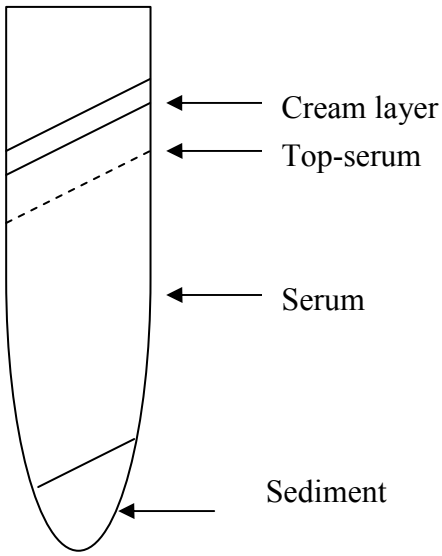


Figure 3.18 Cream layer, top-serum, serum and sediment after centrifugation.

In this study, the large fat globules present in the cream layer was used to represent most fat globules in the whole milk system. Each fat globule in every fraction was covered with milk protein. Differences in the sizes of these fat globule caused differences in the ratio of surface area : volume in each fraction. The estimation of the proportion of fat recovered in the cream layer is reported in Chapter 6.

To estimate total fat-bound protein in the cream layer in this present study, it was necessary to subtract the non-fat bound protein in the top-serum trapped in the cream layer from the total protein (in the unwashed cream layer). Because of the high "g" force of centrifugation, the protein content in the bulk serum, particularly the micellar casein settles to a significant extent forming a concentration and composition gradient in the serum layer. It was assumed that the topmost portion of bulk serum immediately under the cream layer (the "top-serum") gave a reasonable representation of the composition of the trapped serum (representing the non-fat-bound portion of the protein in the cream layer). Thus, the best approximation for the total fat-bound protein in the cream layer is obtained by substracting the protein in the serum trapped in the cream layer, which is derived from the moisture content (of the cream layer). Total nitrogen, moisture content, total solids and fat content analyses were carried out for the cream layer and top-serum. Homogenized then preheated

FWM for Milk No.8 at preheating 75 °C, 11 s was used to demonstrate the calculation of fat-bound protein with and without top-serum substraction (section 3.12.1).

3.12.1 Calculation of fat-bound protein with top-serum substraction

1. Fat-bound TN = Total fat-bound TN – non-fat-bound TN

2. Trapped serum % TN = % TN in top-serum

3. Assuming that trapped serum in the cream layer is equal to top-serum, which is underneath the cream plug after centrifugation, the percentage of trapped top-serum TN in the cream layer is.

$$= \left(\frac{\%\ moisture\ in\ the\ cream\ layer}{\%\ moisture\ in\ top\ serum} \times \%\ TN\ in\ top\ serum \right)$$

$$= \frac{46.93}{90.53} \times 0.41$$

$$= 0.213$$

4. Percentage of fat-bound TN in the cream layer

$$= \%\ TN\ in\ the\ cream\ layer - \%\ top\ serum\ TN\ in\ the\ cream\ layer$$

$$= 0.91 - 0.213$$

$$= 0.695$$

5. Total fat-bound protein in the cream layer expressed as g protein / g fat

$$= \frac{\%\ fat\text{-}bound\ TN\ in\ the\ cream\ layer \times 6.38}{\%\ fat\ in\ the\ cream\ layer}$$

$$= \frac{0.695}{45.50} \times 6.38$$

$$= 0.097$$

3.12.2 Calculation of fat-bound protein without top-serum substraction

Total fat-bound protein in the cream layer without top-serum substracted (g) / fat (g) is:

$$= \frac{\left(\frac{\%TN\ in\ the\ creamlayer}{100} \times 6.38 \right)}{\left(\frac{\%\ fat\ in\ the\ cream\ layer}{100} \right)} \qquad = \frac{(0.91/100)}{(45.50/100)}$$

$$= 0.127$$

The levels of total fat-bound protein in the cream layer without top-serum substracted were used in Chapter 6. The justification for doing this is explained in Chapter 6.

3.12.3 Measurement of milk fat globule particle size distribution

The size distribution of particles in whole milks (assumed to all be fat globules) was measured using a Mastersizer 2000 (Malvern Instruments, Malvern, Worcestershire, England).

3.13 Polyacrylamide gel electrophoresis (PAGE)

Sodium dodecyl sulphate (SDS) PAGE was used to quantify whey proteins and casein proteins. SDS is a negatively charged surface-active substance. It is used to disrupt non-covalent bonds, through its ability to adsorb to hydrophobic and positively charged sites on proteins. Different proteins bond with SDS to almost the same extent on a mass basis. Thus, once coated with SDS, the proteins can be separated by electrophoresis on the basis of the molecular size of protein-SDS complexes.

Standard methods for the preparation of resolving gels and stacking gels followed the procedures described in the Food Science Gel Electrophoresis Manual of Fonterra, Palmerston North, New Zealand, version 3, 1998. Reduced-SDS PAGE analysis was used in this study in order to break down all disulphide bonds in the milk samples.

Preparation of the cream layer for PAGE analysis

Weigh 1 g of cream layer and then mix with 1 g of SDS sample buffer. After the solution was well-mixed, 25 μL of the mixture was transferred by a pipette to an Eppendorf tube, which contain 960 μL of SDS. Flush the pipette tip with the solution in the tube a few times. Vortex mix the samples for 5 s and then place 20 μL of 2-mercaptoethanol in the tube and flush the pipette tip a few times with the solution in the tube. Close the lid of the tube securely before transferring it to a boiling rack. Boil the sample for 4 minutes and cool down with cold water. Centrifuge the tubes at 5000 rpm for 3 minutes to separate the fat. Then, the solution underneath the cream layer is ready to use in the gel electrophoresis.

PAGE analysis of each sample of cream layer was replicated six times using different gels. The average of six densitometer readings was used in the data presented in Chapter 6.

Loading the sample on to the gel

A Hamilton syringe (Hamilton company, Reno, Nevada, USA) was used to transfer 10 μL of the sample to the gel sandwich assembly (Mini-Protein II Alignment card, catalog number 165-2957). The Hamilton syringe was first flushed 3 times with the sample. The sample was transferred to the slot in the gel without air bubbles trapped in the syringe. Rinse the syringe with the Milli Q water onto a tissue. Repeat the loading step from the beginning for the next sample.

Running the gel

The gel sandwich was connected to the electrophoresis unit (Biorad model 1000/500 power pack). Run the gel until the dye band just begins to seep through the bottom of the glass plates. Turn off the power pack and remove the gel from the gel sandwich.

Staining and de-staining

Stain the gel in Amido black for 24 hours. De-stain the gel with 10 % acetic acid until the background is clear.

Quantitative determination of individual protein by densitometer

Scan the gel by a densitometer (Computing Densitometer, Molecular Dynamics, Sunnyvale, California).

Normalization of densitometer reading

Each densitometer reading (α_s-casein, β-casein, κ-casein, γ-casein, β-lg and α-la) was divided by the fraction of fat in each sample of cream player in order to normalized the data. The average reading from six densitometer readings was used in Chapter 6.

Uncertainty of densitometer reading

Densitometer reading of FWM at the 75°C, 11 s preheat treatment (Milk No. 8) was used to demonstrate the calculation of the uncertainty of densitometer reading and the results of the uncertainty for FWM at every heating process stage is reported in Table 3.11.

For example, the uncertainty of densitometer reading for 4 readings of α_s-casein can be calculated as follow:

$$Uncertainty = Arithmetic\ mean \pm (2 * standard\ deviation)$$

$$= \left(\frac{199 + 226 + 250 + 229}{4}\right) \pm (2 * 21)$$

$$= 226 \pm 42$$

Table 3.11 Uncertainty of densitometer reading for FWM at the 75°C, 11 s preheat treatment (Milk No.8).

Protein	Uncertainty of densitometer reading			
	Homog. FWM	Preheated FWM	Inter. heated FWM	UHT FWM
α_s-casein	226±42	223±44	244±46	247±48
β-casein	218±62	207±48	250±66	245±46
κ-casein	72±38	70±24	63±14	60±22
γ-casein	8±6	6±2	3±2	3±2
β-lg	23±8	26±8	78±22	81±28
α-la	4±4	4±4	12±6	19±6

3.14 Statistical analysis

Results were analysed statistically using Minitab 14 for Windows. The split-plot analysis with replicates was applied to some experimental designs in Chapters 5, 6 and 7 because "replicates" were obtained by doing trials on more than one milk number. The distribution of data in this study was assumed to be normal.

The split-plot analysis with replication from Potcner & Kowalski (2004) was applied to analyse the data when identical sets of experimental were done with two or more milk numbers; all the milks used within a set originating from the same batch (number) of supplied raw fresh milk. An experimental design for split-plot analysis is shown in

Figure 3.19 (similar to Figure 5.7 in Chapter 5) and the result is reported in Table 3.9. In this analysis the main plot is evaporator preheat treatment, and the sub-plot is UHT preheat treatment. The response is fouling rate.

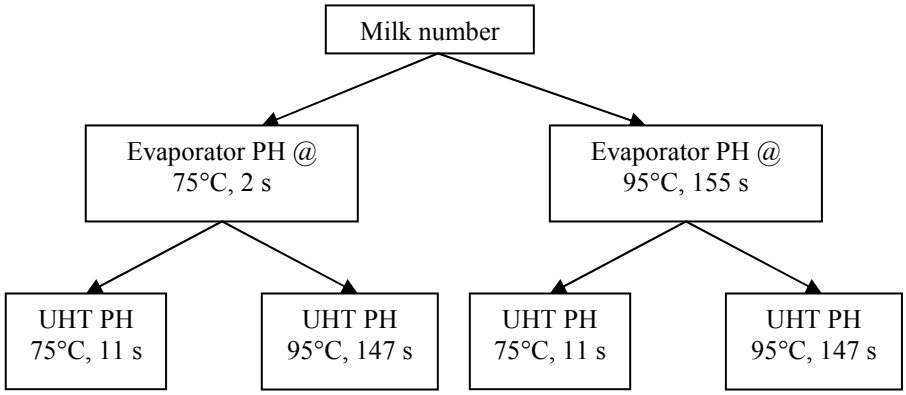


Figure 3.19 Experimental design for Recon.

The example in Table 3.12, which is similar to the analysis for Table 5.11 in Chapter 5, shows how to analyse the data by using the principle of the split-plot analysis. Fouling rates (FR) were transformed to log (FR+1) because some fouling rates were zero and the residual plots of log (FR+1) gave a better balance than the residual plots of log (FR) or of FR in most cases. Therefore, log (FR+1) was analysed using the General Linear Model for analysis of variance, the results of which were then further processed using the split-plot approach.

Table 3.12 Results for FR: LH and HH WMP
General Linear Model: log (FR+1) versus Milk No., Evaporator PH and UHT PH

```
Factor          Type   Levels  Values
Milk No.        fixed       2  4, 5
Evaporator PH   fixed       2  95 155, 75 2
UHT PH          fixed       2  75 11, 95147

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source                   DF    Seq SS    Adj SS    Adj MS        F      P
Milk No.                  1  0.030805  0.030805  0.030805   392.20  0.032
Evaporator PH             1  0.019235  0.019235  0.019235   244.90  0.041
Milk No.*Evaporator PH    1  0.000432  0.000432  0.000432     5.50  0.257
UHT PH                    1  0.081051  0.081051  0.081051  1031.92  0.020
Evaporator PH*UHT PH      1  0.002673  0.002673  0.002673    34.03  0.108
Milk No.*UHT PH           1  0.021150  0.021150  0.021150   269.27  0.039
Error                     1  0.000079  0.000079  0.000079
Total                     7  0.155424

S = 0.00886252   R-Sq = 99.95%   R-Sq(adj) = 99.65%
```

In the split-plot analysis, the F statistic for the effects of Milk No. (replicate / "block"), evaporator preheat treatment, and UHT preheat treatment within evaporator preheat treatment were adjusted using the output of the Minitab factorial AoV with replication. (For this it was necessary to combine the notional "interaction" parameter involving "block" (fixed effects) with the notional full analysis of variance (AoV) "error" parameters).

a) Effect of Milk No.

$$F = \frac{Adj\ mean\ square\ (MS)\ of\ the\ effect\ of\ Milk\ No.}{Adj\ MS\ of\ the\ "interaction"\ of\ Milk\ No. \times evaporator\ PH}$$

(Note: the denominator is used as the main plot error)

$$F = \frac{0.030805}{0.000432}$$

$$F_{calculated} = 71.31$$

The degree of freedom for the effect of Milk No. is one, and the degree of freedom for the "interaction" of Milk No. and milk preparation is also one. Thus, the p-value for the effect of Milk No. is 0.075. An F value is easily converted to a p-value using the appropriate function in a software package such as Minitab.

When there was no effect of the "interaction" between Milk No. and evaporator preheat treatment because the number of replicates was not enough to give an interaction, the p-value of the effect of Milk No. is reported as such.

b) Effect of evaporator preheat treatment

$$F = \frac{Adj\ MS\ of\ the\ effect\ of\ evaporator\ preheat\ treatment}{Adj\ MS\ of\ the\ "interaction"\ of\ Milk\ No. * evaporator\ PH}$$

(Note: the denominator is used as the main plot error)

$$F = \frac{0.019235}{0.000432}$$

$$F = 44.53$$

The degree of freedom for the effect of milk preparation is one and the degree of freedom for the "interaction" between Milk No. and milk preparation is also one. Thus, the p-value for the effect of evaporator preheat treatment is 0.095.

When there was no the effect of the "interaction" between Milk No. and evaporator preheat treatment because the number of replicate was not enough to give an interaction, the p-value of the effect of milk preparation is reported as such.

c) Effect of UHT preheat treatment within evaporator preheat treatment

$$F = \frac{Adj\ MS\ of\ the\ effect\ of\ UHT\ PH + Adj\ MS\ of\ the\ "interaction"\ of\ UHT\ PH * evaporator\ PH}{Adj\ MS\ of\ the\ "interaction"\ of\ Milk\ No. * UHT\ PH + Adj\ MS\ of\ "error"}$$

$$F = \frac{0.081051 + 0.002673}{0.021150 + 0.000079}$$

$$F_{calaulated} = 3.96$$

The overall degrees of freedom for the numerator is two, which is from the addition of one degree of freedom for the effect of UHT PH and another one degree of freedom for the "interaction" between UHT PH and evaporator preheat treatment. The degrees of freedom of the denominator is two, which is from the addition of one degree of freedom of the "interaction" between Milk No. and UHT PH and another one degree of freedom of the error. Thus, the p-value for the effect of UHT preheat treatment within evaporator preheat treatment is 0.202.

When there was no "interaction" effect between UHT PH and evaporator PH owing to insufficient replication, only the Adj MS of the effect of UHT PH is the numerator. If there is no "interaction" between Milk No. and UHT PH, only the Adj. MS of the error is used as the denominator. This general approach was used for all split-plot analyses.

In some cases, one-way analysis of variance was used.

All factors and all possible "interactions" were included in the initial analysis of variance. As all experiments were done on the pilot plant scale, using equipment in heavy demand, the number of runs was limited. Data from fouling experiments often exhibit considerable

scatter, and the aim was to identify trends. The conventional rule for interpreting the significance of results ($p < 0.05$) was relaxed as defined in Table 3.13.

Table 3.13 Interpretation of p-values used in the present study. The wording is taken from www.stat.ualberta.ca/~hooper/teaching/misc/.

P-value	Description
≥ 0.15	No evidence against the null hypothesis. The data appear to be consistent with the null hypothesis.
0.10-0.15	Weak evidence against the null hypothesis in favour of the alternative.
0.05-0.10	Moderate evidence against the null hypothesis in favour of the alternative.
0.01-0.05	Strong evidence against the null hypothesis in favour of the alternative.
≤ 0.01	Very strong evidence against the null hypothesis in favour of the alternative.

3.15 Determination of the change in the composition of liquid milk during UHT processing

To estimate depletion in milk solids (due to deposition in the UHT plant), the initial feed milk (FWM, RCB and Recon) (sampled before UHT processing) and UHT FWM (sampled after two hours of processing) were analysed for fat, protein, lactose, ash and ash components (calcium, phosphate and phosphorus). Depletions were compared with the weight of corresponding deposit components in the high-temperature heater. Calculations were made as in the following example :

The amount of fat in feed FWM for Milk No. 8, UHT preheated at 75 °C, 11 s.

$$= \left(\frac{\%\ w/w\ fat\ in\ liquid\ milk}{100} \right) \times volume\ of\ milk\ in\ 2\ hours\ (L) \times \frac{actual\ processing\ time\ (s)}{7200\ s} 1000$$

$$= \left(\frac{3.59}{100}\right) \times \left(231 \times \frac{6930}{7200}\right) \times (1000)$$

$$= 8{,}293 \text{ g}$$

The amount of fat in UHT FWM after 2 hour processing for Milk No. 8, preheating at 75 °C, 11 s was subjected to the same process of calculation. The difference between the amount of fat in the feed and the amount in the UHT milk is reported as the depletion in fat for FWM during UHT processing. Full results are given in Chapter 7. (In the above calculation, the density of milk was, for convenience, taken as 1,000 kg/m^3. It was not deemed necessary to calculate a very accurate value).

Chapter 4

Determination of fouling rate

4.1 Introduction

Fouling of heat exchangers is most readily indicated by a drop in heat transfer rate. Heat transfer rate changes could be indirectly measured by the change with time in the log mean temperature difference or in the temperature difference (ΔT, hot water inlet temperature – milk outlet temperature) across the heating surface provided the flow rates of product and heating medium, and the product inlet and outlet temperatures, were all controlled to constant values. Due to the slow response to fouling of the log mean temperature difference across the whole heating surface of the high-temperature heater, the temperature difference at the hottest end of the high-temperature heater was used in this study because it gave a sharper and quicker response and provided the controlled milk outlet temperature (140 °C) and the controlled flow rates of milk and hot water. Thus, ΔT gives the most adequate indication of the heat transfer rate as affected by fouling in the high-temperature heater. ΔT is the also main indicator used in industry because it requires minimal instrumentation to measure fouling in commercial milk plants.

There are reports in the literature that there is an induction period before measurable fouling begins (Burton, 1968, 1988; Paterson & Fryer, 1988; Grandison, 1988b; Changani *et al.*, 1997; Visser & Jeurnink, 1997). Furthermore, Visser & Jeurnink (1997), and that this may be the consequence of certain necessary reactions having to occur at the fouling surface or in the bulk liquid.

At the outset of the current work, it was unknown whether a significant induction period would be encountered in fouling trials with the milk preparations under study. Thus, the objectives of the work reported in this chapter were: to measure the evolution of temperature difference with time, to see whether an induction period was indicated in the trials, and if so whether the intensity of UHT and evaporator preheat treatments affected it or not.

4.2 Estimation of induction period

The least squares approach to fitting the temperature difference versus time data was used to investigate whether an induction period existed in a fouling trial. Two methods of calculating the fouling rate from experimental data were compared. Both were linear regression procedures. In the first mathematical method, an induction period was assumed to exist and the data was fitted accordingly. A least squares approach was used to fit two sequential lines and the break-point between them: the first a horizontal line (zero change in ΔT), the second a line of constant positive slope (indicating the fouling rate). In the second method, the temperature difference-time data for the whole of a fouling run was fitted by linear regression using least squares with no allowance made for an induction period. These approaches are shown diagrammatically in Figure 4.1.

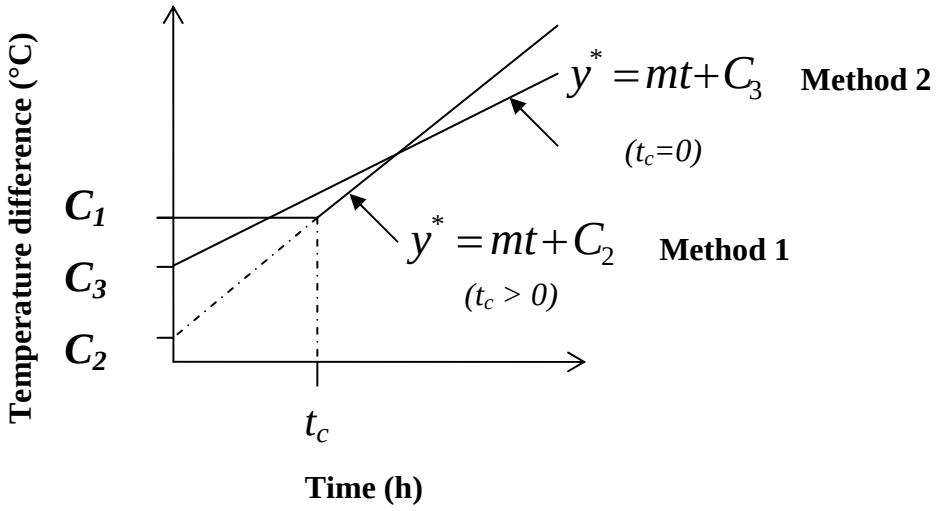


Figure 4.1 Diagrammatic plots of temperature difference in the high-temperature heater (water inlet minus milk outlet) versus time showing the approaches to determining the fouling rate, m, when an induction period, t_c, was present or was not present (t_c = 0). y^* = predicted temperature difference; C_1, C_2 and C_3 are constants.

Method 1

The solid lines in Figure 4.1 show the method 1 curves as fitted by least squares analysis. C_1, C_2 and m were the parameters adjusted to minimise the residual sum of squares.

$$y^* = mt + C_2 \quad \text{for} \quad t > t_c \qquad \text{Eq. 4.1}$$

and $$y^* = C_1 \quad \text{for} \quad t < t_c \qquad \text{Eq. 4.2}$$

where $y^* = predicted\ \ temperature\ difference\left(°C\right)$

$$t_c = \frac{\left(C_1 - C_2\right)}{m} \qquad \text{Eq. 4.3}$$

$$m = \ fouling\ \ rate \quad (°C/h) \qquad \text{Eq. 4.4}$$

The Solver function in Microsoft Excel was used to find the values of C_1, C_2, and m that minimised $\sum\left(y_{\exp} - y^*\right)^2$, where $y_{\exp} = measured\ \ temperature\ difference$

To avoid finding a false minimum of the residual sum of squares (RSS) and to achieve the lowest RSS, the RSS was calculated in three ways by fitting three forms of Eq. 4.3 (forms in which m, C_1 and then C_2 were the subject of the equation) in turn to the experimental data. These all gave the same minimum value, confirming that a true minimum RSS had been found.

Method 2

This method assumed that an induction period was absent, i.e. that $t_c = 0$ (Figure 4.1):

$$y^* = mt + C_3 \qquad \text{Eq. 4.5}$$

Equation 4.5 was fitted to the data using the Solver function in Microsoft Excel to search for the minimum residual sum of squares.

Examples of the application of methods 1 and 2 are shown in section 4.3.

4.3 Results

The experimental data shown in Figure 4.2 displayed what appeared to be a clear period of induction. Method 1 (section 4.2) was applied to look for an induction time.

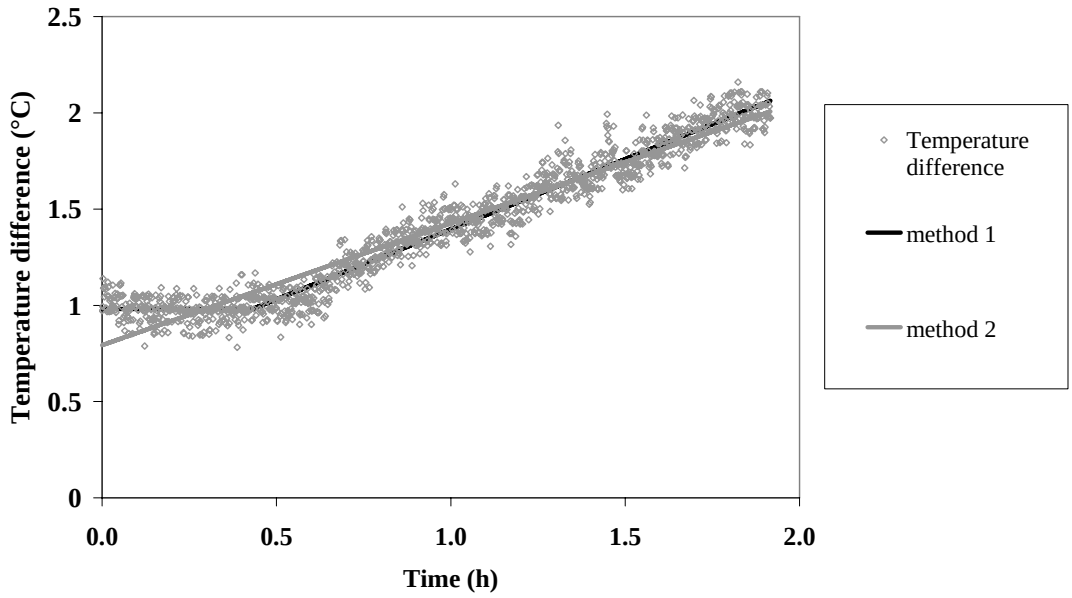


Figure 4.2 Temperature difference versus time data fitting for a fouling trial with Recon subjected to the 95 °C, 147 s UHT preheat treatment (Milk No. 8).

Method 1 gave a best fit with an induction period of 0.43 h. This was compared with a straight-line fit, Method 2. Method 1, with the induction time, lowered the minimum RSS from 12.567 to 7.356 (Table 4.1). Fitting the induction period results in the fouling rate from Method 1 being higher than that from Method 2 in which the induction time is ignored, as shown in Table 4.1.

Table 4.1 Parameters and minimum RSS for Methods 1 and 2 applied to the data shown in Figure 4.2 (Recon subjected to the 95 °C, 147 s UHT preheat treatment; Milk No. 8).

Parameters	Method 1	Method 2
t_c, induction time (h)	0.430	-
C_1 (°C)	0.981	-
C_2 (°C)	0.667	0.793
m, fouling rate (°C/h)	0.728	0.633
Minimum residual sum of squares (RSS)	7.356	12.567

In most fouling trials, an induction period was not obvious by inspection of the data, e.g. Figure 4.3, even when the RSS was lowered by the inclusion of an induction time, as shown in Table 4.2.

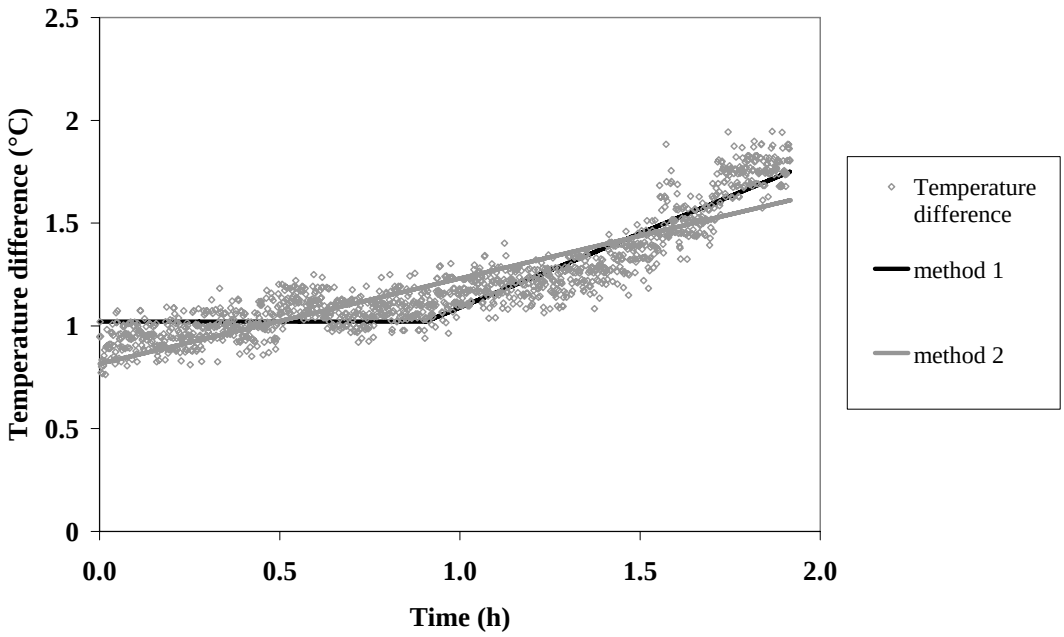


Figure 4.3 Fouling rate fitting for a fouling trial with homogenized then preheated FWM subjected to the 95 °C, 147 s UHT preheat treatment (Milk No. 9).

The fitted parameters are reported in Table 4.2.

Table 4.2 Parameters and minimum RSS for Methods 1 and 2 applied to the data shown in Figure 4.3 (homogenized then preheated FWM subjected to the 95 °C, 147 s UHT preheat treatment; Milk No. 9).

Parameters	Method 1	Method 2
t_c, induction time (h)	0.905	-
C_1 (°C)	1.021	-
C_2 (°C)	0.368	0.816
m, fouling rate (°C/h)	0.721	0.415
Minimum residual sum of squares (RSS)	12.987	18.309

Table 4.2 showed that Method 1 gave a lower RSS than did Method 2 with an induction period of 0.905 h. Method 2 gave a fouling rate that is much lower than the fouling rate of Method 1. Although Method 1 was considered a better method than Method 2 because it gave a lower RSS, but neither method is particularly good at fitting these data.

When this analysis was applied to all the available data (Appendix 1, Table A1.1), only four of runs showed a considerable reduction in RSS when Method 1 was fitted, compared with the linear regression method (Method 2); many of the trials showed very little reduction in RSS. For eleven trials, RSS was slightly higher when an induction period was modelled (Method 1).

The effects of UHT preheat treatment and evaporator preheat treatment on the induction period obtained using Method 1 were statistically analysed as shown in Table A1.2-A1.5. The results are summarised in Table 4.3.

These results showed only weak evidence for an induction period for RCB varying with the intensity of UHT preheat treatment ($p = 0.147$). No evidence was found for an induction period varying with the intensity of UHT preheat treatment for FWM or for Recon ($p = 0.418$ and 0.350, respectively) nor for an induction period for RCB varying with the intensity of evaporator preheat treatment ($p = 0.254$).

Table 4.3 Results of analysis of variance for the effects of UHT and evaporator preheat treatments on the induction period of FWM, RCB and Recon determined by applying Method 1. The original data were transformed to log (induction period + 1).

Preheat treatment	p-value		
	Homogenized then preheated FWM (Milk Nos. 2, 3, 6-11 and 2b)	**RCB** (Milk Nos. 2, 3, 8 and 2b)	**Recon** (Milk Nos. 2, 3, 8 and 2b)
UHT preheat treatment (75 °C, 11 s, 85 °C, 147 s and 95 °C, 147 s)	0.418	0.147 (prepared from SMP1 only)	0.350
Evaporator preheat treatment (75 °C, 2 s, 85 °C, 155 s and 95 °C, 155 s)	-	0.254	-

4.4 Discussion

The results in Appendix 1 (Table A1.1) show that a significant induction period was found in some fouling trials which varied randomly with the intensity of UHT and evaporator preheat treatments and with milk preparation; there were no trends.

An induction period was found for most fouling trials using Method 1 which usually resulted in a slightly reduced minimum RSS, but not for all trials. In a few cases, the minimum RSS increased. Burton (1968, 1988) and Grandison (1988b) reported “induction periods” in the range 0 h to 1.49 h. Although induction periods could be identified for a number of runs in the present study, induction period duration varied with unknown factors.

Fouling rate was calculated for the purposes of this study as the slope of the plot of temperature difference versus time from 0 h to 2 processing h using linear regression (Method 2), using the linear regression routine in Microsoft Excel. This gave slightly different estimates of fouling rate when compared with those given by Solver in Excel (Appendix1, Table A1.1). Therefore, the fouling rates shown in Chapter 5 are slightly different from the fouling rates obtained by fitting Method 2 and shown in Appendix 1 (Table A1.1).

4.5 Conclusion

There was no evidence for an induction period that varied consistently with the intensity of UHT and evaporator preheat treatments. Thus, Method 2, the linear regression method with no induction time allowed for, was chosen for use in determining the fouling rate in all trials to avoid biasing the overall fouling trials in this study.

[1]Chapter 5

Effect of preheat treatments on UHT plant fouling by whole milks

5.1 Introduction

Fouling of heat transfer surfaces by milk, especially under the high temperature conditions existing in the final heating sections of indirectly heated ultra-high temperature (UHT) sterilizing plants, is an ongoing problem in the dairy industry. A number of researchers have reported that preheating fresh whole milk prior to UHT processing results in lower extents of fouling (Bell & Sanders, 1944; Burton, 1968; Lalande *et al.*, 1984; Patil & Reuter, 1986a; Mottar & Moermans, 1988). In contrast, Newstead (2003, personal communication) found that preheating, both during powder manufacture (evaporator preheating) and immediately prior to UHT processing (UHT preheating), resulted in increased fouling rates for RCB (made from SMP and milk fat) and Recon (made from WMP). Further, Newstead *et al.* (1999) found that the effects of UHT preheating on the rate of fouling by FWM were variable, and there was evidence of variation in behaviour between batches of milk.

Changes in proteins in each milk preparation are siginificant when these milks are heated to greater than 60 °C. Such changes involve changes in proteins in the MFGM and changes of proteins in the milk plasma[2].

---

[1]The early part of the work reported in this chapter was presented at an international conference in 2006 (Srichantra, A, Newstead, D, McCarthy, O J, Paterson, A H J (2006). Effect of preheating on fouling of a pilot scale UHT sterilizing plant by fresh, recombined and reconstituted whole milks. In: D.I. Wilson, J.Y.M. Chew, P.J. Fryer & A.P.M. Hasting (eds.) *Fouling, Cleaning and Disinfection in Food Processing* Conference: Proceedings, University of Cambridge, Department of Chemical Engineering, pp. 228-237. Jesus College, Cambridge, UK, March 20-22. This presentation was subsequently published in the journal *Food and Bioproducts Processing* (Srichantra, A, Newstead, D F, McCarthy O J and Paterson A H J (2006) Effect of preheating on fouling of a pilot scale UHT sterilizing plant by recombined, reconstituted and fresh whole milks. *Food and Bioproducts Processsing* **84(C4)** 279-285).

[2]Milk plasma consists of casein micelles and milk serum.

Changes in the MFGM and milk plasma of fresh whole milk involve changes in casein, whey proteins, natural MFGM and probably salts during pasteurisation, homogenization and preheating (Walstra & Jenness, 1984; Houlihan *et al.*, 1992a; Dalgleish & Sharma, 1993; Sharma & Dalgleish, 1993; Sharma & Dalgleish, 1994; Van Boekel & Walstra, 1995; Lee & Sherbon, 2002). Heat-induced changes in the MFGMs of RCB and Recon are different because of the effects of drying during milk powder manufacture.

These same processing steps result in changes in the MFGM and milk plasma of RCB involving the association of caseins and whey proteins (Oortwijn & Walstra, 1979; Dalgleish, 1990; Dalgleish & Banks, 1991; McCrae & Muir, 1991; Singh & Creamer, 1991a; Corredig & Dalgleish, 1996a; Sharma *et al.*, 1996a, 1996b; Oldfield *et al.*, 2005a).

Heating of Recon also results in changes in the MFGM involving caseins and whey proteins (Anema & McKenna, 1996; McKenna, 2000; Ye *et al.*, 2005; Ye *et al.*, 2007).

Thus, the objective of this work was to investigate the effect of UHT preheat treatment and evaporator preheat treatment on fouling rates of FWM, RCB and Recon. Fouling by FWM was further studied with regard to the effect of homogenization itself, and the relative effects of homogenization before and after UHT preheat treatment. The effect of the presence of fat was investigated by a comparison of fouling rates between reconstituted skim milk and RCB, made from the same skim milk powder.

5.2 Effect of UHT preheating on fouling rate

Two UHT plants were used to measure the fouling rates of FWM, RCB and Recon as detailed in the description of process design and milk preparation (Chapter 3). In the study of the effects of preheat treatment on fouling by FWM, the milk was always homogenized before UHT preheat treatment. The RCB and Recon were, of course, homogenized as part of recombining and reconstitution processes, before UHT preheat treatment.

For all statistical analyses in this chapter, fouling rates (FR) were transformed to $Log_{10}(FR+1)$ because some fouling rates were zero and the residual plots of $\log_{10}(FR+1)$ gave a better balance than the residual plots of $\log_{10}(FR)$ or of FR in most cases. Thus, in the following sections the mean fouling rates listed (sections 5.2.2 and 5.2.3) are geometric means.

5.2.1 UHT plant 1, Milk No. 1

RCB was prepared from SMP1 (evaporator preheat treatment 75 °C, 2 s) and Recon was prepared from WMP (evaporator preheat treatment 95 °C, 33 s). FWM, SMP and WMP were all derived from the same batch of raw fresh whole milk. Three UHT preheat treatments (75 °C, 1 s; 75 °C, 123 s; and 95 °C, 123 s) were applied to each milk preparation prior to sterilization. The results are shown in Table 5.1.

Table 5.1 shows that there was a general, if not wholly consistent trend of fouling rates increasing with the intensity (temperature × time) of UHT preheat treatment.

Table 5.1 Fouling rates of FWM, RCB and Recon for UHT preheat treatments 75 °C, 1 s; 75 °C, 123 s and 95 °C, 123 s (Milk No. 1, UHT plant 1).

UHT preheat treatment	Fouling rate (°C h^{-1})		
	FWM	RCB	Recon
75 °C, 1 s	0.09	0.12	0.43
75 °C, 123 s	0.27	0.35	0.36
95 °C, 123 s	0.32	0.16	0.78

Work on UHT plant 1 was preliminary. The plant was old and was difficult to operate: the trial was not replicated. Instead, work was moved from UHT plant 1 to UHT plant 2 in which the heat exchanger had been purposely designed to facilitate the measurement of fouling rates. The process diagrams of the heat exchangers for UHT plants 1 and plant 2 are shown in Chapter 3.

5.2.2 UHT plant 2, Milk Nos. 2 and 3

Two full replicates, using different batches of milk, Milk Nos. 2 and 3, were used to measure fouling rates for FWM, RCB and Recon. SMP (prepared using evaporator preheat treatment at 75 °C, 2 s) and WMP (prepared using evaporator preheat treatment at 95 °C, 33 s) were, respectively, derived from the same original batches of milk as the corresponding FWM. The experimental design is shown in Chapter 3.

UHT and evaporator preheat treatment were changed at UHT plant 2. The UHT preheat treatment at 75 °C, 123 s from the UHT plant 1 was changed to 85 °C, 147 s for UHT

plant 2 experiments because the intermediate preheat treatment, 75 °C, 123 s, was insufficiently differentiated from the low preheat treatment, 75 °C, 1 s. The preheat treatment at 85 °C was intended to allow investigation of the effect on fouling of an intermediate preheat treatment. The preheat holding time changes from 123 s to 147 s and from 123 s to 155 s were owing to longer connecting tubes being used in UHT plant 2, compared with UHT plant 1.

Evaporator preheat treatments for RCB and Recon were the same as for the UHT plant 1 preliminary trial. The fouling rate experiments were replicated (using Milk Nos. 2 and 3) and the results are shown in Figure 5.1.

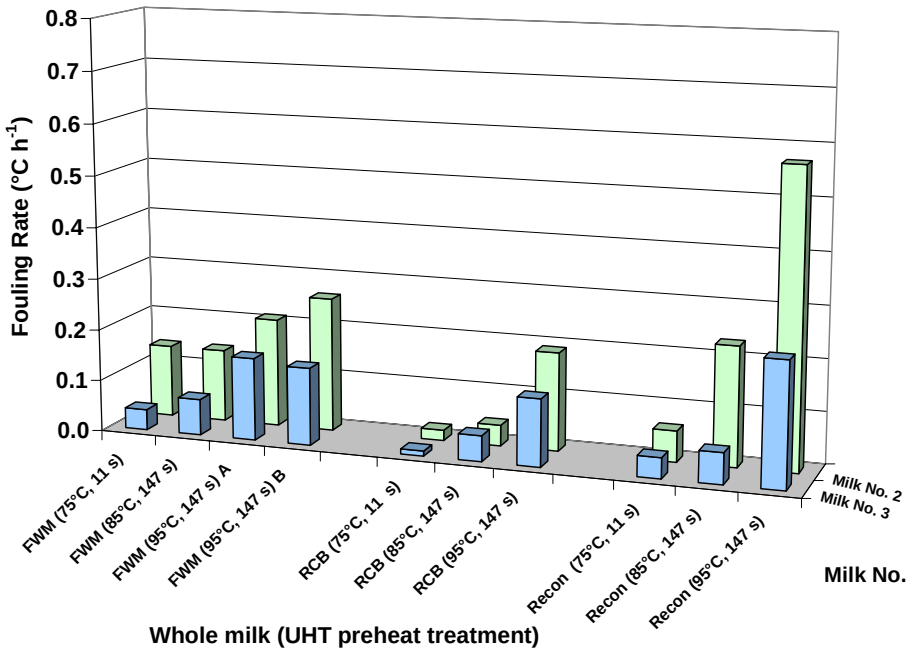


Figure 5.1 The effect of UHT preheat treatment on fouling rates of FWM, RCB and Recon (Milk Nos. 2 and 3). A and B are duplicates.

These results indicated that, in general, relatively small increases in fouling rate occurred when UHT preheating intensity was increased from 75 °C, 11 s to 85 °C, 147 s, but much larger ones occurred when preheating intensity was increased to 95 °C, 147 s. The experimental split-plot design with replicates (i.e., more than one batch of starting milk (milk number)) is shown in Figure 5.2.

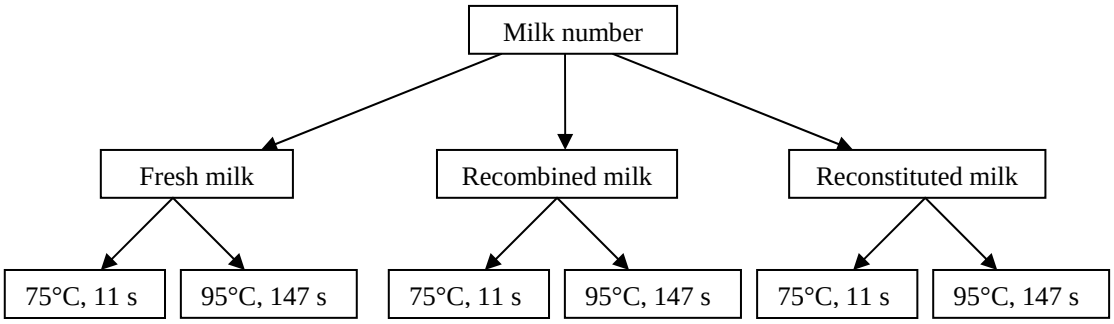


Figure 5.2 Split-plot design with replicates using different batches of milk (Milk Nos. 2 and 3) for investigating the effect of milk preparation (FWM, RCB and Recon) and UHT preheat treatment on fouling rate.

The main plot for this analysis is the factor milk preparation (FWM, RCB or Recon). The sub-plot is the factor UHT preheat treatment, which is nested in the factor milk preparation. The results of the full statistical analysis are reported in Appendix 2 (Table A2.1).

The statistical significance results for the factors of Milk No., milk preparation and UHT preheat treatment within milk preparation are summarised in Table 5.2.

Table 5.2 Probability values (p) from analysis of variance of fouling rates of FWM, RCB and Recon (Milk Nos. 2 and 3).

Factors	p-value[1]
Milk preparation, df = 2, 2 (FWM, RCB and Recon)	0.230
UHT preheat treatment within milk preparation, df = 6, 8	< 0.001
Milk No. (Milk Nos. 2 and 3), df = 1, 2	0.151

[1]Main factors: *UHT preheat treatment, milk preparation and Milk No.*

Geometric means of fouling rates from Figure 5.1 are presented in Table 5.3.

Table 5.3 Geometric means of fouling rates of FWM, RCB and Recon for different UHT preheat treatments (Milk Nos. 2 and 3).

UHT preheat treatment	Geometric mean fouling rate (°C h^{-1})		
	FWM	**RCB**	**Recon**
75 °C, 11 s	0.087	0.017	0.053
85 °C, 147 s	0.10	0.044	0.14
95 °C, 147 s	0.19	0.15	0.40

Geometric means in Table 5.3 showed an increase in fouling rate with the intensity of UHT preheat treatment (over the full range of treatment) for RCB (factor of nine), for Recon (factor of eight) and for FWM (factor of two) ($p = 0.001$).

Different batches (numbers) of fresh whole milk are treated as if the milk batches were true replicates, which strictly is not correct in terms of statistical analysis. However, it was realistic to do this because it was the only way to look at the effect of UHT preheat treatment on UHT fouling with natural variation between batches of FWM. It is not practicable to keep milk for a long period of time as an original large milk batch because the milk would not be stable for long enough to complete all the experiments.

More replicates of FWM, RCB and Recon were used to study the effect of UHT preheat treatment on UHT fouling. The results are shown in the next section (section 5.2.3).

5.2.3 UHT plant 2, Milk Nos. 2, 3 and 6-11

The fouling rate data obtained with Milk Nos. 2 and 3, discussed above, were combined with similar data obtained with Milk Nos. 6-11 and are plotted in Figure 5.3.

The original data were analysed using a split-plot with replicates according to the same experimental design as shown in Figure 5.2. Full results of this statistical analysis for Figure 5.3 data are reported in Appendix 2 (Table A2.2), and summarised p values in Table 5.4.

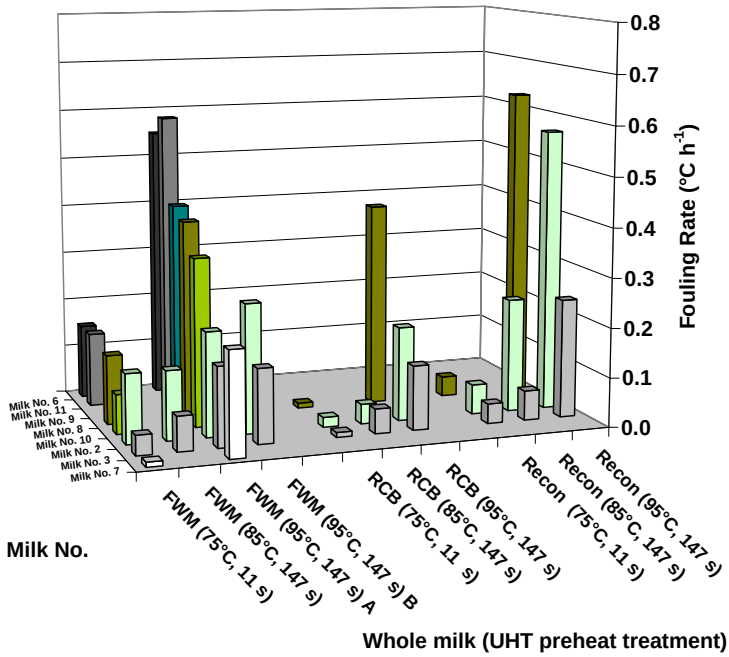


Figure 5.3 The effect of UHT preheat treatments on fouling rates of FWM, RCB and Recon (Milk Nos. 2, 3 and 6-11).

Table 5.4 Probability values (p) from analysis of variance of fouling rates of FWM, RCB and Recon (Milk Nos. 2, 3 and 6-11).

Factors	p-value[1]
Milk preparation, df = 2 (FWM, RCB and Recon)	0.025
UHT preheat treatment within milk preparation, df = 6, 19	< 0.001
Milk No.(Milk Nos. 2, 3 and 6-11), df = 7	0.002

[1]Main factors: *UHT preheat treatment, milk preparation and Milk No.*

The geometric means of fouling rates of FWM, RCB and Recon from Figure 5.3 are presented in Table 5.5.

Table 5.5 Geometric means of fouling rates of FWM, RCB and Recon for different UHT preheat treatments (Milk Nos. 2, 3 and 6-11).

UHT preheat treatment	Geometric mean fouling rate (°C h^{-1})		
	FWM (Milk No. 2, 3 and 6-11)	**RCB** (Milk No. 2, 3 and 8)	**Recon** (Milk No. 2, 3 and 8)
75 °C, 11 s	0.099	0.013	0.047
85 °C, 147 s	0.10	0.044	0.14
95 °C, 147 s	0.32	0.23	0.47

Table 5.5 confirmed and extended the results shown in Table 5.3 showing that fouling rates very strongly increased with the intensity of UHT preheat treatment, nested within milk preparation ($p < 0.001$). It appears that an increase in fouling rate over the full UHT preheating intensity range for RCB (factor of 18) was greater than that of Recon (factor of 10) and both were greater than for FWM (factor of 3). This trend remained similar to that found for Milk Nos. 2 and 3 (section 5.2.2). The high-intensity preheat treatment (95 °C, 147 s) appeared to have had a considerably bigger effect on the fouling rate relative to the low-intensity treatment (75 °C, 11 s) than did the medium-intensity treatment (85 °C, 147 s).

5.3 Effect of evaporator preheat treatment on UHT fouling by RCB

In each of the experiments whose results are reported here, SMPs made with different evaporator preheat treatments were prepared from the same batch of FWM and were recombined with anhydrous milk fat and water to make RCBs. The details of the experimental design are explained in Chapter 3.

The two UHT plants were used in this investigation. Milk No.1 was used to measure fouling rates in UHT plant 1. Milk Nos. 2 and 3 were used to measure fouling rates in UHT plant 2. The results are shown in sections 5.3.1 and 5.3.2.

5.3.1 Effect of evaporator preheat treatment on the fouling rates of RCB (Milk No.1, UHT plant 1)

Three different evaporator preheat treatments (75 °C, 2 s; 75 °C, 155 s; and 95 °C, 155 s) were applied to skim milk, derived from Milk No.1, before drying. After recombination,

RCB was UHT preheated at 75 °C, 11 s prior to UHT processing. The results of fouling rate measurements are shown in Table 5.6.

Table 5.6 Effect of evaporator preheat treatment on fouling rates of RCB for the UHT preheat treatment of 75 °C, 11 s (Milk No. 1, UHT plant 1).

Evaporator preheat treatment	Fouling rate ($°C\ h^{-1}$)
75 °C, 2 s	0.12
75 °C, 155 s	0.18
95 °C, 155 s	0.43

The results in Table 5.6 show that an increase in evaporator preheating intensity appeared to cause an increase in fouling rate. The trend is similar to the effect of UHT preheat treatment (Table 5.1). There was a greater increase in fouling rate when evaporator preheating intensity increased from 75 °C, 155 s to 95 °C, 155 s than when it increased from 75 °C, 2 s to 75 °C, 155 s. This trend was similar to the effect of UHT preheat treatment on the fouling rate (section 5.2).

Further experiments on the effect of evaporator preheat treatment on fouling rates of RCB were carried out on UHT plant 2.

5.3.2 Effect of evaporator preheat treatments on fouling rate of RCB (Milk Nos. 2 and 3, UHT plant 2)

Milk Nos. 2 and 3 were used to measure fouling rate on UHT plant 2. Three different evaporator preheat treatments (75 °C, 2 s, 85 °C, 155 s and 95 °C, 155 s) were applied to skim milk prior to drying.

Evaporator preheat treatments on UHT plant 2 were different from those on UHT plant 1. The evaporator preheat treatment 75 °C, 155 s was changed to 85 °C, 155 s in order to provide more even steps between evaporator preheat treatments. Replicate results (Milk Nos. 2 and 3) are shown in Figure 5.4.

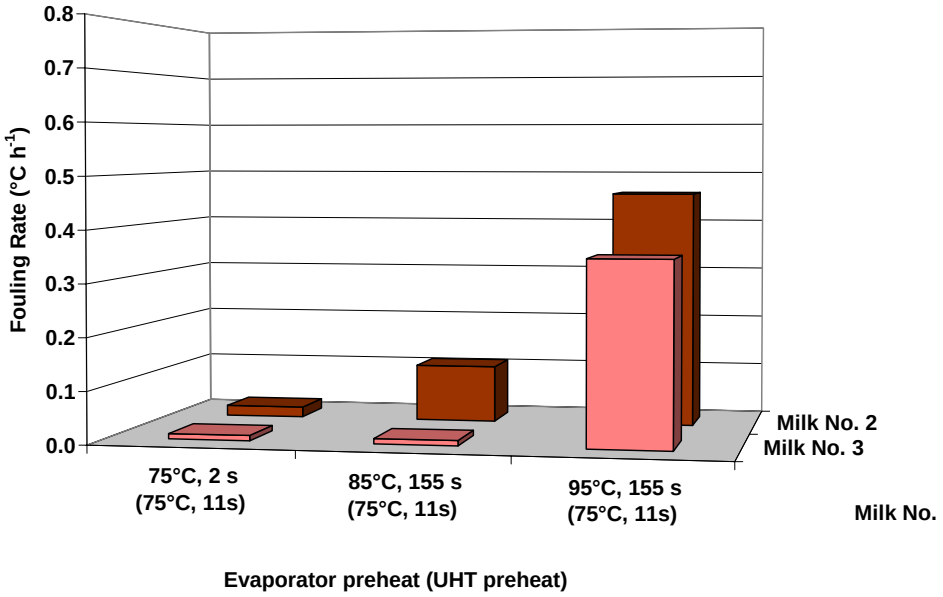


Figure 5.4 The effect of evaporator preheat treatments (75 °C, 2 s; 85 °C, 155 s; and 95 °C, 155 s) on fouling rates of RCB at the UHT preheat treatment of 75 °C, 11 s (Milk Nos. 2 and 3).

The original data on the fouling rate of Milk Nos. 2 and 3 were statistically analysed using two-way analysis of variance because SMPs were prepared from different milk batches. The statistical results in Appendix 2 (Table A2.3) are reported in Table 5.7.

Table 5.7 Probability values (p) from analysis of variance of the fouling rate of RCB (Milk Nos. 2 and 3).

Factors	p-value[1]
Evaporator preheat treatment (75 °C, 2 s, 85 °C, 155 s and 95 °C, 155 s), df = 2	0.018
Milk No. (Milk Nos. 2 and 3), df = 1	0.155

[1]Main factors: *Evaporator preheat treatment and Milk No.*

The geometric means of the original data in Figure 5.4 are reported in Table 5.8.

Table 5.8 Geometric means of fouling rates of RCB at the evaporator preheat treatments of 75 °C, 2 s, 85 °C, 155 s, and 95 °C, 155 s, and the UHT preheat treatment of 75 °C, 11 s (Milk Nos. 2 and 3).

Evaporator preheat treatment	Geometric mean fouling rate ($°C\ h^{-1}$)
75 °C, 2 s	0.017
85 °C, 155 s	0.056
95 °C, 155 s	0.40

Geometric means in Table 5.8 increased with the intensity of evaporator preheat treatment (strong evidence : $p = 0.018$). These results are similar to those shown in Table 5.6 (UHT plant 1) and parallel to those for the effect of UHT preheat treatment demonstrated in sections 5.2.2 and 5.2.3.

The effects of evaporator and UHT preheat treatment on fouling by RCB are compared in detail in the next section (section 5.4).

5.4 A comparison of the effects of UHT and evaporator preheat treatments on fouling rates of RCB (Milk Nos. 2 and 3, UHT plant 2)

The results showing the effect of UHT preheat treatment on fouling rate for Milk Nos. 2 and 3 (milk preparations: FWM, RCB and Recon; section 5.2.2) and the results showing the effects of evaporator preheat treatment for Milk Nos. 2 and 3 (RCB; section 5.3.2) are plotted together in Figure 5.5. These results were analysed to compare the effects of UHT and evaporator preheat treatments on fouling rates of RCB.

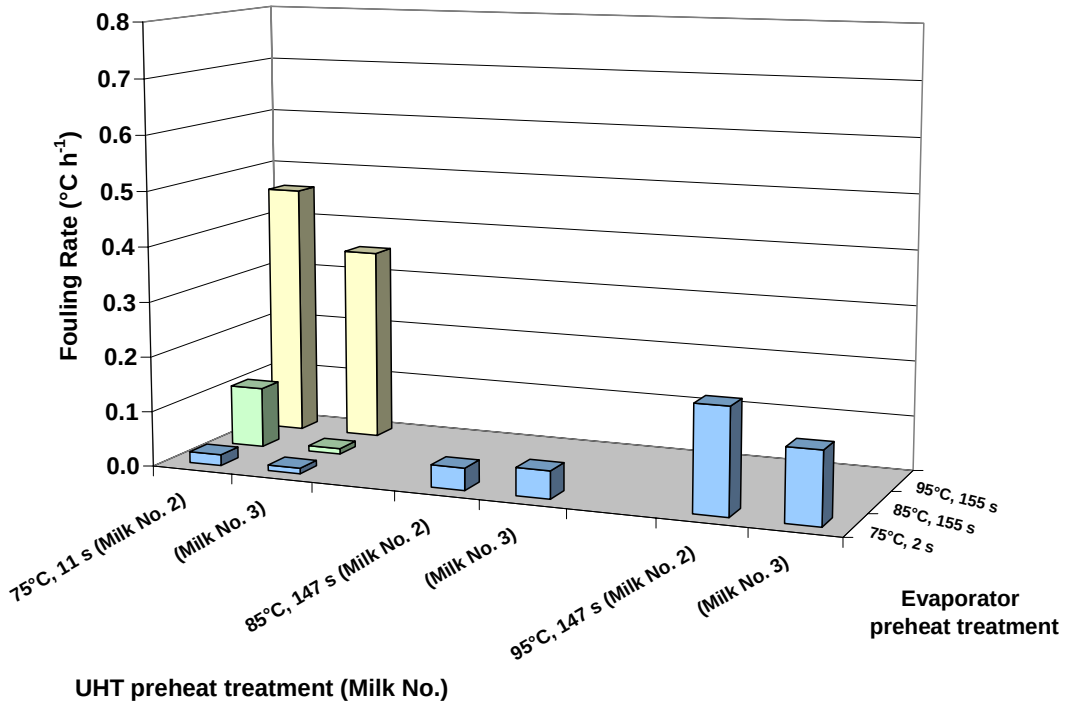


Figure 5.5 The effect of UHT and evaporator preheat treatments on the fouling rate of RCB (Milk Nos. 2 and 3, UHT plant 2).

The geometric mean fouling rates in Figure 5.5 are presented in Table 5.9.

Table 5.9 Geometric means of fouling rates of RCB for different UHT and evaporator preheat treatments (Milk Nos. 2 and 3, UHT plant 2).

Evaporator preheat treatment	Geometric mean fouling rate for different UHT preheat treatments (°C h^{-1})		
	75 °C, 11 s	85 °C, 147 s	95 °C, 147 s
75 °C, 2 s	0.017	0.044	0.23
85 °C, 155 s	0.056	n/a[1]	n/a
95 °C, 155 s	0.40	n/a	n/a

[1]n/a: No measurements made for these combinations.

Figure 5.5 and Table 5.9 show that the effects of evaporator preheat treatment during skim milk powder manufacture on the fouling rate of derived RCB had a very similar pattern to that observed for the effects of UHT preheat treatment (section 5.2 and 5.3). The fouling

rate increased with evaporator preheating intensity, and the high-intensity preheat treatment (95 °C, 155 s) had a markedly bigger effect on the fouling rate relative to the low-intensity treatment (75 °C, 2 s) than did the medium-intensity treatment (85 °C, 155 s).

To investigate further the effects of evaporator preheating, the fouling rates of Recon made from low heat and high heat whole milk powder were measured. The results are presented in the next section (section 5.5).

5.5 Effect of evaporator preheat treatment and UHT preheat treatment on the fouling rate of Recon (Milk Nos. 4 and 5, UHT plant 2)

Whole milk powder (WMP) was made from Milk Nos. 4 and 5, using two evaporator preheat treatments, 75 °C, 2 s and 95 °C, 155 s, prior to drying. Then, each WMP was reconstituted with water and the Recon was UHT preheated at 75 °C, 11 s and 95 °C, 147 s prior to UHT sterilization, during which fouling rates were measured. The design and processing procedures for this experiment are described in Chapter 3. The results are shown in Figure 5.6.

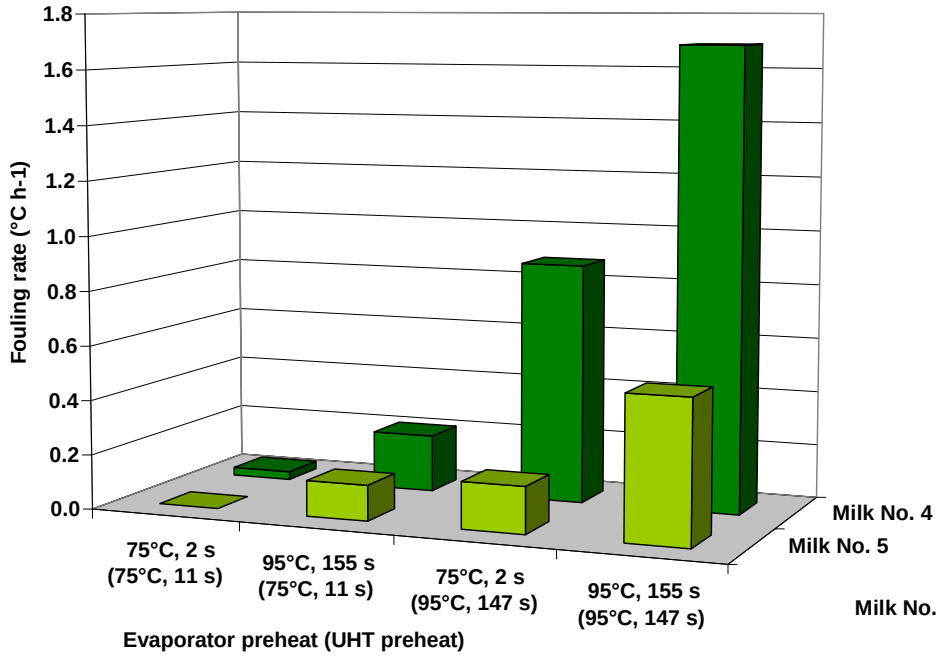


Figure 5.6 The effect of evaporator and UHT preheat treatments on the fouling rate of Recon (Milk Nos. 4 and 5).

The split-plot analysis with replicates was applied to the original data according to experimental design shown in Figure 5.7.

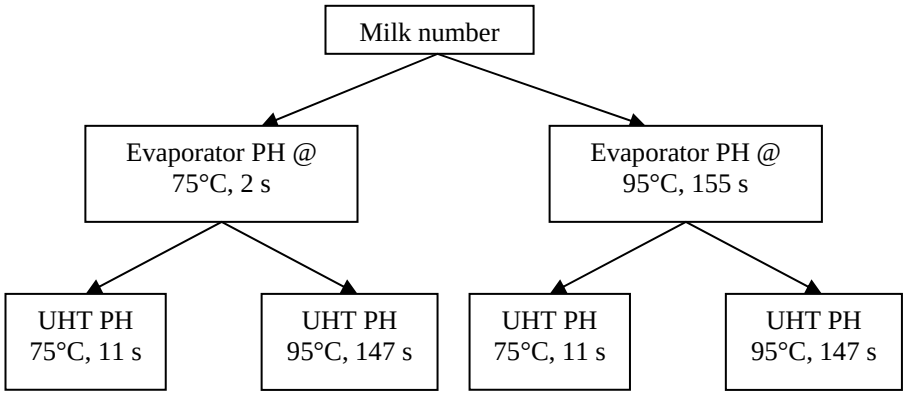


Figure 5.7 Split-plot design with replicates using different milk batches (Milk Nos. 4 and 5) for investigating the effects of evaporator and UHT preheat treatments on the fouling rate of Recon.

The effect of evaporator preheat treatment is the main plot and the effect of UHT preheat treatment is the sub-plot. The results of the statistical analysis based on Figure 5.7 are reported fully in Appendix 2 (Table A2.4), and summarised in Table 5.10.

Table 5.10 Probability values (p) from analysis of variance of the effect of evaporator preheat treatment on the fouling rate of Recon (Milk Nos. 4 and 5).

Factors	p-value[1]
UHT preheat treatment within evaporator preheat treatment, df = 2, 2	0.202
Evaporator preheat treatment (75 °C, 2 s and 95 °C, 155 s), df = 1, 1	0.095
Milk No.(Milk Nos. 4 and 5), df = 1, 1	0.075

[1]Main factors: *UHT preheat treatment, evaporator preheat treatment and Milk No.*

The original data on fouling rate presented in Figure 5.6 were transformed for analysis as already described (section 5.2). The resulting geometric mean fouling rates are presented in Table 5.11.

Table 5.11 Geometric means of fouling rates of Recon (Milk Nos. 4 and 5).

Evaporator preheat treatment	Geometric mean fouling rate for different UHT preheat treatments (°C h^{-1})	
	75 °C, 11 s	**95 °C, 147 s**
75 °C, 2 s	0.015	0.48
95 °C, 155 s	0.17	1.022

Geometric means of fouling rates at a given UHT preheat treatment (Table 5.11) increased with the intensity of evaporator preheat treatment ($p = 0.095$). Although the apparent increase in fouling rate due to increased UHT preheat intensity within evaporator preheat treatment was not significant ($p = 0.202$), this increasing trend of the fouling rate of Recon constitutes a similar trend to the increase of the fouling rate of RCB with the intensity of evaporator preheat treatment, as reported in Tables 5.6 and 5.8.

5.6 Effect of UHT preheat treatment on fouling by homogenized then preheated FWM and preheated then homogenized FWM

The results for FWM, RCB and Recon presented in section 5.2 showed that fouling rate increased with the intensity of UHT preheat treatment for all batches of milk (Milk Nos. 1, 2, 3 and 6-11). The contrast between these results and those reported by Bell & Sanders (1944), Burton (1968), Lalande *et al.* (1984), Patil and Reuter (1986a) and Mottar and Moermans (1988), as showing the opposite trend for fresh whole milk, were startling. This led to speculation that the cause may have been due to some overlooked general difference in the starting milk. All the milk in the present study was homogenized before preheat treatment: while the milk used by Patil and Reuter (1986a) and Burton (1968) does not appear to have been homogenized at all, and in the Lalande *et al.* (1984) and Mottar and Moermans (1988) studies, the milk was homogenized after preheating. This being the only obvious difference in milk that could be established, the effects of UHT preheat treatment before and after homogenization were compared. (The milk used by Bell & Sanders (1944), Burton (1968), Patil and Reuter (1986a) and Mottar and Moermans (1988) was raw fresh whole milk, in contrast to the pasteurized and standardized FWM used in this work. This difference is not deemed to be significant with respect to fouling rate, as the fouling rates measured for the least intense preheat treatment in the present study were very low).

The data obtained with Milk Nos. 6 and 7 were combined with similar data obtained with Milk No. 9. All of the data are presented in Figure 5.8, and were analysed together.

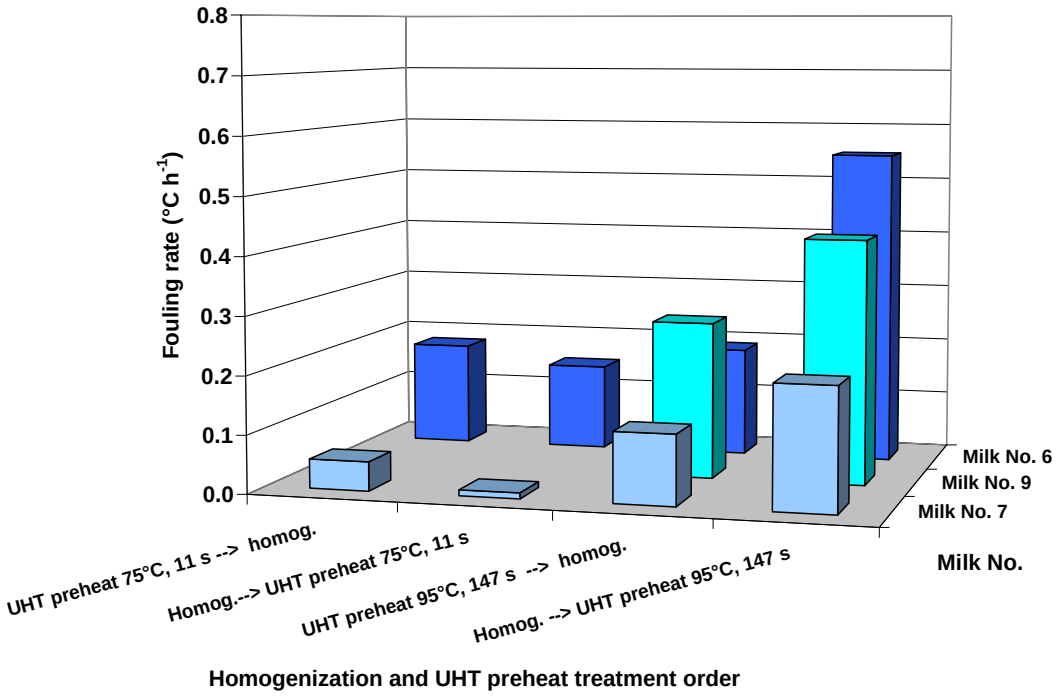


Figure 5.8 The effect of homogenization and UHT preheat treatment order on the fouling rates of FWM (Milk Nos. 6, 7 and 9).

Geometric means of fouling rates shown in Figure 5.8, and the results of the statistical analysis shown in Tables A2.5, are presented in Table 5.12.

Table 5.12 Geometric means of fouling rates of FWM with different orders of homogenization and UHT preheat treatment (Milk Nos. 6, 7 and 9).

UHT preheat treatment	**Geometric mean fouling rate (°C h^{-1})**	
	Homogenized then preheated FWM	**Preheated then homogenized FWM**
75 °C, 11 s	0.076 (Milk Nos. 6 and 7)	0.11 (Milk Nos. 6 and 7)
95 °C, 147 s	0.39 (Milk Nos. 6, 7 and 9)	0.19 (Milk Nos. 6, 7 and 9)
p-value	0.149	0.416

These results in Table 5.12 confirmed that fouling rate increased with the intensity of UHT preheat treatment when FWM was homogenized then UHT preheated ($p = 0.149$). The fouling rates of preheated then homogenized FWM showed much less effect of UHT preheat treatment, but still did not show a trend of fouling rate decreasing with the intensity of UHT preheat treatment. This result disagrees with Mottar and Moermans (1988), whose milk was preheated and then homogenized at the preheating temperature, and with those of Bell & Sanders (1944) and Patil & Reuter (1986a) who did not homogenize their milk at all.

5.7 Effect of homogenization on UHT fouling by FWM (Milk Nos. 10 and 11, UHT plant 2)

Fresh whole milk is usually subjected to homogenization in indirect UHT plants. Homogenization normally takes place prior to sterilization to delay fat separation in the UHT milk container. Although homogenization cannot be avoided in commercial milk plants, the relative effects of homogenization and no homogenization on the fouling rate of raw fresh whole milk were investigated because Bell & Sanders (1944), Burton (1968) and Patil & Reuter (1986a) used non-homogenized raw fresh whole milk when they reported that the extent of fouling decreased with the intensity of UHT preheat treatment. FWMs Nos.10 and 11 were each processed with homogenization and with no homogenization prior to UHT preheating at 75 °C, 11 s and 95 °C, 147 s, and fouling rates measured during subsequent UHT sterilization. The results are shown in Figure 5.9.

The geometric means of the fouling rates shown in Figure 5.9 and the results from the analysis of variance are shown in Table 5.13. The full statistical analysis is reported in Appendix 2 (Tables A2.6 and A2.7).

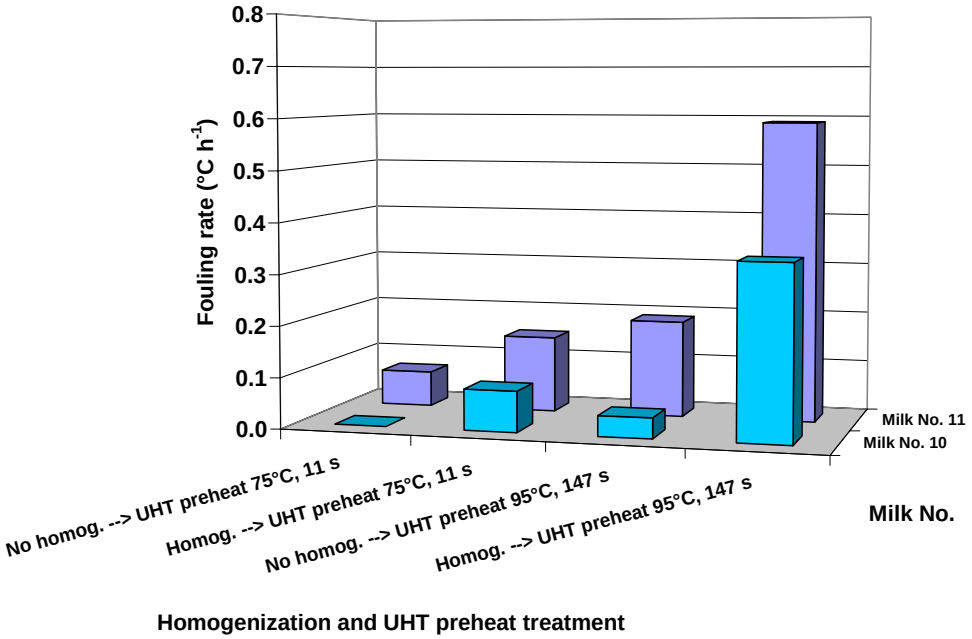


Figure 5.9 The effect of homogenization and no homogenization on the fouling rate of FWM (Milk Nos. 10 and 11).

Table 5.13 Geometric means of fouling rates of FWM with homogenization and with no homogenization prior to UHT preheat treatment (Milk Nos. 10 and 11).

UHT preheat treatment	**Geometric mean fouling rate (°C h^{-1})**		p-value
	Homogenization then preheat treatment	**No homogenization then preheat treatment**	
75 °C, 11 s	0.12	0.034	0.025
95 °C, 147 s	0.46	0.11	0.046
p-value	0.127	0.260	-

The results in Table 5.13 showed that the geometric means of the fouling rates of homogenized then preheated FWM were greater than those for unhomogenized FWM at the UHT preheat treatments 75 °C, 11 s and 95 °C, 147 s. Both fouling rates of homogenized then preheated FWM and not homogenized and then preheated FWM increased with the intensity of UHT preheat treatment. The fouling rate in this study did not decrease with the intensity of UHT preheat treatment as reported by Bell & Sanders

(1944), Burton (1968) and Patil & Reuter (1986a), who used unhomogenized fresh whole milk.

Although investigating the effect of the presence of protein coated fat globules on UHT fouling of FWM was not the main objective in this chapter, it was necessary to have information on this to aid interpretation of the results on the effects of homogenization. This was examined using skim milk powder to prepare reconstituted skim milk and RCB. The results are reported in section 5.8.

5.8 Effect of the presence of fat globules on UHT fouling by RCB (Milk Nos. 2a and 3a, UHT plant 2)

To investigate the effect of the presence of fat globules on fouling rates, fouling rates of homogenized reconstituted skim milk and RCB, prepared from the same batches of SMP, were measured and compared. Two batches of SMP1 (evaporator preheat treatment 75 °C, 2 s) derived from Milk Nos. 2a and 3a, respectively, were used in this study. The results are shown in Figure 5.10.

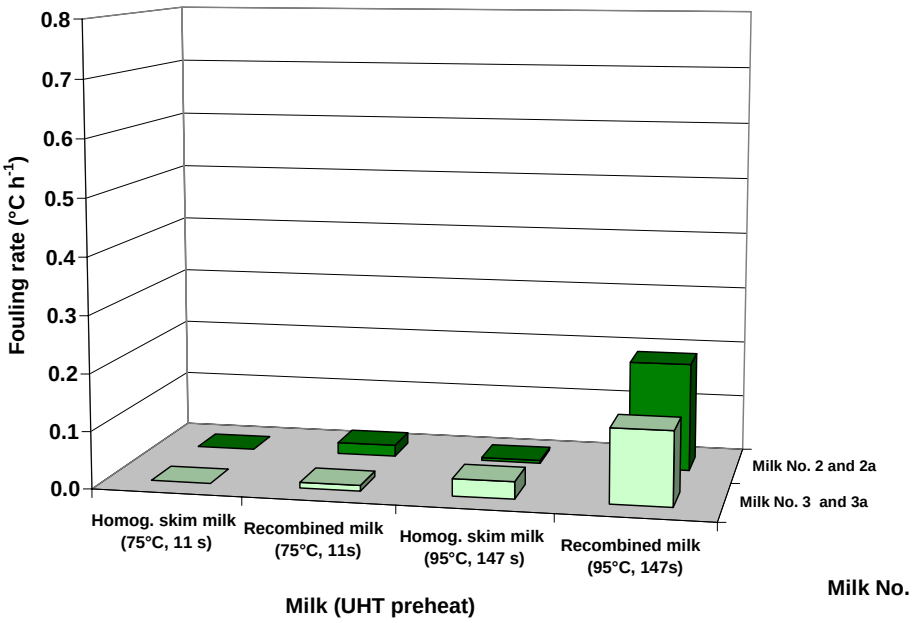


Figure 5.10 Fouling rates of homogenized reconstituted skim milk and RCB (Milk Nos. 2a and 3a: reconstituted skim milk; Milk Nos. 2 and 3: RCB).

The results of the one-way analysis of variance in Table A2.8 are presented in Table 5.14, and geometric means of the duplicate fouling rates shown in Figure 5.10 are summarised in Table 5.15.

Table 5.14 Probability values (p) from analysis of variance for the effect of homogenization and UHT preheat treatment on the fouling rates for reconstituted skim milk and RCB (Milk Nos. 2a and 3a: reconstituted skim milk; Milk Nos. 2 and 3: RCB).

Factors	p-value[1]
Combination of milk preparation and UHT preheat treatment, df = 3 (reconstituted skim milk with UHT preheat treatment 75 °C, 11 s, reconstituted skim milk with UHT preheat treatment 95 °C, 147 s, RCB with UHT preheat treatment 75 °C, 11 s, and RCB with UHT preheat treatment 95 °C, 147 s)	0.033

[1]Main factor: Combination of milk preparation and UHT preheat treatment.

Table 5.15 Geometric means of fouling rates of homogenized reconstituted skim milk and RCB (Milk Nos. 2a and 3a).

UHT preheat treatment	Geometric mean fouling rate (°C h^{-1})	
	Homogenized reconstituted skim milk	RCB
75 °C, 11 s	0.00	0.017
95 °C, 147 s	0.017	0.154

Table 5.15 shows that at the preheat treatment 95°C, 147 s the fouling rates of reconstituted skim milk were slightly higher when fat globules were present. There was moderate evidence that the fouling rate of RCB at the preheat treatment 95°C, 147 s was the greatest among the different combinations of milk preparation (reconstituted skim milk and recombined milk) and UHT preheat treatment (75 °C, 11 s and 95 °C, 147 s) ($p = 0.033$).

5.9 Effect of homogenization on the fouling rate of reconstituted skim milk (Milk Nos. 2a and 3a)

As shown in sections 5.7 and 5.8, homogenization affected fouling rates of FWM and RCB, which contain fat globules. When the effect of homogenization of skim milk was investigated (section 5.8), the effect of preheat treatment on fouling rate showed a trend similar to that shown by FWM and RCB. Therefore, the effect of homogenization on milk plasma alone was investigated by measuring the fouling rate of homogenized and unhomogenized reconstituted skim milk. Reconstituted skim milk was prepared from SMP1 (evaporator preheat treatment: 75 °C, 2 s). Two batches of SMP1 derived from Milk Nos. 2 and 3 were used in this study. After reconstitution, one portion of the skim milk was subjected to homogenization and the other was not. Fouling rates were then measured at two UHT preheat treatments (75 °C, 11 s and 95 °C, 147 s). The results are shown in Figure 5.11.

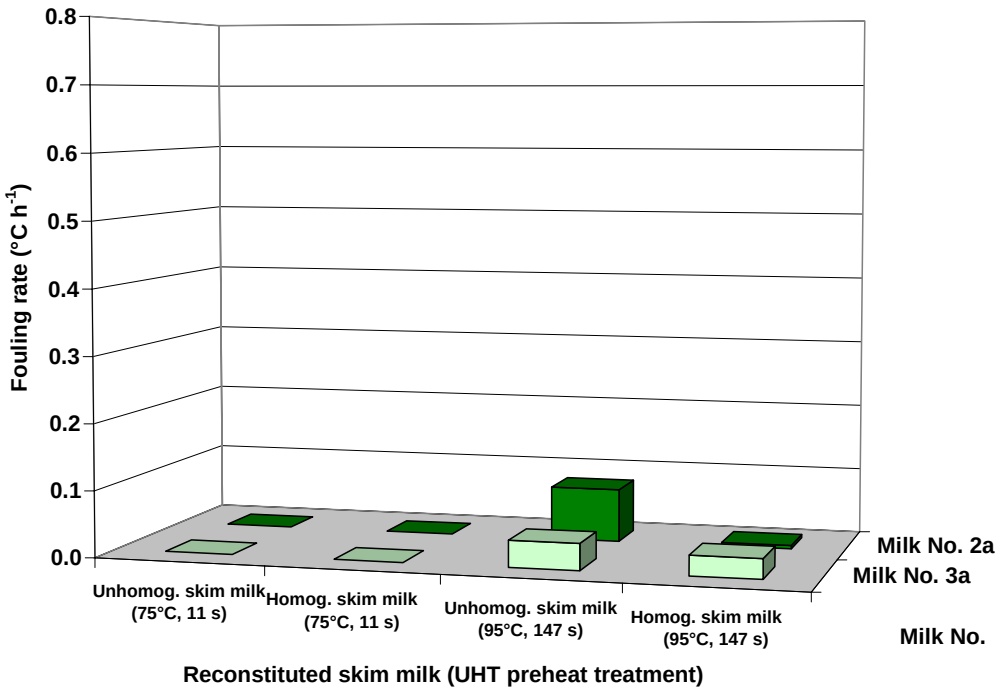


Figure 5.11 The effect of homogenization and UHT preheat treatment on the fouling rate of reconstituted skim milk (Milk Nos. 2a and 3a).

The results of one-way analysis of variance in Appendix 2 (Table A2.9) are presented in Table 5.16 and geometric means of the fouling rates shown in Figure 5.11 are summarised in Table 5.17.

Table 5.16 Probability values (p) from analysis of variance for the effect of homogenization and UHT preheat treatment on the fouling rate of reconstituted skim milk (Milk Nos. 2a and 3a).

Factors	p-value[1]
Combination of homogenization and UHT preheat treatment, df = 3 (reconstituted skim milk with homogenization 75 °C, 11 s, reconstituted skim milk with homogenization 95 °C, 147 s, reconstituted skim milk with no homogenization s 75 °C, 11 s, and reconstituted skim milk with no homogenization 95 °C, 147 s)	0.081

[1]Main factor: Combination of homogenization and UHT preheat treatment.

Table 5.17 Effect of homogenization on the fouling rate of reconstituted skim milk (Milk Nos. 2a and 3a).

UHT preheat treatment	Geometric mean fouling rate (°C h^{-1})	
	Homogenization	No homogenization
75 °C, 11 s	0.00	0.00
95 °C, 147 s	0.017	0.060

The results (Table 5.17) showed that all the fouling rates of reconstituted skim milk were low and there was no reduction in fouling rate with the intensity of UHT preheat treatment. The geometric mean of the fouling rates of unhomogenized reconstituted skim milk at the UHT preheat treatment of 95 °C, 147 s was the greatest among the combinations of homogenization and UHT preheat treatment (p = 0.081).

It can be concluded that any effects of homogenization of reconstituted skim milk on fouling were minor, compared with those obtained when fat globules were present.

5.10 Effect of milk powder ageing

Aged skim milk powder (SMP1, 2 and 3) and aged whole milk powder (Milk No. 2b) were used to prepare RCB and Recon. Fouling rates of these milk preparations were measured and compared with the previously obtained fouling rates of the corresponding freshly prepared RCB and Recon (Milk No. 2). The results (Tables A2.10 and A2.11) showed that

there was no significant evidence for an effect of milk powder ageing on the fouling rate of derived RCB and derived Recon.

5.11 Discussion

Effect of UHT preheat treatment on the fouling rate of FWM, RCB and Recon as normally prepared for UHT processing

In the present work, the more intense UHT preheat treatment of all milk preparations resulted in higher fouling rates in the high-temperature section of the UHT plant (Table 5.3 and 5.5). These trends were well replicated and the results were fully consistent even though overall fouling rates varied between milk batches. This means that although changes in milk composition with year, time of year and stage of lactation may affect the absolute fouling rates, they do not influence the general effect of UHT preheating on these fouling rates. Although Gray (1988) and Auldist *et al.* (1998) showed that levels of milk components clearly vary with the stage of lactation, year, and time of year, the present study confirms that these factors do not affect the trend of the effect of UHT preheating on the fouling rate when different milk batches are used.

The fouling rate, as measured after homogenization and subsequent preheat treatment varied among three different milk preparations, FWM, RCB and Recon (Table 5.4; $p = 0.025$). Although every whole milk was prepared to standardized fat and total solids contents, the fouling rate of Recon was generally greater than the fouling rates of FWM and RCB. This trend was different from that reported by Newstead *et al.* (1999), namely that UHT fouling rates of RCB were greater than those of Recon. This is possibly due to differences in evaporator preheat treatments used for skim milk powder and whole milk powder between Newstead *et al.* (1999) and this study, and differences in geographic regions for fresh whole milk. Newstead *et al.* (1999) used a variety of commercial powders for which the preheat treatments were not specifically controlled from batch to batch (David Newstead, personal communication, 2003). In this study, the evaporator preheat treatment of 95 °C, 33 s for whole milk powder (for Recon) was greater than that for skim milk powder (for RCB), which RCB was prepared from SMP1 with evaporator preheat treatment of 75 °C, 2 s. This could have caused the fouling rates of Recon to be greater than those of RCB.

Newstead *et al.* (1999) reported that relatively severe UHT preheating (90 °C, 120 s) increased fouling rates of FWM, RCB and Recon, but the results did not show clear trends with intensity of preheat treatment applied before homogenization for FWM, milk batches or whole milk preparation. The principal aim of the work in this chapter was to confirm the trend of the effect of UHT preheat treatment on fouling rate. The present work confirms that fouling rates of pasteurised and homogenized FWM, RCB and Recon increase with the severity of UHT preheat treatment for different milk batches (Tables 5.1, 5.3, 5.5 and 5.13). Thus, this study confirms and extends the work of Newstead *et al.*

The results for FWM in this study (Tables 5.1, 5.3 and 5.5) are in conflict with those of Bell & Sanders (1944), Burton (1968), Lalande *et al.* (1984), Patil & Reuter (1986a) and Mottar & Moermans (1988), who reported that UHT preheat treatment reduced fouling of the UHT plant by fresh whole milk. It was suspected, initially, that differences in starting material, the method of measuring the extent of fouling, homogenization and/or order of homogenization and UHT preheat treatment may have been reasons for this opposite trend.

Pasteurized FWM was used as the starting milk material in this work whereas raw fresh whole milk was used in all of those earlier studies except that of Lalande *et al.* (1984). This was due to the regulatory requirement that the milk had to be pasteurised before it entered the pilot plant used. The effect of pasteurisation on UHT plant fouling was expected to be quite small. Although the effect of pasteurisation *per se* on UHT fouling was not included in this study, the small response to heat treatment at 75 °C, 123 s, compared with 1 s (Table 5.1), confirmed this expectation.

The method of measurement of fouling varied with the different workers. Bell & Sanders (1944) reported that the rate of increase of pressure loss in the UHT plant became smaller with the intensity of UHT preheat treatment. Burton (1968) used a hot-wire laboratory method and found that the deposit on the hot wire decreased with the severity of preheating. Patil & Reuter (1986a) and Mottar & Moermans (1988) measured UHT fouling in terms of the deposit weight in a plate heat exchanger and found that the deposit weight decreased with the intensity of preheating. These results show that differences in the method of fouling measurement among these workers did not change the dependence of UHT fouling on UHT preheating; that is, all studies (except that of Mottar & Moermans (1988) who homogenized the milk after preheating) indicated that preheating reduced

fouling by raw, unhomogenized fresh whole milk. The method used to measure fouling rates in this study was the rate of change of a temperature difference, which reflected the gradual decrease in the rate of heat transfer in the high-temperature heater owing to deposit formation. The effects of homogenization and of the order of homogenization and UHT preheat treatment on the fouling rate of FWM are further discussed below.

Effect of evaporator preheat treatment on fouling rates of RCB and Recon

Fouling rates of both RCB and Recon increased with the intensity of evaporator preheat treatments (Table 5.9 for RCB and Table 5.11 for Recon). These similar trends suggest that the cause may be the same for different milk preparations although the absolute fouling rate at a particular preheat treatment varied with milk batch (Milk No.). These batch-to-batch differences must have been largely due to variations in milk composition mainly with year and the stage of lactation (Gray, 1988; Auldist *et al.*, 1998) as discussed earlier (In New Zealand, for bulk herd milk, stage of lactation and time of year are equivalent).

Comparison between the effect of evaporator preheat treatment and the effect of UHT preheat treatment

Preheating had the same general effects on the fouling rate of RCB and Recon whether it was applied prior to evaporation or prior to UHT processing. Evaporator preheat treatment and UHT preheat treatment had a cumulative effect on the fouling rate of RCB (Table 5.9) and Recon (Table 5.11) for a given UHT preheat treatment. Fouling rates of RCB were higher when the evaporator preheat treatment was more severe (Tables 5.6 and 5.8) and at a fixed evaporator preheat treatment, the fouling rate of RCB was higher when UHT preheat treatment was more severe (Tables 5.1, 5.3 and 5.5). A similar trend occurred with the fouling rate of Recon (Table 5.11).

An experimental comparison was not made between the fouling rates of reconstituted skim milk, prepared using the same evaporator preheating as in the case of whole milk powder, and the fouling rates of Recon (from the same original milk batch) because high-heat skim milk powder is not widely used in UHT processed products.

Effect of homogenization

Homogenization appears to be a processing step of some significance with respect to the influence of preheat treatment on UHT fouling. When fat globules are present, the reduction of the size of fat globules by homogenization of FWM caused a higher fouling rate (Table 5.13). The same data show that the fouling rate of FWM with no homogenization did not decrease with the intensity of UHT preheat treatment at all, compared with FWM that was subjected to homogenization. It might be expected that the effect of homogenization would be the result of its effect on fat globules. This is supported by the results for reconstituted skim milk and RCB (Table 5.15). These results show that the presence of fat globules is important for the effect of UHT preheat treatment on UHT fouling. Furthermore, the results in Table 5.17 show that there was no major effect of UHT preheat treatment of reconstituted skim milk on UHT fouling, i.e. when the fat globules were not present. Thus, the existence of fat globules has an important influence on UHT fouling.

Differences in the MFGMs of different types of whole milk were postulated to relate to UHT fouling by Newstead *et al.* (1999). As FWM, RCB and Recon are subjected to different processing steps, their MFGMs would be expected to differ.

- When pasteurised FWM is homogenized, homogenization increases the total surface area of the fat globules because the average size of the fat globules decreases. The new surface membrane of fat globules in homogenized FWM consists of casein micelles, casein fragments, some whey protein and some native MFGM. Incorporation of whey protein into the MFGM of homogenized then preheated or preheated then homogenized raw fresh whole milk occured on preheating (Dalgleish & Sharma, 1993; Sharma & Dalgleish, 1993; Sharma & Dalgleish, 1994; Van Boekel & Walstra, 1995; Lee & Sherbon, 2002). Thus, the composition of the MFGM of homogenized then preheated pasteurised FWM prior to UHT processing is likely to consist of casein micelles, casein sub-micelles, whey protein and some native MFGM.
- When skim milk powder is recombined with anhydrous milk fat, the fat globule membrane formed consists mainly of casein micelles, whey proteins, aggregates of whey proteins themselves and aggregates of whey proteins and casein micelles (Oortwijn & Walstra, 1979; McCrae & Muir, 1991; Sharma *et al.*, 1996a, 1996b).

There is no natural MFGM in the MFGM of RCB. This is a major difference between RCB on the one hand, and Recon and FWM on the other.

- In the case of Recon, FWM with native MFGM is evaporator preheated, evaporated, dried, reconstituted and homogenized, and then preheated prior to UHT processing. All milk constituents, including fat globules and their membranes, casein micelles and whey protein, were preheated twice - once prior to evaporation, and again prior to UHT treatment. Thus, the composition of the MFGM of Recon prior to UHT processing consisted of some natural MFGM (this differs from the MFGM of FWM, according to the processing steps during milk powder manufacture), casein micelles, casein sub-micelles, whey protein, aggregates of whey protein and casein micelles-whey protein complexes. The composition of the MFGM of Recon would be expected to be similar to that of the MFGM of homogenized FWM.

Effect of homogenization before or after UHT preheat treatment

When the effect of homogenization (Tables 5.12 and 5.13) was further investigated, it was found that increasing the intensity of UHT preheat treatment increased fouling rate whether homogenization was applied before or after UHT preheat treatment, and when homogenization was not applied at all.

Raw data for all fouling trials are shown in Appendix 1 (Table A1.1). They show that there was no measurable evidence of fouling in the preheater and the intermediate heater. There are some data in Table A1.1 that suggest fouling occurred in the preheater and intermediate heater but there is no consistency. The results for the measurement of deposit formation in the preheater and intermediate heater are discussed in the Chapter 7.

5.12 Conclusion

The fouling rate of FWM, RCB and Recon increases with the intensity of UHT preheat treatment and also with the intensity of evaporator preheat treatment of RCB and Recon. The cumulative effect of evaporator and UHT preheat treatments of RCB (prepared from the same SMP) shows that the effect of evaporator preheat treatment is greater than the effect of UHT preheat treatment. The conclusion in this present work was that the fouling rate does not decrease with the intensity of UHT preheat treatment, whether FWM is

homogenized before or after UHT preheat treatment, or is not homogenized at all and this is at odds with the results of Bell & Sanders (1944), Burton (1968), Lalande *et al.* (1984), Patil & Reuter (1986a) and Mottar & Moermans (1988), who reported a reduction of UHT fouling by fresh whole milk with increasing intensity of UHT preheat treatment. The results for reconstituted skim milk and RCB show that the role of fat is important. When fat globules are not present, the effect of homogenization on the fouling rate of reconstituted skim milk is minor. Speculation on the possible relationship between the effect of preheat treatment on fouling rate and the compositions of the MFGMs of FWM, RCB and Recon led to investigation of these compositions. The results are reported in Chapter 6.

Chapter 6
Effect of preheat treatments on the milk fat globule membrane of whole milks

6.1 Introduction

The results in Chapter 5 showed that fouling rates increased with the severity of both UHT and evaporator preheat treatments. Differences in the milk fat globule membrane (MFGM, any membrane formed on the surface of fat globules) of FWM, RCB and Recon were considered as possible causes of the differences in fouling rate among these milk preparations. Reports on the fat and protein contents of the material deposited by FWM in the high-temperature heater (Burton, 1968; Lalande *et al.*, 1984; Visser *et al.*, 1997) do not consider any relationship between differences in the MFGM, fouling rate, and the intensity of UHT and evaporator preheat treatments. Therefore, heat-induced changes in the MFGMs of FWM, RCB and Recon were further studied. The results are reported in this chapter.

There were three objectives in this study.
1. To investigate the effect of UHT preheat treatment and heating process stage on the fat-bound protein in the MFGMs of FWM, RCB and Recon.
2. To investigate the effect of evaporator preheat treatment on the fat-bound protein in the MFGM of RCB.
3. To investigate the relative effects of preheat treatment before and after homogenization on the fat-bound protein in the MFGM of FWM.

6.2 Experimental approach to measurement of total fat-bound protein and fat-bound individual proteins in FWM, RCB and Recon

The total protein in the cream layer obtained by centrifuging a milk sample was determined by the Kjeldhal nitrogen method and the individual proteins in the cream layer were determined by PAGE analysis as described in Chapter 3. These cream layers, obtained upon centrifugation of the various milk preparations, contained about 40-50 % moisture as

a component of trapped milk serum. This milk serum contained non-fat-bound protein (refered to as “top-serum protein” in this section).

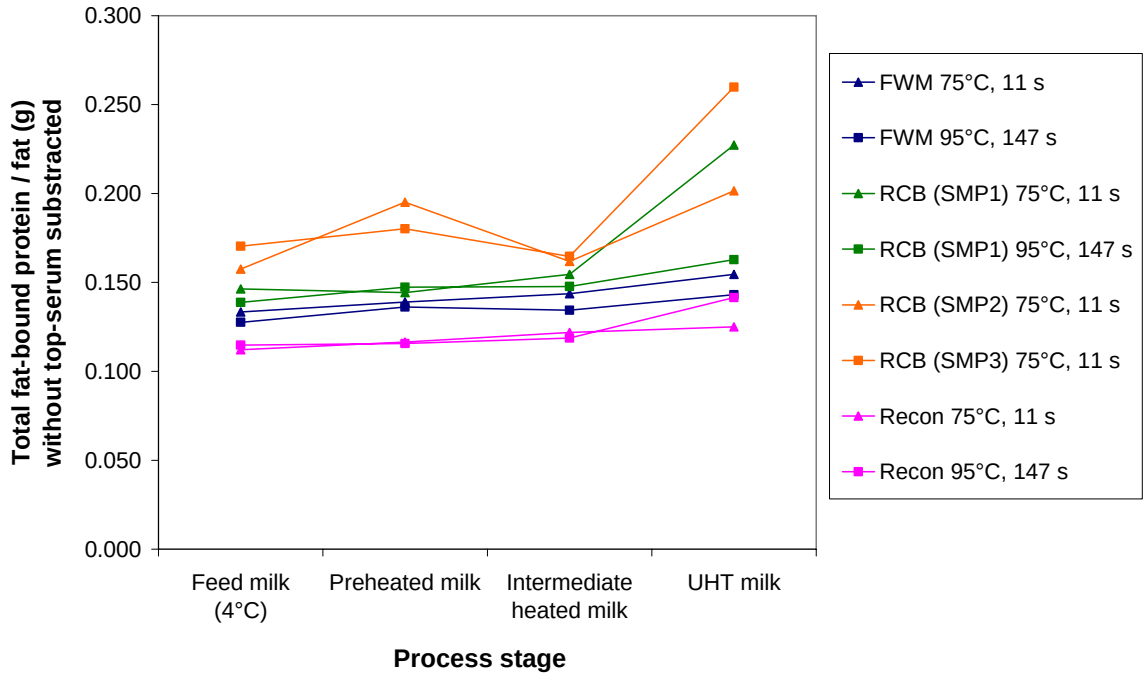
At the g-force used, the serum protein and casein micelles in the serum layer partially sedimented, so that after centrifugation the serum protein and casein micelles in the top-serum fraction, immediately under the cream layer and, presumably, the serum trapped in the cream layer itself, were considerably depleted in protein, particularly micellar casein protein, relative to the bulk of the serum portion. The protein content of the top-serum fraction was taken as an estimate of the non-fat-bound protein in the trapped serum portion of the cream layer and was substracted from the total protein content of the cream layer to give the true “corrected” fat-bound protein content. Calculations are shown in sections 3.12.1 and 3.12.2.

6.2.1 Effect of correcting for top-serum protein in the cream layers of FWM, RCB and Recon as measured by examining the variation of fat-bound protein with preheat treatment and heating process stage

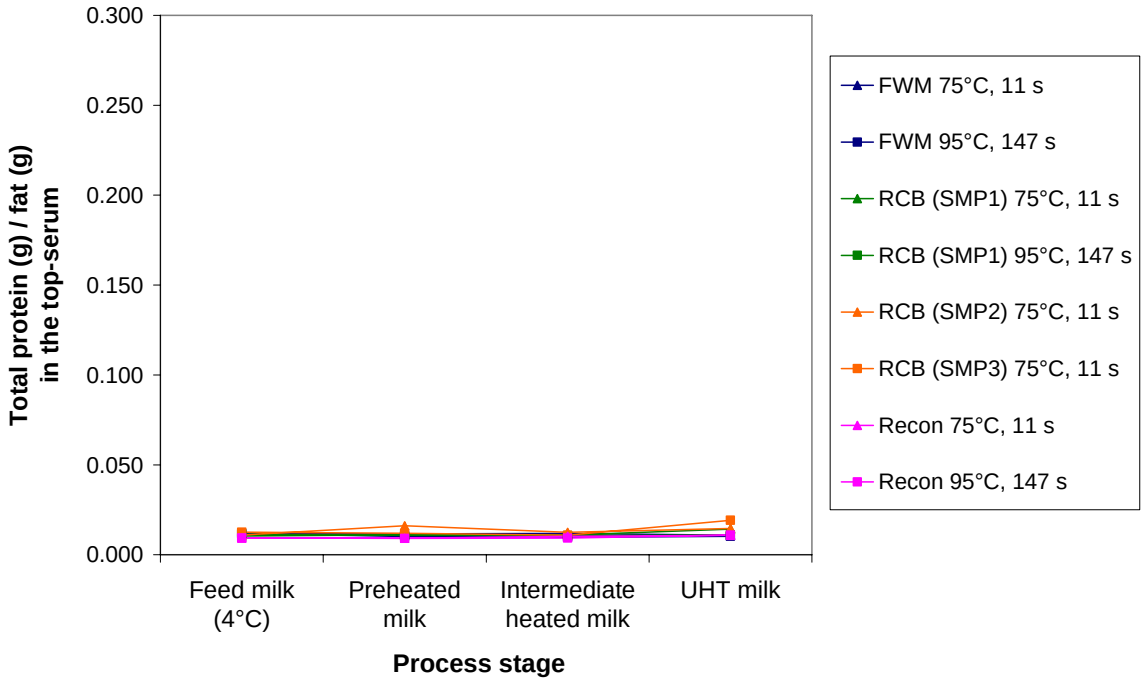
The method for determining the amount and composition of the fat-bound protein were investigated using the data collected using milk preparations derived from Milk No. 10. Thus, there is no effect of batch-to-batch variation of the milk composition among FWM, RCB and Recon. The resulting estimates of total fat-bound protein in the cream layer as measured by the Kjeldahl nitrogen method are shown in Figure 6.1 (a), (b) and (c).

The level of total fat-bound protein by the Kjeldahl nitrogen method (Figure 6.1 (a)) showed that uncorrected total fat-bound protein in the cream layer increased only very slightly with heating process stage until the final UHT preheat treatment was applied. In Figure 6.1 (b), the level of total fat-bound protein in the top-serum varied slightly with milk preparation. After the top-serum was substracted, the result, in Figure 6.1 (c), showed that the change in total fat-bound protein in the cream layers of FWM, RCB and Recon was small, compared with the total fat-bound protein without substraction in Figure 6.1 (a).

(a)


0.300
0.250
0.200
0.150
0.100
0.050
0.000
Total fat-bound protein / fat (g) without top-serum substracted
Feed milk (4°C)
Preheated milk
Intermediate heated milk
UHT milk
Process stage
FWM 75°C, 11 s
FWM 95°C, 147 s
RCB (SMP1) 75°C, 11 s
RCB (SMP1) 95°C, 147 s
RCB (SMP2) 75°C, 11 s
RCB (SMP3) 75°C, 11 s
Recon 75°C, 11 s
Recon 95°C, 147 s

(b)


0.300
0.250
0.200
0.150
0.100
0.050
0.000
Total protein (g) / fat (g) in the top-serum
Feed milk (4°C)
Preheated milk
Intermediate heated milk
UHT milk
Process stage
FWM 75°C, 11 s
FWM 95°C, 147 s
RCB (SMP1) 75°C, 11 s
RCB (SMP1) 95°C, 147 s
RCB (SMP2) 75°C, 11 s
RCB (SMP3) 75°C, 11 s
Recon 75°C, 11 s
Recon 95°C, 147 s

(c)

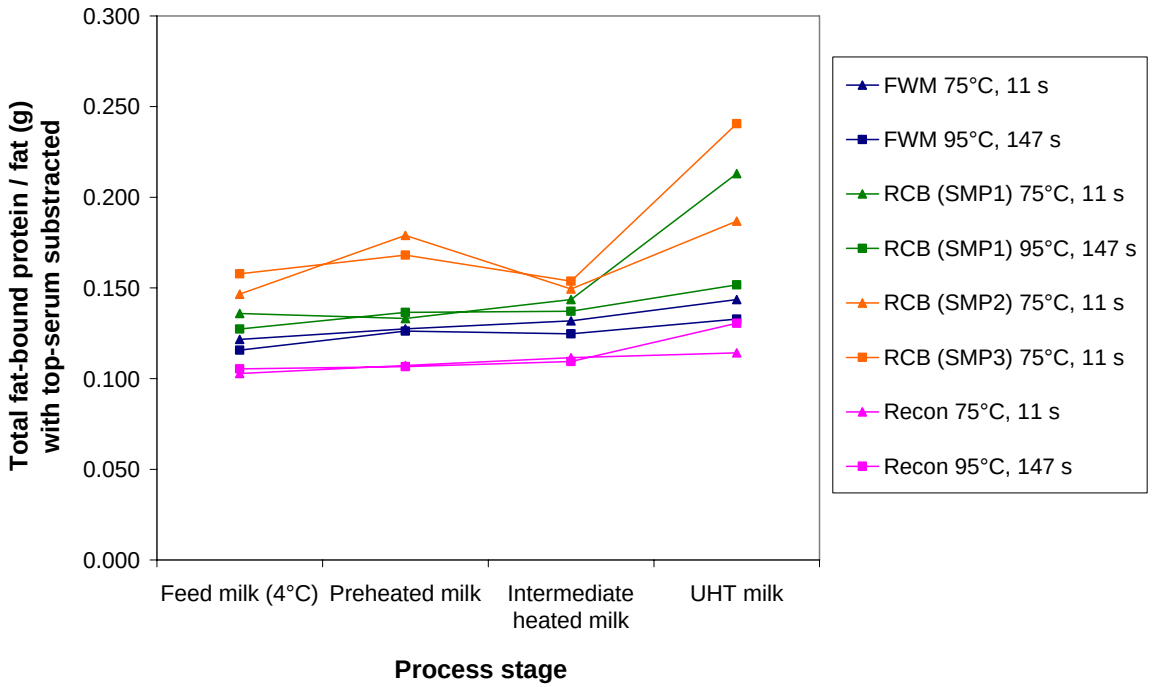


Figure 6.1 Total fat-bound protein / fat (g) in the cream layers of homogenized then preheated FWM, RCB and Recon by the Kjeldahl nitrogen method (UHT preheat treatments 75 °C, 11 s and 95 °C, 147 s, Milk No. 10).

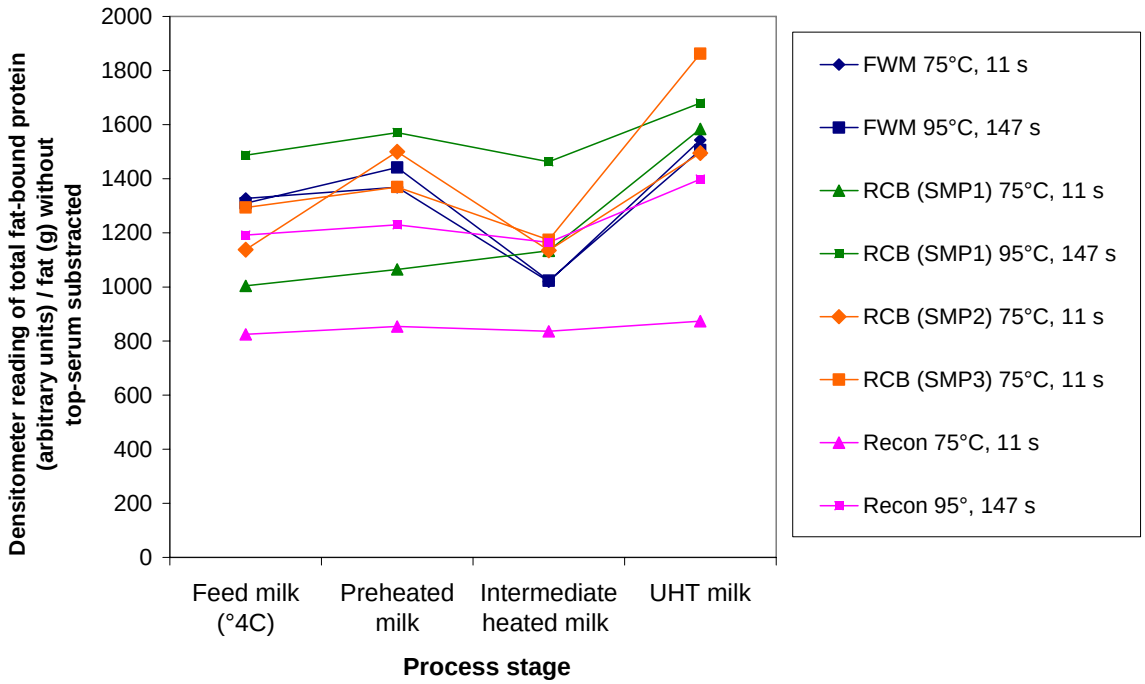
(a) total fat-bound protein / fat (g) without top-serum substracted

(b) total protein / fat (g) in top-serum

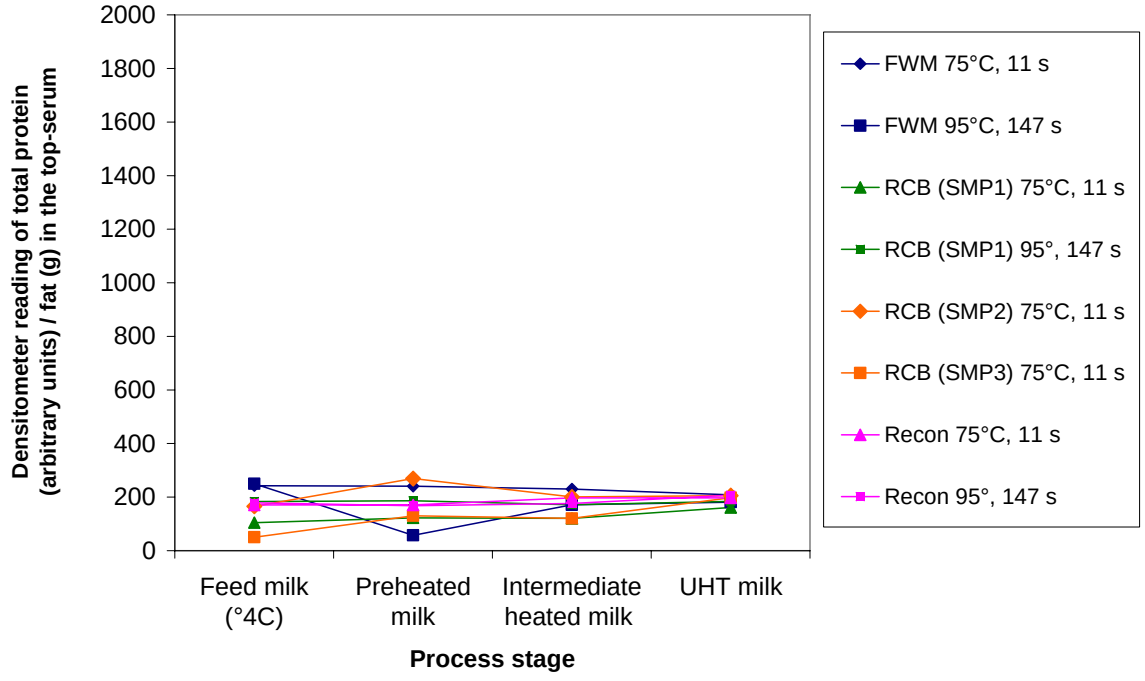
(c) total fat-bound protein / fat (g) with top-serum substracted

The results of total fat-bound individual protein / fat (g) by PAGE analysis (the sum of all component proteins) with the same cream layers measured by the PAGE analysis are reported in Figure 6.2 (a), (b) and (c).

(a)


Densitometer reading of total fat-bound protein (arbitrary units) / fat (g) without top-serum substracted
2000
1800
1600
1400
1200
1000
800
600
400
200
0
Feed milk (°4C)
Preheated milk
Intermediate heated milk
UHT milk
Process stage
FWM 75°C, 11 s
FWM 95°C, 147 s
RCB (SMP1) 75°C, 11 s
RCB (SMP1) 95°C, 147 s
RCB (SMP2) 75°C, 11 s
RCB (SMP3) 75°C, 11 s
Recon 75°C, 11 s
Recon 95°, 147 s

(b)


Densitometer reading of total protein (arbitrary units) / fat (g) in the top-serum
2000
1800
1600
1400
1200
1000
800
600
400
200
0
Feed milk (°4C)
Preheated milk
Intermediate heated milk
UHT milk
Process stage
FWM 75°C, 11 s
FWM 95°C, 147 s
RCB (SMP1) 75°C, 11 s
RCB (SMP1) 95°, 147 s
RCB (SMP2) 75°C, 11 s
RCB (SMP3) 75°C, 11 s
Recon 75°C, 11 s
Recon 95°, 147 s

(c)

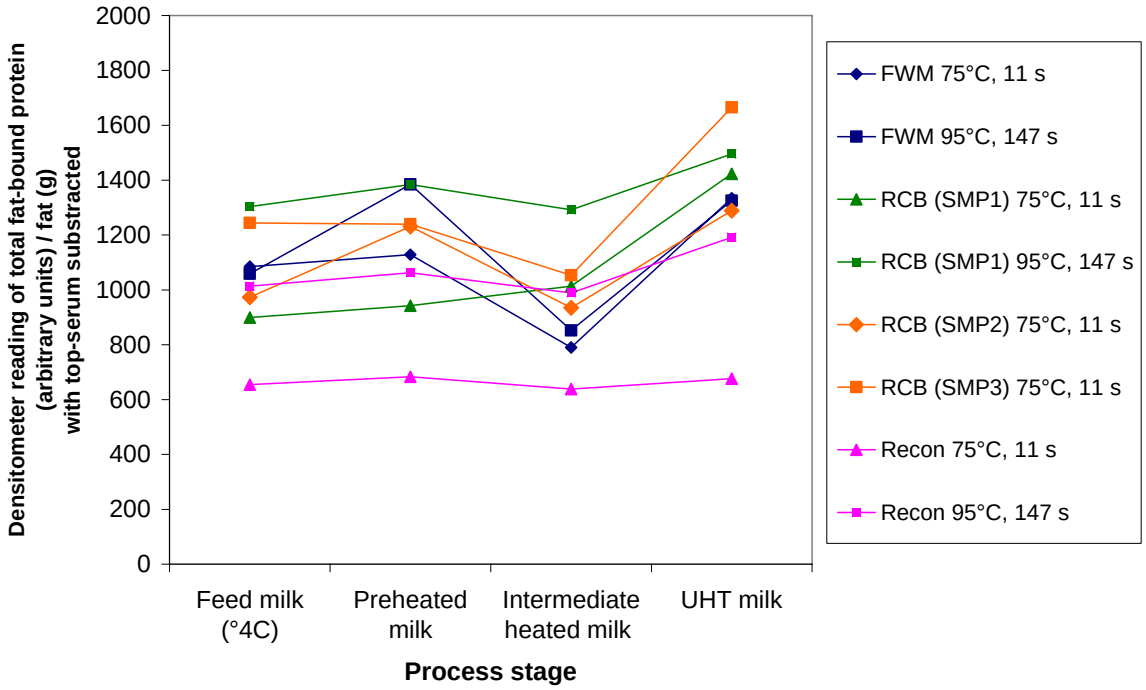


Figure 6.2 Total fat-bound protein in the cream layers of homogenized then preheated FWM, RCB and Recon by PAGE analysis (UHT preheat treatments 75 °C, 11 s and 95 °C, 147 s, Milk No. 10).

(a) Densitometer reading of total fat-bound protein / fat (g) without top-serum substracted

(b) Densitometer reading of total protein / fat (g) in the top-serum

(c) Densitometer reading of total fat-bound protein / fat (g) with top-serum substracted

The total protein in the top-serum as shown in Figure 6.2 (b) was, generally, much less than a quarter of the total protein in the same cream layer and varied slightly with the milk preparation and UHT preheat treatments. After it was substracted from the total fat-bound protein in the cream layers in Figure 6.2 (a), the results showed (Figure 6.2 (c)) that total fat-bound protein by PAGE analysis only slightly changed.

The total protein in the top-serum of FWM, RCB and Recon in Figure 6.2 (a) and (c) showed sharp minima in the data for all milk preparations during intermediate temperature heating. The most probable cause of this variation was the difficulty of “clean” recovery of the soft cream layers from intermediate heated milk, which these cream layers are in both

phases of liquid and semi-phases solid. Accepting these preparation problems, if these minima are ignored, the total fat-bound protein in FWM, RCB (prepared from SMP1, SMP2 and SMP3) and Recon in Figure 6.1 and 6.2 showed a slightly increasing trend with heating process stage for both UHT preheat treatments. The higher UHT preheat treatment gave the greater total fat-bound protein in FWM, RCB (prepared from SMP1 only) and Recon.

These results (Figure 6.1 and 6.2) represent the variation of total fat-bound protein in the cream layer with heating process stage and preheating intensity. As the trends shown in Figures 6.1 (c) and 6.2 (c) are essentially the same as those evident in Figures 6.1 (a) and 6.2 (a), it was decided in ongoing work to assume that the measured total protein content and the measured individual protein contents of cream layers with no correction for top-serum would adequately represent the process-induced variations in total and individual fat-bound proteins in the cream layers for all whole milk preparations investigated.

6.2.2 Mass balances between the fat and protein in the cream layer, top-serum, serum and sediment fractions of whole milk and the measured fat and protein contents of the milk sample

After centrifugation of total 288 g milk, the cream layer, top-serum, serum and sediment was separated as described in Chapter 3. The sums of the weights of fat and protein in these fractions were compared with the weights of fat and protein measured in 288 g FWM, RCB and Recon (before centrifugation). The original percentages of fat and protein in each fraction for FWM, RCB and Recon are shown in Appendix 3 (Table A3.1). The result is reported in Table 6.1.

Table 6.1 shows that there were some discrepancies between the sums of the total weights of fat and protein in the cream layer, top-serum, serum and sediment and the measured weights of fat and protein in homogenized then preheated FWM, RCB and Recon. These discrepancies of up to ± 15 % were probably due to difficulties in cleanly removing the cream layers from centrifuge tubes.

Table 6.1 Comparison between sums of the weights of fat and protein in the cream layer, top-serum, serum and sediment and the measured weights of fat and protein in 288 g of homogenized then preheated FWM, RCB and Recon (Milk No. 10).

Heating process stage	Milk, UHT preheat treatment	Cream layer		Top-serum		Serum		Sediment		Total weight		Liquid milk		Differences	
		Fat (g)	Protein (g)	Fat (g)	Protein (g)	Fat (g)	Protein (g)	Fat (g)	Protein (g)	Fat (g)	Protein (g)	Fat (g)	Protein (g)	Fat (g)	Protein (g)
Past. & homog. FWM	FWM, 75 °C, 11 s	6.36	0.85	0.09	0.21	3.39	6.10	0.49	3.41	10.34	10.57	9.82	9.85	0.52	0.72
UHT FWM		5.83	0.90	0.08	0.19	3.39	5.90	0.70	3.18	10.00	10.16	9.82	9.90	0.18	0.26
Past. & homog. FWM	FWM, 95 °C, 147 s	6.51	0.83	0.10	0.20	3.15	5.95	0.42	3.26	10.18	10.24	9.73	9.78	0.45	0.46
UHT FWM		6.14	0.88	0.08	0.18	3.30	5.42	0.80	3.40	10.32	9.87	9.71	9.74	0.62	0.13
RCB	RCB (SMP1), 75 °C, 11 s	6.45	0.94	0.06	0.18	2.88	4.31	0.74	4.41	10.13	9.84	9.71	9.65	0.42	0.19
UHT RCB		4.79	1.09	0.05	0.19	1.95	3.29	1.42	5.61	8.20	10.18	9.79	9.72	-1.59	0.46
RCB	RCB (SMP1), 95 °C, 147 s	6.43	0.89	0.05	0.17	2.63	4.38	0.62	4.02	9.73	9.47	9.36	9.17	0.37	0.30
UHT RCB		6.33	1.03	0.04	0.17	2.33	3.64	0.84	5.00	9.54	9.83	9.36	9.19	0.18	0.64
RCB	RCB (SMP2), 75 °C, 11 s	6.18	0.97	0.06	0.16	3.13	4.49	0.62	4.25	10.00	9.87	9.59	9.70	0.41	0.17
UHT RCB		5.37	1.08	0.05	0.19	1.65	4.76	0.94	5.34	8.01	11.37	9.71	9.78	-1.70	1.59
RCB	RCB (SMP3), 75 °C, 11 s	5.33	0.91	0.08	0.16	3.40	4.51	1.25	5.09	10.06	10.67	9.73	9.70	0.33	0.97
UHT RCB		5.14	1.34	0.05	0.19	1.66	3.63	2.73	5.91	9.59	11.07	9.71	9.66	-0.12	1.40
Recon.	Recon, 75 °C, 11 s	6.41	0.72	0.10	0.16	3.17	4.70	0.46	4.71	10.14	10.28	9.53	9.61	0.61	0.67
UHT Recon.		6.45	0.81	0.07	0.18	3.11	4.55	0.46	4.48	10.09	10.02	9.62	9.61	0.47	0.41
Recon.	Recon, 95 °C, 147 s	6.46	0.74	0.11	0.15	3.40	5.20	0.43	4.15	10.40	10.24	9.82	9.63	0.58	0.62
UHT Recon.		5.98	0.85	0.09	0.18	3.40	4.11	0.79	4.63	10.27	9.76	9.76	9.65	0.50	0.12

Note:
- Total weight of fat (g) = Sum of the weight of fat in the cream layer, top-serum, serum and sediment.
- Total weight of protein (g) = Sum of the weight of protein in the cream layer, top-serum, serum and sediment.
- Differences of the weight of fat (g) = Total weight of fat (g) – the weight of fat in liquid milk (g).
- Differences of the weight of protein (g) = Total weight of protein (g) – the weight of protein in liquid milk (g).

6.2.3 The proportion of total fat recovered in the cream layers of FWM, RCB and Recon on centrifugation

After centrifugation, not all of the fat in the milk separated into the cream layer. The range of recovered fat contents in Table 6.2 represent the low and high values of fat content for each milk preparation, regardless heating process stage. The full results for the proportion of total fat recovered in the cream layer for every milk number and milk preparation is shown in Appendix 3 (Tables A3.2-A3.6).

Table 6.2 Percentage concentration of fat and proportion of fat recovered in the cream layers of homogenized then preheated FWM, RCB and Recon (Milk Nos. 7-10 and 2b).

Milk	Evap. preheat	UHT preheat treatment	% concentration of fat in cream layer as recovered		% proportion of total fat recovered in the cream layer	
			Range		Range	
			Low	High	Low	High
FWM	n/a	75 °C, 11 s	44.10 (Milk No. 10)	47.20 (Milk No. 10)	52	55
		95 °C, 147 s	42.90 (Milk No. 7)	48.80 (Milk No. 9)	54	57
RCB	75 °C, 2 s	75 °C, 11 s	33.30 (Milk No. 8)	46.20 (Milk No. 2b)	44	56
		95 °C, 147 s	35.70 (Milk No. 8)	44.90 (Milk No. 2b)	46	56
	85 °C, 155 s	75 °C, 11 s	30.9 (Milk No. 2b)	41.7 (Milk No. 2b)	42	57
	95 °C, 155 s	75 °C, 11 s	33.4 (Milk No. 2b)	43.4 (Milk No. 2b)	48	50
Recon	95 °C, 33 s	75 °C, 11 s	37.70 (Milk No.8)	46.40 (Milk No. 2b)	45	57
		95 °C, 147 s	43.90 (Milk No. 8)	47.60 (Milk No. 2b)	54	58

Table 6.2 shows that the general range of the fat recovered in the cream layers of FWM, RCB and Recon represented 42-58% of the total fat in the milk. The fat globules or globule clusters in these cream layers would have had the largest sizes in the original milk sample. The smaller fat globules would have been covered by a greater amount of protein per g of fat, because of their smaller size and because smaller fat globules in milk have thicker membranes. Smaller globules would therefore have been denser than the larger ones and thus harder to recover into the cream layer. The estimated fractions of total surface area and volume of the fat globules recovered in the cream layer and unrecovered in the top-serum, serum and sediment are reported in the next section.

6.2.4 Estimation of the fraction of total surface area and the fraction of total volume represented by the recovered fat globules (cream layer) and the unrecovered fat globules (top-serum, serum and sediment) of homogenized then preheated FWM, RCB and Recon

The unrecovered fat in the top-serum, serum and sediment was estimated by substracting the fat recovered in the cream layer (section 6.2.1) from the total fat. This information, together with the particle size distribution, was used to plot the relationship between the cumulative fraction of total fat globule surface area and the cumulative fraction of total fat globule volume as the globule size increased. The diameter of the fat globule size was also included the membrane covering the surface of fat globules. The estimation of the fraction of fat-bound protein in the recovered cream layer and unrecovered top-serum, serum and sediment required an assumption that the loading of fat-bound protein per unit surface area was the same for all globule sizes[1]. This assumption implies further that the fat globules in the recovered cream layer were all of a size greater than the critical "zero-buoyancy" size, for which the amount of the, relatively dense, protein on the surface just balanced the tendency of the less-dense fat to float during centrifugation. This procedure was carried out for 3 separate particle-size distributions covering the typical range observed in the three whole milk samples under investigation. The calculation of the estimation of volume and surface area for different sizes of fat globules (ranged from 0.04 to 2.24 μm) is shown in Appendix 3 (beneath Table A3.7). The plot between the fraction of surface area and the fraction of volume for different sizes of fat globules is reported in Figure 6.3.

Figure 6.3 shows that the relationship between the fractions of total fat globule surface area and total fat globule volume were very little different among the limited range of globule size distributions observed for the original FWM, RCB and Recon. From Table 6.2, about 42-58 % of total fat was recovered in the cream layers of FWM, RCB and Recon (representing the larger fat globules) and this corresponded to the range 0.25-0.12 total area.

[1] This is not the case for the greater protein load (mg/m^2 fat) on the smaller fat globules for FWM (Cano-Ruiz & Richter, 1997) and for RCB (Sharma, 1993).

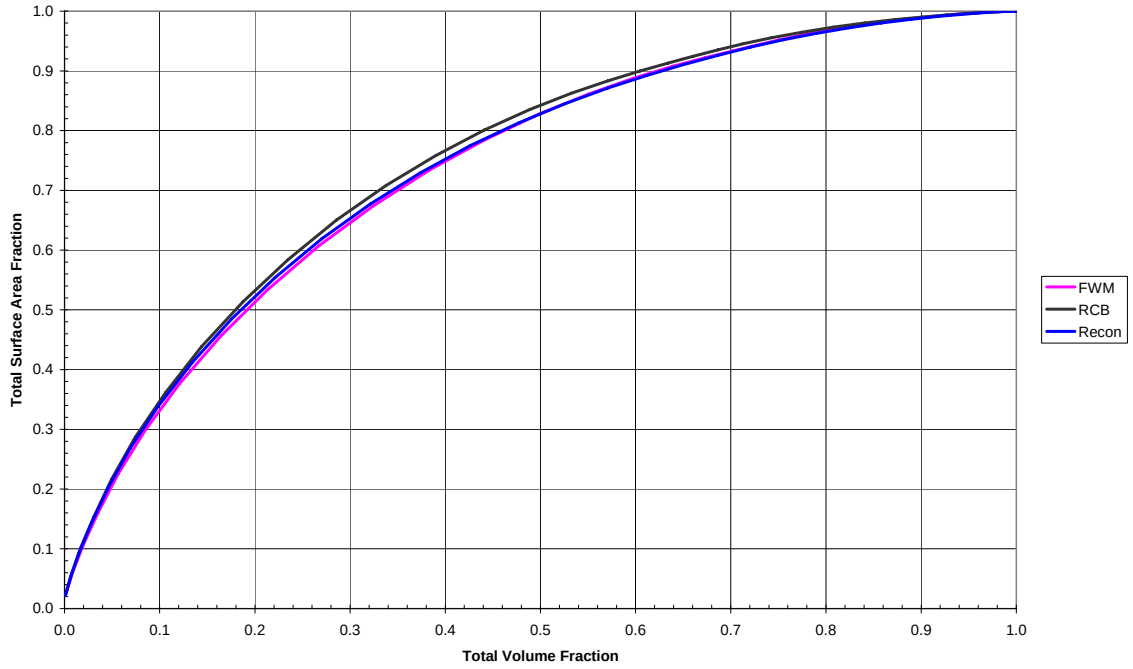


Figure 6.3 Cumulative fraction of total fat globule surface area versus cumulative fraction of total volume for ascending fat-globule size classes of 0.04 to 2.24 μm diameter of homogenized then preheated FWM, RCB and Recon (Milk No. 2).

To demonstrate the estimation of the proportion of fat-bound protein in the unrecovered portion, three "critical" sizes, regardless of milk preparation (they were derived from the proportion of fat recovered in the cream layer and the data on particle size distribution for FWM, RCB and Recon) were selected from the data underlying Figure 6.3. The process of this estimation is explained in Appendix 3 (Table A3.7). The estimated total fat-bound protein in milk can be calculated as follows :

$$\text{Estimated total fat-bound protein in milk} = \frac{\text{Volume (or mass) fraction of fat in the cream layer} \times \text{total fat-bound protein in the cream layer (g / g fat)}}{\text{Corresponding fraction of surface area of recovered fat globules (derived from Fig. 6.3 relationship)}}$$

Eq. (6.1)

The result of this estimation is shown in Table 6.3.

Table 6.3 Estimation of total fat-bound protein in milk from total fat-bound protein measured in the cream layers of whole milks (Milk No. 2).

Critical sizes of fat globule (μm)	Proportion of fat recovered in the cream layer		Estimated total fat-bound protein in milk (g) / fat (g)		
	Fraction of volume	Fraction of surface	Total fat-bound protein in the cream layer = 0.10 g/ g fat	Total fat-bound protein in the cream layer = 0.15 g/ g fat	Total fat-bound protein in the cream layer = 0.20 g/ g fat
0.18	0.56	0.20	0.28	0.42	0.56
0.20	0.51	0.16	0.32	0.48	0.64
0.22	0.47	0.15	0.31	0.47	0.63

Table 6.3 shows that for the particle size distributions measured for the homogenized milks under investigation the estimated total fat-bound protein in milk was 3-4 times larger than the estimated total fat-bound protein in the cream layer. With this estimation, it showed that total fat-bound protein in the cream layer of homogenized milks represented ~ 30-35 % of total proteins bound to fat globules in the whole milk system.

Cano-Ruiz & Richter (1997) and Sharma (1993) reported that the smaller fat globules were covered with high amount of protein / surface area. In this study, it was assumed that the amount of protein covering the fat globules / surface area was the same for small or large fat globules.

6.3 Effect of pasteurisation on the total fat-bound protein in the cream layer of FWM

Pasteurised FWM (72 °C, 15 s) was used as the initial FWM for UHT processing in the present study but raw FWM was used by others who have studied the effect of preheating on whole milk fouling: Bell & Sanders (1944); Burton (1968); Patil & Reuter (1986a) and Mottar & Moermans (1988). Thus, milk Nos. 9 and 10 were used to determine the total fat-bound protein in raw and pasteurised FWM with no homogenization. The results are shown in Table 6.4 and the statistical analysis is reported in Appendix 3 (Table A3.8).

Table 6.4 Total fat-bound protein by the Kjeldhal method in pasteurised and raw FWM without homogenization (Milk Nos. 9 and 10).

Milk	Geometric mean total fat-bound protein (g) / fat (g) in the cream layer
Raw FWM, df = 1	0.0100
Pasteurised FWM, df =1	0.0085
p-value	0.219

There may have been a small reduction in the total fat-bound protein owing to pasteurisation.

6.4 Effect of milk preparation on total fat-bound protein and individual fat-bound proteins in homogenized then preheated FWM, RCB and Recon

FWM, RCB and Recon were subjected to UHT preheat treatments 75 °C, 11 s and 95 °C, 147 s. Total fat-bound protein was measured by the Kjeldahl nitrogen method and the individual fat-bound proteins were determined comparatively by PAGE analysis.

The original data on total fat-bound protein and individual fat-bound proteins in FWM, RCB and Recon were transformed to $\log_{10}$ because the residual plots of $\log_{10}$ transformation gave a more uniform balance around the mean than did the untransformed data. As there were some zero values among the PAGE data, making a direct log transformation impossible, the number one was added to the original data for both total fat-bound protein and individual fat-bound proteins before they were transformed to $\log_{10}$. As number of milk batches were used in this study, the “effect” of milk batch (milk number) was included in every statistical analysis to allow for overall effects of differences in milk composition between batches. The split-plot design for this experiment is reported in Figure 6.4.

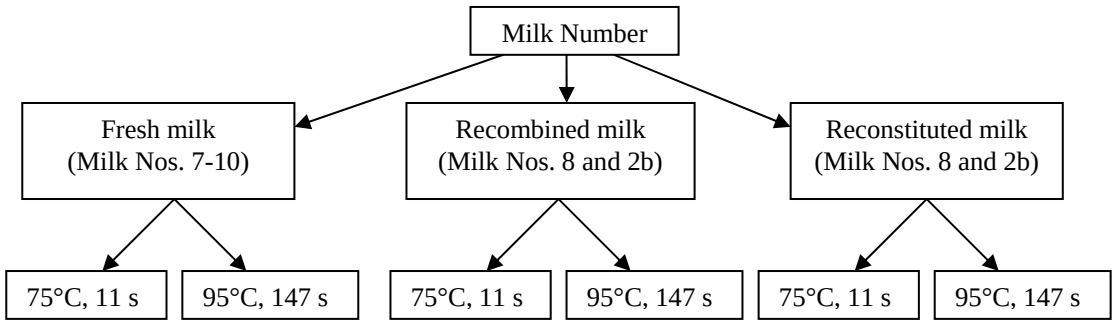


Figure 6.4 Split-plot design with replicates using different batches of milk (Milk Nos. 7-10 and 2b) for the effects of milk preparation and UHT preheat treatment on total fat-bound protein for homogenized then preheated FWM, RCB and Recon.

Fat-bound proteins for Milk No. 8-10 and 2b were analysed according to the split-plot design shown in Figure 6.4 (Potcner & Kowalski, 2004). The main plot for this design is the factor milk preparation (FWM, RCB and Recon). The sub-plot is the factor UHT preheat treatment, which is nested within the factor milk preparation. The geometric means for individual milk preparations of the total fat-bound protein averaged across heating process stages are shown in Table 6.5. The full statistical analysis is reported in the Appendix 3 (Table A3.9).

Table 6.5 The effect of milk preparation and UHT preheat treatment on total fat-bound protein by the Kjeldhal method in homogenized then preheated FWM, RCB and Recon (Milk Nos. 7-10 and 2b), averaged across heating process stages.

Milk preparation	**Geometric mean total fat-bound protein (g) / fat (g) in the cream layer**		
	FWM **(Milk Nos. 7-10)**	**RCB** **(Milk Nos. 8 and 2b)**	**Recon** **(Milk Nos. 8 and 2b)**
UHT preheat treatment at 75 °C, 11 s	0.139	0.167	0.127
UHT preheat treatment at 95 °C, 147 s	0.137	0.158	0.125

Table 6.5 shows that total fat-bound protein varied with milk preparation at both UHT preheat treatments ($p < 0.001$). Total fat-bound protein for FWM and Recon were not much different but total fat-bound protein for RCB was greater.

PAGE analysis of the individual protein fractions in the same cream layers showed significant variation with milk preparation and UHT preheat treatment as shown in Table 6.6, corresponding to the mean values shown in Table 6.7. The raw data was shown on the CD. The full statistical analysis is reported in Appendix 3 (Table A3.10).

Table 6.6 Probalility values (p) for the effects of milk preparation, UHT preheat treatment within milk preparation and milk number on individual fat-bound proteins by PAGE analysis in homogenized then preheated FWM, RCB and Recon (Milk Nos. 7-10 and 2b).

Proteins in the cream layer	**p-values**		
	Milk preparation (df = 2)	**UHT preheat within milk preparation** (df = 3)	**Milk No.** (df = 4)
α_s-casein	0.197	0.847	0.072
β-casein	<0.001	0.001	<0.001
κ-casein	0.836	0.005	<0.001
γ-casein	<0.001	0.004	0.041
Total casein	<0.001	0.001	<0.001
β-lg	0.044	<0.001	0.873
α-la	0.002	<0.001	0.071
Total whey protein	0.026	<0.001	0.721
TOTAL PROTEIN	<0.001	<0.001	0.001

Main factors : Milk preparation (*FWM, RCB and Recon.*), Milk Nos. (*7-10 and 2b*) and UHT preheat treatment (*75 °C, 11 s and 95 °C, 147 s*).

Table 6.7 The effects of milk preparation and UHT preheat treatment on total fat-bound protein and individual fat-bound proteins by PAGE analysis in the cream layers of homogenized then preheated FWM, RCB and Recon (Milk Nos. 7-10 and 2b).

Protein in the cream layer	UHT preheat treatment	Geometric mean densitometer reading (arbitrary units)		
		FWM (Milk Nos. 7-10)	**RCB** (Milk No. 10)	**Recon** (Milk No. 10)
α_s-casein	75 °C, 11 s	406	267	217
	95 °C, 147 s	429	271	222
β-casein	75 °C, 11 s	470	256	200
	95 °C, 147 s	422	259	205
κ-casein	75 °C, 11 s	130	63	63
	95 °C, 147 s	129	61	63
γ-casein	75 °C, 11 s	12	6	3
	95 °C, 147 s	13	5	3
Total casein	75 °C, 11 s	1118	593	484
	95 °C, 147 s	1070	597	494
β-lg	75 °C, 11 s	108	29	68
	95 °C, 147 s	166	54	77
α-la	75 °C, 11 s	23	5	17
	95 °C, 147 s	39	9	20
Total whey protein	75 °C, 11 s	132	35	84
	95 °C, 147 s	206	63	97
TOTAL PROTEIN	75 °C, 11 s	1269	631	569
	95 °C, 147 s	1302	670	592

Table 6.7 shows that total fat-bound protein was dominated by casein, particularly α_s-casein and β-casein, but these two caseins exhibit no trend with either UHT preheat treatment. However, both fat-bound total whey protein and fat-bound total casein varied with milk preparation. When the milk was preheated at the more severe UHT preheat

treatment, fat-bound total whey protein became greater for all milk preparations and increases in fat-bound total whey protein was larger for FWM than for RCB and Recon.

6.5 Effect of heating process stage on total fat-bound protein and individual fat-bound proteins in homogenized then preheated FWM, RCB and Recon

The effect of heating process stage on total fat-bound protein as determined by the Kjeldhal nitrogen method, and the individual fat-bound proteins by PAGE analysis in FWM, RCB and Recon at the UHT preheat conditions 75 °C, 11 s and 95 °C, 147 s are reported in this section by individual milk preparation.

6.5.1 Homogenized then preheated FWM

For each milk number, the effect of heating process stage was nested within UHT preheat treatment. Process diagram of this experimental design is shown in Figure 6.5.

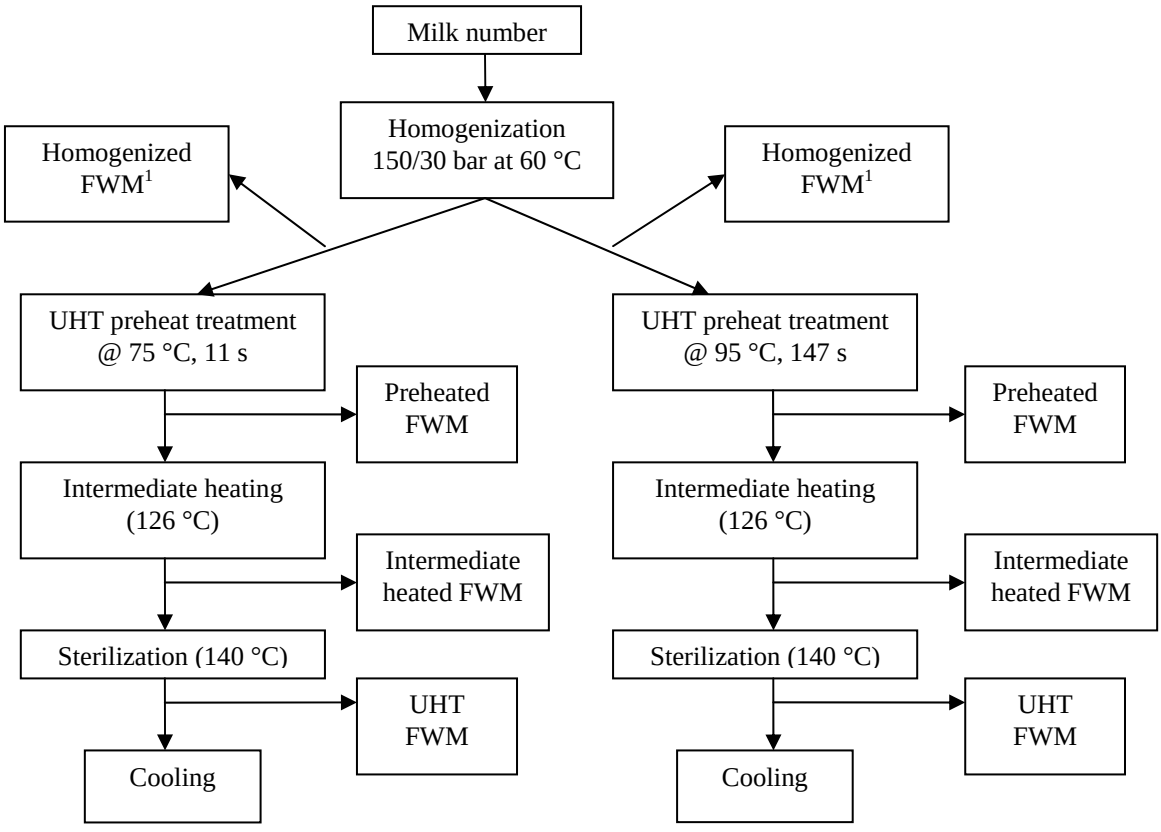


Figure 6.5 Split-plot design with replicates using different batches of milk (Milk Nos. 7-10) for the effect of heating process stage on total fat-bound protein for homogenized then preheated FWM.

[1]Homogenized FWM prior to preheat treatment.

In Figure 6.5, the main plot is the factor UHT preheat treatment, 75 °C, 11 s and 95 °C, 147 s. The sub-plot is the factor heating process stage, which it is nested within the factor UHT preheat treatment.

The geometric means of total fat-bound protein at the four different heating process stages (prior to preheating, after preheating, after intermediate heating and after UHT sterilization and cooling) are shown in Table 6.8.

Table 6.8 The effects of heating process stage on total fat-bound protein by the Kjeldhal method in the feed, preheated, intermediate heated and UHT sterilized and cooled homogenized then preheated FWM (Milk Nos. 7-10). Geometric mean fouling rates corresponding to the UHT preheat conditions and milk number shown are included.

Heating process stage	Geometric mean total fat-bound protein (g) / fat (g) in the cream layer	
	75 °C, 11 s **(Milk Nos. 7, 8 and 10)**	**95 °C, 147 s** **(Milk Nos. 7-10)**
Homog. FWM[1]	0.132	0.129[1]
Preheated FWM (75 or 95 °C)	0.134	0.136
Intermediate heated FWM (126 °C)	0.142	0.137
UHT sterilized FWM (140 °C)	0.148	0.146
Geometric mean fouling rate (°C/h)	0.07	0.34

[1]Homog. FWM prior to preheat treatment.

The statistical analysis results showed that total fat-bound protein increased with heating process stage ($p < 0.001$, Table A3.11). Total fat-bound protein increased during intermediate temperature heating when FWM was preheated at 75 °C, 11 s. When FWM was preheated at 95 °C, 147 s, total fat-bound protein showed a large increase during preheating. The ultimate extent of these increases after the final UHT preheat treatment are about equivalent.

When similar cream layers were analysed by PAGE, the statistical results summarised in Table 6.9 were obtained. The full statistical analysis is reported in Appendix 3 (Table A3.12).

Table 6.9 Probability (p-value) for the effects of heating process stage within UHT preheat treatment on total fat-bound protein and individual fat-bound proteins by PAGE analysis in homogenized then preheated FWM (Milk Nos. 7-10).

Proteins in the cream layer	**p-value**		
	Heating process stage within UHT preheat (df = 6)	**UHT preheat** (df = 1)	**Milk No.** (df = 3)
α_s-casein	0.070	0.995	0.274
β-casein	0.037	0.778	0.002
κ-casein	< 0.001	0.063	< 0.001
γ-casein	< 0.001	0.106	< 0.001
Total casein	0.155	0.661	0.191
β-lg	< 0.001	< 0.001	0.002
α-la	< 0.001	< 0.001	< 0.001
Total whey protein	< 0.001	< 0.001	< 0.001
TOTAL PROTEIN	0.024	0.473	0.183

Main factors : Milk Nos. (*7-10 and 2b*), heating process stage (*Homogenized and pasteurised FWM, homogenized then preheated FWM, intermediate heated FWM and UHT sterilized and cooled FWM*) and UHT preheat treatment (*75 °C, 11 s and 95 °C, 147 s*).

The geometric means of the comparative changes through the process steps as determined by PAGE analysis are shown in Table 6.10.

Table 6.10 shows that fat-bound total whey protein, fat-bound β-lg and fat-bound α-la increased markedly with heating process stage ($p < 0.001$). The amounts of fat-bound total whey protein, β-lg and α-la were higher on and after preheating when preheating was more severe (95 °C, 147 s).

Table 6.10 The effects of heating process stage on total fat-bound protein and individual fat-bound proteins by PAGE analysis in the feed, preheated, intermediate heated and UHT sterilized and cooled homogenized then preheated FWM. (Milk Nos. 7, 8 and 10 were used for the 75 °C, 11 s UHT preheat treatment and Milk Nos. 7-10 were used for the 95 °C, 147 s UHT preheat treatment).

Proteins in the cream layer	UHT preheat treatment	Geometric mean densitometer reading (arbitrary units)			
		Homog. FWM[1]	Preheated FWM (75 or 95 °C)	Intermediate heated FWM (126 °C)	UHT FWM (140 °C)
α_s-casein	75 °C, 11 s	534	538	165	573
	95 °C, 147 s	519	508	225	569
β-casein	75 °C, 11 s	447	436	525	479
	95 °C, 147 s	359	425	463	450
κ-casein	75 °C, 11 s	146	144	120	113
	95 °C, 147 s	150	119	122	126
γ-casein	75 °C, 11 s	20	16	8	8
	95 °C, 147 s	25	9	10	12
Total casein	75 °C, 11 s	1154	1141	1006	1181
	95 °C, 147 s	1075	1070	980	1165
β-lg	75 °C, 11 s	61	77	172	171
	95 °C, 147 s	62	230	229	231
α-la	75 °C, 11 s	13	14	34	47
	95 °C, 147 s	17	45	51	58
Total whey protein	75 °C, 11 s	75	90	207	219
	95 °C, 147 s	80	276	280	290
TOTAL PROTEIN	75 °C, 11 s	1230	1234	1220	1399
	95 °C, 147 s	1157	1347	1266	1456

[1]Homog. FWM prior to preheat treatment.

The similar trends among fat-bound β-lg, α-la and total whey protein markedly increased during intermediate heating and remained constant during sterilization when FWM was preheated at 75 °C, 11 s. At the preheat treatment of 95 °C, 147 s, they increased considerably during preheating and remained essentially constant during intermediate heating and sterilization.

Fat-bound κ-casein showed a decreasing trend with heating process stage. A considerable decrease took place during intermediate heating when FWM was preheated at 75 °C, 11 s and during preheating when FWM was preheated at 95 °C, 147 s. However, the overall decrease in fat-bound κ-casein at the 95 °C, 147 s UHT preheat treatment was similar to that in fat-bound κ-casein at the 75 °C, 11 s UHT preheat treatment.

6.5.2 Recombined whole milk

The experimental design shown in Figure 6.4 was applied to the effect of heating process stage on total fat-bound protein in RCB (Milk No. 8). The geometric means of total fat-bound protein measured by the Kjeldhal method are shown in Table 6.11.

Table 6.11 shows that total fat-bound protein increased from the feed RCB to UHT sterilized RCB for both UHT preheat treatments. Total fat-bound protein markedly increased during intermediate temperature heating when RCB was preheated at both the 75 °C, 11 s and 95 °C, 147 s UHT preheat treatments.

However, total fat-bound protein for intermediate heated RCB and UHT sterilized RCB at the 95 °C, 147 s UHT preheat treatment was similar to that at the 75 °C, 11 s UHT preheat treatment.

Table 6.11 The effect of heating process stage on total fat-bound protein by the Kjeldhal method in the feed, preheated, intermediate heated and UHT sterilized and cooled RCB (prepared using the evaporator preheat treatment 75 °C, 2 s) (Milk No. 8).

Heating process stage	Total fat-bound protein (g) / (g) fat in the cream layer	
	UHT preheat treatment 75 °C, 11 s	**UHT preheat treatment 95 °C, 147 s**
RCB[1]	0.141	0.148[1]
Preheated RCB (75 or 95 °C)	0.159	0.162
Intermediate heated RCB (126 °C)	0.199	0.195
UHT sterilized RCB (140 °C)	0.179	0.173
Fouling rate (°C/h)	0.00	0.29

[1]RCB prior to preheat treatment.

The results of the measurement of total and individual fat-bound proteins in the cream layer of Milk No. 8 is reported in Table 6.12.

Table 6.12 shows that fat-bound total whey protein strongly increased with heating process stage up to and including intermediate heating. The increase at the 95 °C, 147 s UHT preheat treatment was nearly double that the 75 °C, 11 s UHT preheat treatment.

Fat-bound β-lg, fat-bound α-la and fat-bound total whey protein markedly increased on intermediate heating and remained essentially constant during sterilization when RCB was preheated at 75 °C, 11 s. At the preheat treatment 95 °C, 147 s, these increased considerably during preheating and showed relatively little change after preheating. This trend was similar to that shown by FWM (Table 6.10).

Table 6.12 The effect of heating process stage on total fat-bound protein and individual fat-bound proteins by PAGE analysis in the feed, preheated, intermediate heated and UHT sterilized and cooled RCB (Milk No. 8).

Proteins in the cream layer	UHT preheat treatment	Densitometer reading (arbitrary units)			
		RCB[1]	Preheated RCB (75 or 95 °C)	Intermediate heated RCB (126 °C)	UHT RCB (140 °C)
α_s-casein	75 °C, 11 s	604	626	832	696
	95 °C, 147 s	587	608	793	654
β-casein	75 °C, 11 s	567	575	799	712
	95 °C, 147 s	582	572	728	641
κ-casein	75 °C, 11 s	178	174	162	135
	95 °C, 147 s	169	135	160	131
γ-casein	75 °C, 11 s	21	17	12	13
	95 °C, 147 s	20	10	11	12
Total casein	75 °C, 11 s	1370	1391	1805	1554
	95 °C, 147 s	1357	1325	1692	1438
β-lg	75 °C, 11 s	37	49	147	122
	95 °C, 147 s	38	188	230	180
α-la	75 °C, 11 s	9	5	21	28
	95 °C, 147 s	9	19	34	32
Total whey protein	75 °C, 11 s	46	54	168	151
	95 °C, 147 s	47	207	263	212
TOTAL PROTEIN	75 °C, 11 s	1417	1445	1973	1704
	95 °C, 147 s	1404	1532	1955	1650

[1]RCB prior to preheat treatment.

Fat-bound κ-casein showed a decreasing trend overall with heating process stage. This trend was similar to the trend shown by FWM. The decrease in fat-bound κ-casein in RCB at the 95 °C, 147 s UHT preheat treatment was similar to that at the 75 °C, 11 s UHT preheat treatment.

6.5.3 Reconsituted whole milk

The experimental design shown in Figure 6.4 was applied to Recon (Milk No. 8). Data on total fat-bound protein by the Kjeldhal nitrogen method are reported in Table 6.13.

Table 6.13 The effects of UHT preheat treatment and heating process stage on total fat-bound protein by the Kjeldhal nitrogen method in the feed, preheated, intermediate heated and UHT sterilized and cooled Recon (Milk No. 8).

UHT preheat treatment	Total fat-bound protein (g) / fat (g) in the cream layer	
	75 °C, 11 s	**95 °C, 147 s**
Recon[1]	0.122	0.120
Preheated Recon (75 or 95 °C)	0.125	0.128
Intermediate heated Recon (126 °C)	0.159	0.120
UHT sterilized Recon (140 °C)	0.138	0.141
Fouling rate (°C/h)	0.0011	0.33

[1]Recon prior to preheat treatment.

Table 6.13 shows that total fat-bound protein increased with heating process stage for both low and high preheat treatment. The increase in total fat-bound protein at the UHT preheat treatment 95 °C, 147 s was slightly greater than the increase at the UHT preheat treatment 75 °C, 11 s.

There was an increase in total fat-bound protein during intermediate heating at the 75 °C, 11 s UHT preheat treatment whereas there was a small increase during preheating at the 95 °C, 147 s UHT preheat treatment.

The results of the measurement of total and individual fat-bound proteins in the cream layer of Milk No. 8 is reported in Table 6.14.

Table 6.14 The effects of UHT preheat treatment and heating process stage on total fat-bound protein and individual fat-bound proteins by PAGE analysis in the feed, preheated, intermediate heated and UHT sterilized and cooled Recon (Milk No. 8)

Proteins in the cream layer	UHT preheat treatment	Densitometer reading (arbitrary units)			
		Recon[1]	Preheated Recon (75 or 95 °C)	Intermediate heated Recon (126 °C)	UHT Recon (140 °C)
α_s-casein	75 °C, 11 s	415	456	626	540
	95 °C, 147 s	462	458	501	581
β-casein	75 °C, 11 s	404	422	552	486
	95 °C, 147 s	439	408	476	518
κ-casein	75 °C, 11 s	143	151	159	135
	95 °C, 147 s	159	134	137	136
γ-casein	75 °C, 11 s	7	9	5	9
	95 °C, 147 s	7	7	7	11
Total casein	75 °C, 11 s	969	1038	1342	1168
	95 °C, 147 s	1067	1007	1121	1246
β-lg	75 °C, 11 s	135	146	194	157
	95 °C, 147 s	148	191	182	173
α-la	75 °C, 11 s	33	36	40	48
	95 °C, 147 s	38	46	46	50
Total whey protein	75 °C, 11 s	168	182	233	205
	95 °C, 147 s	186	237	228	222
TOTAL PROTEIN	75 °C, 11 s	1137	1220	1576	1373
	95 °C, 147 s	1253	1244	1349	1469

[1]Recon prior to preheat treatment.

Fat-bound total whey protein (Table 6.14) increased with heating process stage up to and including intermediate heating when Recon was preheated both 75 °C, 11 s and

95 °C, 147 s. The increases at the 95 °C, 147 s UHT preheat treatment were about equivalent to those at the 75 °C, 11 s UHT preheat treatment.

Fat-bound β-lg, fat-bound α-la and fat-bound total whey protein increased markedly during intermediate heating and slightly decreased during sterilization (except for fat-bound α-la) when Recon was preheated at 75 °C, 11 s. At the 95 °C, 147 s UHT preheat treatment, fat-bound β-lg, fat-bound α-la and fat-bound total whey protein increased considerably during preheating and slightly decreased during intermediate temperature heating and sterilization.

Fat-bound κ-casein showed a decreasing overall trend with heating process stage for both UHT preheat treatments. The decrease in fat-bound κ-casein at the 95 °C, 147 s UHT preheat treatment was greater than that at the 75 °C, 11 s UHT preheat treatment.

At this point, it could be concluded that the pattern of fat-bound β-lg, fat-bound α-la and fat-bound total whey protein increasing with heating process stage was similar for FWM, RCB and Recon. At the same time, fat-bound κ-casein in FWM, RCB and Recon showed the pattern of decreasing with heating process stage, which matched the pattern of increasing fat-bound total whey protein.

6.6 Effect of evaporator preheat treatment on total fat-bound protein and individual fat-bound proteins in RCB

Three evaporator preheat treatments (75 °C, 2 s, 85 °C, 155 s and 95 °C, 155 s) were applied to skim milk before evaporation in making SMPs and three UHT preheat treatments (75 °C, 11 s, 85 °C, 147 s and 95 °C, 147 s) were applied to RCB (made from SMPs) prior to UHT processing. The experiment was not fully replicated for the evaporator preheat treatments 85 °C, 155 s and 95 °C, 155 s because investigating the effect of evaporator preheat treatment (as opposed to UHT preheat treatment) was not a main objective in the present work. The results for total fat-bound protein by the Kjeldhal method are shown in Table 6.15.

Table 6.15 The effects of evaporator preheat treatment (at the constant UHT preheat treatment of 75 °C, 11 s) and heating process stage on total fat-bound protein by the Kjeldhal method in RCB (Milk Nos. 8 and 2b).

Evaporator preheat treatment	Milk No.	UHT preheat treatment	Total fat-bound protein (g) / fat (g) in the cream layer[1]			
			RCB[2]	Preheated RCB (75 °C)	Intermediate heated RCB (126 °C)	UHT RCB (140 °C)
75 °C, 2 s	8	75 °C, 11 s	0.141	0.159	0.199	0.179
	2b	75 °C, 11 s	0.146	0.144	0.155	0.227
85 °C, 155 s	2b	75 °C, 11 s	0.158	0.195	0.162	0.201
95 °C, 155 s	2b	75 °C, 11 s	0.170	0.180	0.165	0.260

[1]The values are individual values.

[2]RCB prior to UHT preheat treatment.

Total fat-bound protein (Table 6.15) showed a generally increasing trend with heating process stage for every evaporator preheat treatment (Milk No. 2b). Total fat-bound protein markedly increased during high temperature heating for the 75 °C, 2 s evaporator preheat treatment. When RCB was preheated prior to evaporation at 85 and 95 °C, 155 s, this increase occurred during preheating.

Total and individual fat-bound proteins in RCB at different evaporator preheat treatments and heating process stage as determined by PAGE analysis for Milk Nos. 8 and 2b are reported in Table 6.16.

Fat-bound total whey protein (Table 6.16) increased on intermediate heating when RCB was preheated prior to evaporation at 75 °C, 2 s and 85 °C, 155 s. There was a small decrease in fat-bound total whey protein with heating process stage when RCB was preheated at the 95 °C, 155 s evaporator preheat treatment, after an initial increase on UHT preheating.

There was no a clear trend found for fat-bound κ-casein in RCB with increasing evaporator preheat treatment.

Table 6.16 The effects of evaporator preheat treatment and heating process stage on total fat-bound protein and individual fat-bound proteins (at the constant UHT preheat treatment of 75 °C, 11 s) by PAGE analysis in the feed, preheated, intermediate heated and UHT sterilized and cooled RCB (Milk Nos. 8 and 2b).

Proteins in the cream layer	Evap. preheat treatment	Milk No.	Densitometer reading (arbitrary units)			
			Feed RCB [1]	Preheated RCB (75 °C)	Intermediate heated RCB (126 °C)	UHT RCB (140 °C)
α_s-casein	75 °C, 2 s	8	604	626	832	696
		2b	498	528	551	763
	85 °C, 155 s	2b	501	654	505	676
	95 °C, 155 s	2b	534	586	532	847
β-casein	75 °C, 2 s	8	567	575	799	712
		2b	325	326	360	506
	85 °C, 155 s	2b	360	460	363	490
	95 °C, 155 s	2b	401	417	382	674
κ-casein	75 °C, 2 s	8	178	174	162	135
		2b	126	128	113	147
	85 °C, 155 s	2b	141	181	117	141
	95 °C, 155 s	2b	156	156	120	156
γ-casein	75 °C, 2 s	8	21	17	12	13
		2b	11	13	9	15
	85 °C, 155 s	2b	10	13	7	11
	95 °C, 155 s	2b	13	16	9	15
Total casein	75 °C, 2 s	8	1370	1391	1805	1554
		2b	959	998	1033	1428
	85 °C, 155 s	2b	1014	1307	995	1321
	95 °C, 155 s	2b	1103	1174	1044	1692
β-lg	75 °C, 2 s	8	37	49	147	122
		2b	35	48	80	117
	85 °C, 155 s	2b	106	162	114	133
	95 °C, 155 s	2b	159	161	101	126
α-la	75 °C, 2 s	8	9	5	21	28
		2b	11	20	20	39
	85 °C, 155 s	2b	19	29	27	42
	95 °C, 155 s	2b	30	34	28	45
Total whey protein	75 °C, 2 s	8	46	54	168	151
		2b	45	67	100	156
	85 °C, 155 s	2b	125	191	142	175
	95 °C, 155 s	2b	189	195	129	171
TOTAL PROTEIN	75 °C, 2 s	8	1417	1445	1973	1704
		2b	1004	1065	1133	1584
	85 °C, 155 s	2b	1139	1498	1137	1496
	95 °C, 155 s	2b	1292	1369	1173	1862

[1]RCB prior to UHT preheat treatment.

6.7 Fat-bound total whey protein and fat-bound total casein as proportions of total fat-bound protein in the cream layers of FWM, RCB and Recon

From PAGE analysis, further investigation was carried out to calculate the fat-bound total casein and fat-bound total whey protein as proportions of total fat-bound protein in the cream layers of FWM, RCB and Recon. UHT FWM with the 95 °C, 147 s UHT preheat treatment was used as an example for this calculation. Densitometer reading of fat-bound total whey protein and fat-bound total casein (Table 6.10) was 290 and 1165 and densitometer reading of total fat-bound protein was 1456.

Thus, whey protein in the total fat-bound protein in the cream layer (%) is:

$$\frac{290}{1456} \times 100 = 19.9\ \%\ \textit{fat-bound whey protein / total fat-bound protein in FWM}$$

And casein in the total fat-bound protein in the cream layer (%) is:

$$\frac{1165}{1456} \times 100 = 80.0\ \%\ \textit{fat-bound casein / total fat-bound protein in FWM}$$

The proportions of fat-bound total whey protein and fat-bound total casein in the cream layers of FWM, RCB and Recon from Milk No. 10 were used to demonstrate the variation in these proportions between these milk preparations when the batch-to-batch (Milk No.) variation was removed (SMP and WMP were prepared from the same original batch of fresh whole milk as reported in Table 6.17).

The results in Table 6.17 show that the proportion of fat-bound total casein was much greater than the proportion of fat-bound total whey protein. The percentage fat-bound total whey protein was in the range 4-20 % and the percentage fat-bound total casein was in the range 83-96 %.

Table 6.17 The proportions of fat-bound total whey protein and fat-bound total casein in the cream layers of homogenized then preheated FWM, RCB and Recon (Milk No. 10).

Milk	Preheat treatment	Densitometer reading (arbitrary units)			Fat-bound total whey protein / total fat-bound protein (%)	Fat-bound total casein / total fat-bound protein (%)
		Fat-bound total whey protein	Fat-bound total casein	Total fat-bound protein		
Past. & homog. FWM	75 °C, 11 s	75	1154	1230	6	94
UHT FWM		219	1181	1399	16	84
Past. & homog. FWM	95 °C, 147 s	80	1075	1157	7	93
UHT FWM		290	1165	1456	20	80
RCB	SMP1, 75 °C, 11 s	46	1146	1193	4	96
UHT RCB		153	1490	1643	9	91
RCB	SMP1, 95 °C, 147 s	68	1373	1445	5	95
UHT RCB		238	1425	1664	14	86
RCB	SMP2, 75 °C, 11 s	125	1014	1139	11	89
UHT RCB		175	1321	1496	12	88
RCB	SMP3, 75 °C, 11 s	189	1103	1292	15	85
UHT RCB		171	1692	1862	9	91
Recon.	75 °C, 11 s	154	815	970	16	84
UHT Recon.		179	914	1095	16	83
Recon.	95 °C, 147 s	210	1009	1222	17	83
UHT Recon.		237	1194	1432	17	83

6.8 The effect of homogenization before or after UHT preheat treatment on total fat-bound protein and individual fat-bound proteins in FWM

Pasteurised FWM was subjected to homogenization before or after UHT preheat treatment. Two UHT preheat treatments, 75 °C, 11 s and 95 °C, 147 s were applied. The original total fat-bound protein data (by the Kjeldhal method and by PAGE analysis) were transformed to the $\log_{10}$ after the addition of the number one before statistical analysis. The total fat-bound protein by the Kjeldhal method is reported in Table 6.18.

Table 6.18 The effect of homogenization before or after UHT preheat treatment on total fat-bound protein, by the Kjeldhal method, in FWM (Milk Nos. 7 and 9), across heating process stage.

Homogenization and UHT preheat treatment	UHT preheat treatment	Milk No.	Total fat-bound protein (g) / fat (g) in the cream layer[1]				
			Past. FWM	Homog. FWM[2]	Homog. then preheated FWM (75 or 95 °C)	Intermediate heated FWM (126 °C)	UHT FWM (140 °C)
Homogenized then preheated	75 °C, 11 s	7	0.011	0.135	0.132	0.140	0.141
	95 °C, 147 s	7	0.012	0.138	0.141	0.145	0.152
		9	0.008	0.119	0.126	0.125	0.136
Homogenization and UHT preheat treatment	**UHT preheat treatment**	**Milk No.**	**Past. FWM**	**Homog. FWM**	**Preheated then homog. FWM (75 or 95 °C)**	**Intermediate heated FWM (126 °C)**	**UHT FWM (140 °C)**
Preheated then homogenized	75 °C, 11 s	7	0.011	n/a	0.059	0.095	0.109
	95 °C, 147 s	7	0.012	n/a	0.071	0.127	0.141
		9	0.008	n/a	0.094	0.099	0.109

[1]The values are individual values.

[2]Homog. FWM prior to UHT preheat treatment.

Generally, total fat-bound protein in homogenized then preheated FWM was much greater than that in preheated then homogenized FWM. Total fat-bound protein increased with severity of heating process stage for both milk batches and both UHT preheat treatments, and for both orders of homogenization and UHT preheat treatment.

Total fat-bound protein increased during both preheating and intermediate heating in FWM preheated at 75 °C, 11 s then homogenized. When FWM was preheated at 95 °C, 147 s and then homogenized, total fat-bound protein increased during both preheating and intermediate heating for Milk No. 7 and considerably increased during preheating for Milk No. 9. This trend is dissimilar to the trend found in homogenized then preheated FWM, for which total fat-bound protein did not change very much with heating process stage.

The individual fat-bound proteins in the same cream layers were analysed by PAGE. The results for homogenized then preheated FWM are reported in Table 6.19 and the results for preheated then homogenized FWM are reported in Table 6.20.

In homogenized then preheated FWM (Table 6.19), homogenization resulted in a marked increase in fat-bound total whey protein in the membrane covering the fat globules (6-7 times), compared with pasteurised FWM. When homogenized FWM was preheated at 75 °C, 11 s, there was an increase in whey protein in the membrane (1.4 times). When homogenized then preheated FWM was further heated during intermediate heating, the amount of whey protein doubled. During sterilization, there was no further association of whey protein. When homogenized FWM was preheated at 95 °C, 147 s, the amount of whey protein in the membrane increased (3.3 times for Milk No. 7 and 3.8 times for Milk No. 9) during preheating. There was no further increase during intermediate heating and sterilization.

When pasteurized FWM was subjected to preheating at 75 °C, 11 s before homogenization (Table 6.20), the amount of fat-bound total whey protein doubled. When the 75 °C, 11 s preheated FWM was homogenized, the association of whey protein in the membrane increased during homogenization (3.3 times) and it further increased during intermediate heating (2.7 times). During sterilization, there was a decrease in whey protein in this membrane (i.e. ~12 % decrease from the level of whey protein in the membrane of intermediate heated FWM).

Table 6.19 Total and individual fat-bound proteins in homogenized then preheated FWM by PAGE analysis at the 75 °C, 11 s UHT preheat treatment (Milk No. 7) and the 95 °C, 147 s UHT preheat treatment (Milk Nos. 7 and 9).

Proteins in the cream layer	UHT preheat treatment	Milk No.	Densitometer reading[1] (arbitrary units)				
			Past. FWM	Homog. FWM[2]	Homog. then preheated FWM (75 or 95 °C)	Intermediate heated FWM (126 °C)	UHT FWM (140 °C)
α_s-casein	75 °C, 11 s (Milk No. 7)	7	11	532	523	524	528
	95 °C, 147 s (Milk Nos. 7 and 9)	7	11	517	511	523	550
		9	0	465	505	492	574
β-casein	75 °C, 11 s (Milk No. 7)	7	8	440	413	437	425
	95 °C, 147 s (Milk Nos. 7 and 9)	7	9	428	387	423	443
		9	0	182	408	372	391
κ-casein	75 °C, 11 s (Milk No. 7)	7	4	98	91	76	69
	95 °C, 147 s (Milk Nos. 7 and 9)	7	3	96	67	73	77
		9	0	148	150	146	148
γ-casein	75 °C, 11 s (Milk No. 7)	7	0	20	15	9	7
	95 °C, 147 s (Milk Nos. 7 and 9)	7	0	19	7	9	9
		9	1	37	15	16	19
Total casein	75 °C, 11 s (Milk No. 7)	7	23	1090	1042	1046	1031
	95 °C, 147 s (Milk Nos. 7 and 9)	7	23	1060	973	1025	1077
		9	1	834	1078	1027	1135
β-lg	75 °C, 11 s (Milk No. 7)	7	8	57	82	159	145
	95 °C, 147 s (Milk Nos. 7 and 9)	7	10	55	191	193	191
		9	8	59	278	265	265
α-la	75 °C, 11 s (Milk No. 7)	7	1	13	13	30	36
	95 °C, 147 s (Milk Nos. 7 and 9)	7	1	13	31	39	42
		9	2	29	52	53	65
Total whey protein	75 °C, 11 s (Milk No. 7)	7	10	70	95	189	181
	95 °C, 147 s (Milk Nos. 7 and 9)	7	11	68	222	232	233
		9	11	88	330	319	329
TOTAL PROTEIN	75 °C, 11 s (Milk No. 7)	7	32	1160	1137	1235	1213
	95 °C, 147 s (Milk Nos. 7 and 9)	7	34	1128	1196	1257	1310
		9	12	922	1408	1346	1465

[1]The values are individual values.

[2]Homog. FWM prior to UHT preheat treatment.

Table 6.20 Total and individual fat-bound proteins in preheated then homogenized FWM by PAGE analysis at the 75 °C, 11 s UHT preheat treatment (Milk No. 7) and the 95 °C, 147 s UHT preheat treatment (Milk Nos. 7 and 9).

Proteins in the cream layer	UHT preheat treatment	Milk No.	Densitometer reading[1] (arbitrary units)				
			Past. FWM	Preheated FWM	Preheated then homog. FWM (75 or 95 °C)	Intermediate heated FWM (126 °C)	UHT FWM (140 °C)
α_s-casein	75 °C, 11 s (Milk No. 7)	7	11	19	218	318	335
	95 °C, 147 s (Milk Nos. 7 and 9)	7	11	22	229	433	447
		9	0	7	352	377	414
β-casein	75 °C, 11 s (Milk No. 7)	7	8	16	181	253	266
	95 °C, 147 s (Milk Nos. 7 and 9)	7	9	17	207	370	412
		9	0	6	270	270	249
κ-casein	75 °C, 11 s (Milk No. 7)	7	4	6	50	67	67
	95 °C, 147 s (Milk Nos. 7 and 9)	7	3	7	41	78	76
		9	0	10	112	117	122
γ-casein	75 °C, 11 s (Milk No. 7)	7	0	0	4	4	4
	95 °C, 147 s (Milk Nos. 7 and 9)	7	0	0	2	6	4
		9	1	0	4	8	8
Total casein	75 °C, 11 s (Milk No. 7)	7	23	41	453	642	671
	95 °C, 147 s (Milk Nos. 7 and 9)	7	23	46	478	888	939
		9	1	23	740	771	792
β-lg	75 °C, 11 s (Milk No. 7)	7	8	17	55	144	122
	95 °C, 147 s (Milk Nos. 7 and 9)	7	10	20	96	165	143
		9	8	22	182	174	161
α-la	75 °C, 11 s (Milk No. 7)	7	1	2	7	20	22
	95 °C, 147 s (Milk Nos. 7 and 9)	7	1	2	23	40	37
		9	2	1	58	71	55
Total whey protein	75 °C, 11 s (Milk No. 7)	7	10	19	62	164	144
	95 °C, 147 s (Milk Nos. 7 and 9)	7	11	22	119	205	180
		9	11	23	240	245	216
TOTAL PROTEIN	75 °C, 11 s (Milk No. 7)	7	32	60	515	806	815
	95 °C, 147 s (Milk Nos. 7 and 9)	7	34	68	597	1093	1119
		9	12	47	980	1016	1008

[1]The values are individual values.

When pasteurized FWM was preheated at 95 °C, 147 s, the fat-bound total whey protein doubled (compared with the amount of fat-bound total whey protein in pasteurised FWM). When the 95 °C, 147 s preheated FWM was homogenized, the association of whey protein further increased (5.4 times for Milk No. 7 and 10.5 times for Milk No. 9) and further increased during intermediate heating (in the case of Milk No. 7, but not Milk No. 9) and slightly decreased during sterilization (i.e. ~12 % decrease from the level of whey protein in the membrane of intermediate heated FWM).

The association of casein with the fat globules during homogenization was considerably larger than for whey protein whether FWM was homogenized before or after preheating (as expected given the increases in the surface of fat globules to be covered). Homogenization resulted in an increase in fat-bound total casein in homogenized FWM (46-47 times), compared with pasteurised FWM (Milk No. 7 ; Table 6.19). When pasteurized FWM was preheated and then homogenized, homogenization resulted in considerably less association of casein with the MFGM, for both low and high preheat treatments, than occurred when homogenization proceeded preheat treatment (Milk No. 7). Only small changes in fat-bound caseins occurred during intermediate heating and sterilization for both the homogenized then preheated FWM and the preheated then homogenized FWM.

When FWM was heated up to the intermediate heating temperature, more severe UHT preheat treatment resulted in an increase (2 times) in the association of whey protein in the membrane covering the fat globules for both the homogenized then preheated FWM and the preheated then homogenized FWM (even though the amount of protein covering the fat globules for the preheated then homogenized FWM was less than that for the homogenized then preheated FWM).

In general, fat-bound total whey protein increased when FWM was heated to 140 °C whether the FWM was homogenized before or after preheated. The increase of fat-bound total whey protein with heating process stage in the homogenized then preheated FWM was greater than that in the preheated then homogenized FWM for both UHT preheat treatments.

6.9 Discussion

Effect of UHT preheat treatment

When homogenized then preheated FWM, RCB and Recon were heated to 140 °C for sterilization, more severe UHT preheat treatment caused greater association of both β-lg and α-la with the respective MFGMs (Tables 6.10, 6.12 and 6.14). This increase of whey protein in the membrane covering the fat globules of (homogenized then preheated) FWM was greater than that in the MFGMs of RCB and Recon. This suggested that the process steps during skim milk powder and whole milk powder manufacture reduced the reactivity for the association of whey protein to the surface of fat globules for RCB and Recon, compared with (homogenized then preheated) FWM. Milk proteins and the natural membrane that covered the fat globules of (homogenized then preheated) FWM were more reactive than in Recon and RCB.

The association of both β-lg and α-la with the MFGMs of FWM, RCB and Recon increased when the milk was heated to 140 °C following the 75 °C, 11 s and the 95 °C, 147 s UHT preheat treatments (Tables 6.10, 6.12 and 6.14). At the UHT preheat treatment 75 °C, 11 s, the increase plateaued on intermediate temperature heating, but at the UHT preheat treatment 95 °C, 147 s, it plateaued on preheating. This result suggested that there was a limitation of the number of binding sites for the association of whey protein with the MFGMs of FWM, RCB and Recon. The more intense UHT preheat treatment caused faster association of whey protein in these MFGMs. On the other hand, the UHT preheat treatment may reduce the propensity of whey protein to associate with the membrane. The greater UHT preheat treatment may lead to the lower subsequent reactivity of whey protein in terms of association with the MFGMs for all three whole milks.

The pattern of the association of whey protein with the MFGMs of FWM, RCB and Recon at low and high UHT preheat treatment occurred at the same time as the dissociation of κ-casein (Tables 6.10, 6.12 and 6.14). Although Sharma & Dalgleish (1994) reported that the association of whey protein with the casein micelles already in the MFGM was limited because the limited binding sites on the κ-casein in the membrane of FWM, the result from this present study suggested that the dissociation of κ-casein was complex. κ-casein may dissociate from the membrane due to the reduction of pH with more severe heating

(from preheating to high-temperature heating) (Singh & Creamer, 1989). On the other hand, whey protein-κ-casein complexes may dissociate from the MFGM of whole milk.

From Chapter 5, the fouling rates of FWM, RCB and Recon were not equivalent even though the three whole milks were prepared with the same fat content and total solids content and their fouling rates increased with more severe UHT preheat treatment. When these data are combined with the results in this chapter, it suggests that an increasing trend of fat-bound total whey protein might relate to an increasing trend of the fouling rates for FWM, RCB and Recon when these three milks are subjected to more severe UHT preheat treatment. Changes in the level of whey protein covering the fat globules must play a role in the deposition of fat and protein in the high temperature heater. However, it is probably not only whey protein alone that is related to the deposition of the fat globules of whole milks in the high-temperature heater, but ash and both casein and whey proteins in the milk plasma may also play a role in the deposition of these fat globules at the same time.

Effect of evaporator preheat treatment

The effect of evaporator preheat treatment on fat-bound total whey protein (for the constant UHT preheat treatment at 75 °C, 11 s) (Table 6.16) showed that fat-bound total whey protein in the MFGM of feed RCB increased with more severe evaporator preheating. This suggested that more intense evaporator preheating caused whey protein to become more reactive with respect to the surface of fat globules. Changes in protein during skim milk powder manufacture were reported by Dalgleish (1990), Singh & Newstead (1989), Oldfield *et al.* (1998a), Oldfield *et al.* (2005a) and Singh *et al.* (2007) who showed that evaporator preheating caused association of β-lg with κ-casein in the casein micelles and the self-aggregation of whey proteins. Thus, the greater association of whey protein with the MFGM of RCB (before UHT processing) could be due to the greater association of the greater self-aggregation of whey protein or the association of β-lg with κ-casein on the surface of casein micelles which are already in the MFGM of RCB.

At the low and medium evaporator preheat treatments (75 °C, 2 s and 85 °C, 155 s), the association of whey protein with the MFGM of RCB (Table 6.16) increased with heating process stage. There appeared to be a limitation of binding sites on intermediate heating at

the 75 °C, 2 s evaporator preheat treatment for Milk No. 8, but not for Milk No. 2b (Table 6.16). There was no such clear trend found when RCB was preheated at 85 °C, 155 s. At the 95 °C, 155 s evaporator preheat treatment, there was no increasing trend in the association of whey protein with the MFGM with heating process stage.

The increase in the amount of whey protein associated with the MFGM of RCB increased with heating process stage, but the extent of the increase became smaller as evaporator preheat treatment intensity increased. This result suggested that there was a limitation for the association of whey protein with the membrane covering the fat globules of RCB. When this information was combined with the data on fouling rate in Chapter 5, it was clear that an increase in fouling rate for RCB with increasing evaporator preheat treatment was not due to an increase in total fat-bound whey protein. It is unclear whether whey protein covering the fat globules related to the deposition of RCB in the high temperature heater.

The greater evaporator preheat treatment resulted in a greater association of κ-casein in the MFGM of feed RCB (Table 6.16). This result suggested that the association of κ-casein may be due to the association of whey protein-casein micelle complexes with the surface of fat globules during recombination.

When RCBs, prepared using different evaporator preheat treatments, were heated to 140 °C, there was no dissociation of κ-casein from the MFGM of RCB. This trend differed from the trend with the effect of UHT preheat treatment found for RCB.

Effect of homogenization before or after UHT preheat treatment for FWM

At low or high UHT preheat treatment, homogenization prior to UHT preheat treatment resulted in a greater ultimate association of whey protein and casein with the MFGM of FWM than did homogenization after UHT preheat treatment (Tables 6.19 and 6.20). This result suggested that the membrane covering the fat globules of homogenized then preheated FWM was thicker than the membrane of preheated then homogenized FWM. Effectively, preheating then homogenization resulted in greater interfacial spreading of casein on the surface of fat globules than when the milk had not been preheated before homogenization (Sharma & Dalgleish, 1994). The thinner membrane of preheated then

homogenized FWM was possibly due to the association of small molecules, which had disintegrated from the casein micelles in milk plasma. These small molecules were considered as κ-casein (Anema, 2007; Skelte Anema, personal communication, 2008).

There was a strong trend of the dissociation of κ-casein from the membrane covering the fat globules with increasing severity of heating process stage for the homogenized then preheated FWM (Tables 6.10). This dissociation of κ-casein was in the range 16-24 %, which was small, as the milk was heated up to 140 °C Anema (personal communication, 2008). The pattern of this dissociation was opposite to the pattern of the association of whey protein for both low and high preheat treatments (Table 6.10). This result suggested that the dissociation of κ-casein was possibly related to the association of whey protein to the surface of fat globules in the homogenized then preheated FWM. On the other hand, dissociation of κ-casein plateaued on intermediate heating. This result suggested that there was no connection between whey protein association with the MFGM, and κ-casein dissociation, after intermediate heating at either UHT preheat treatment.

For both low and high UHT preheat treatments, the pattern of the association of whey protein with the membrane of fat globules in preheated then homogenized FWM (Table 6.20) with heating process stage suggested that there was no limitation of binding sites for the association of whey protein to the membrane of fat globules during preheating and intermediate heating and this association was not as strong as the association of whey protein with the membrane of homogenized then preheated FWM.

From the results in Chapter 5, the fouling rate of homogenized then UHT preheat treatment FWM was greater than that of homogenized after UHT preheat treatment FWM. When this information was combined with the data in Tables 6.19 and 6.20, it suggested that there was a difference between the deposition of fat globules for homogenized then preheated FWM and for preheated then homogenized FWM. For homogenized then preheated FWM, the thicker membrane covering the fat globules may have contributed to the higher fouling rates in the high-temperature heater.

Generally, the Kjeldahl data does not show an effect of UHT preheat treatment on total fat-bound protein in any of the three whole milk preparations (Tables 6.8, 6.11 and 6.13).

Total fat-bound protein increased slightly with heating process stage in the case of RCB and Recon. It was clear from PAGE data that total fat-bound protein was dominated by fat-bound α_s-casein and β-casein (which are present in milk in the greatest proportions among all the milk proteins).

The estimated total fat-bound protein recovered in the cream layer represented the range 30-35 % of total fat-bound protein in the whole milk (Table 6.3). Within the range of large fat globules in the recovered cream layers, the estimated fraction of total surface area was 0.12-0.25. Thus, the greater part of the total surface area available in whole milk was that of the smaller fat globules in the top-serum, serum and sediment.

6.10 Conclusion

The association of whey protein with the MFGMs of FWM and RCB and Recon increased with the severity of UHT preheat treatment and with heating process stage. The more intense evaporator preheating during skim milk powder or whole milk powder manufacture caused whey protein to become less reactive with respect to the surface of fat globules in feed RCB or Recon. Homogenization of FWM before UHT preheat treatment gave a thicker membrane on the surface of fat globules than did homogenization of FWM after UHT preheat treatment. When homogenized then preheated FWM was heated up to 140 °C, there was a role of κ-casein, related to whey protein association with the membrane covering the fat globules. The pattern of the association of whey protein was not the same when FWM was subjected to homogenization after UHT preheat treatment. The possible role of whey protein associated with the MFGMs of FWM, RCB and Recon in the deposition of milk components in the high-temperature heater is further discussed in Chapter 7.

Chapter 7
Effect of preheat treatments on the total weight and composition of fouling deposits from whole milks

7.1 Objectives

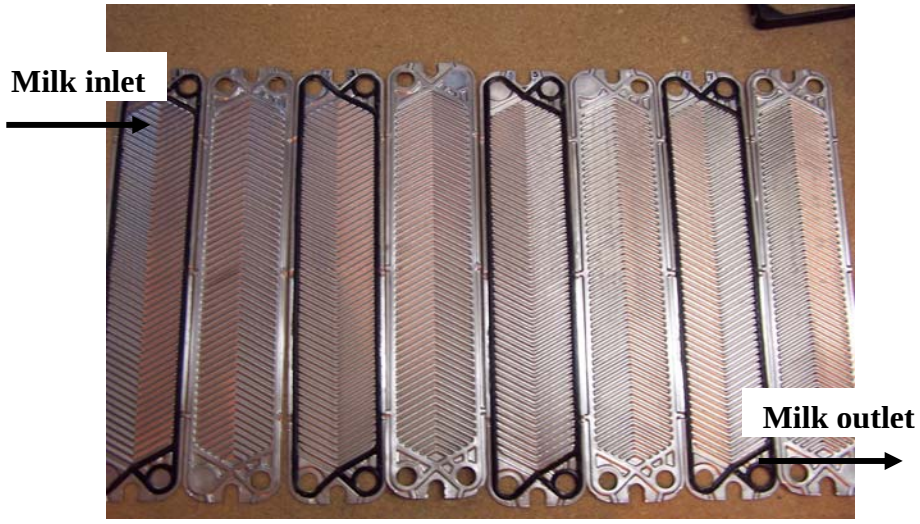
On the basis of the data presented in Chapters 5 and 6, there were five objectives in this study.

1. To investigate relationships between fouling rate and total weight for FWM, RCB and Recon.
2. To investigate changes in total weight and composition of deposits from FWM, RCB and Recon with the intensity of UHT preheat treatment.
3. To investigate the effect of homogenization before or after UHT preheat treatment on the total weight and composition of deposit from FWM.
4. To investigate changes in total weight and composition of deposit from RCB with the intensity of evaporator preheat treatment.
5. To investigate the effect of the presence of fat globules in the milk on total weight and composition of deposits from RCB and reconstituted skim milk.

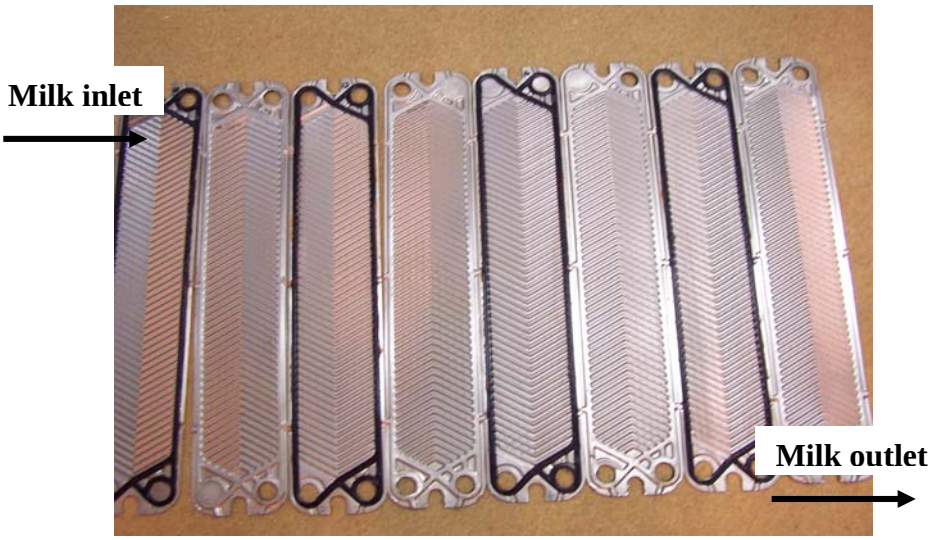
7.2 Deposit appearance

The deposits from FWM, RCB and Recon in the preheater, intermediate heater and high temperature heater were investigated by dismantling the heat exchanger after two hours of processing. The procedure for drying the deposit in the high-temperature heater was explained in Chapter 3.

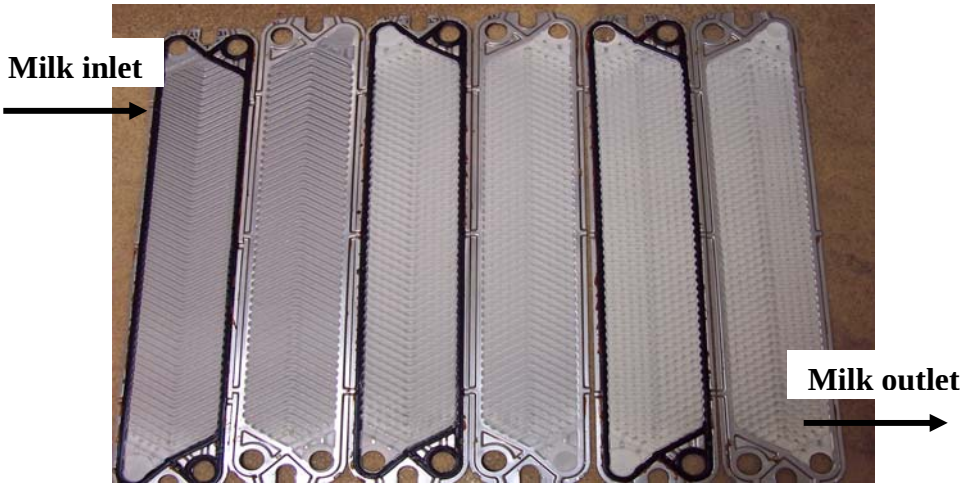
Figure 7.1 (a), (b) and (c) show the typical deposit distributions in the preheater, intermediate heater and high heater after a 2 h run with homogenized then preheated FWM.



(a) Preheater (Milk temperature increased from 10 °C to 95 °C)



(b) Intermediate heater (Milk temperature increased from 95 °C to 126 °C)



(c) High heater (Milk temperature increased from 126 °C to 140 °C)

Figure 7.1 (a)-(c) Surfaces of milk-contact plates from the preheater, intermediate heater and high heater showing the extent of fouling deposition after 2 h of processing from FWM with the UHT 95 °C, 147 s UHT preheat treatment (Milk No. 9).

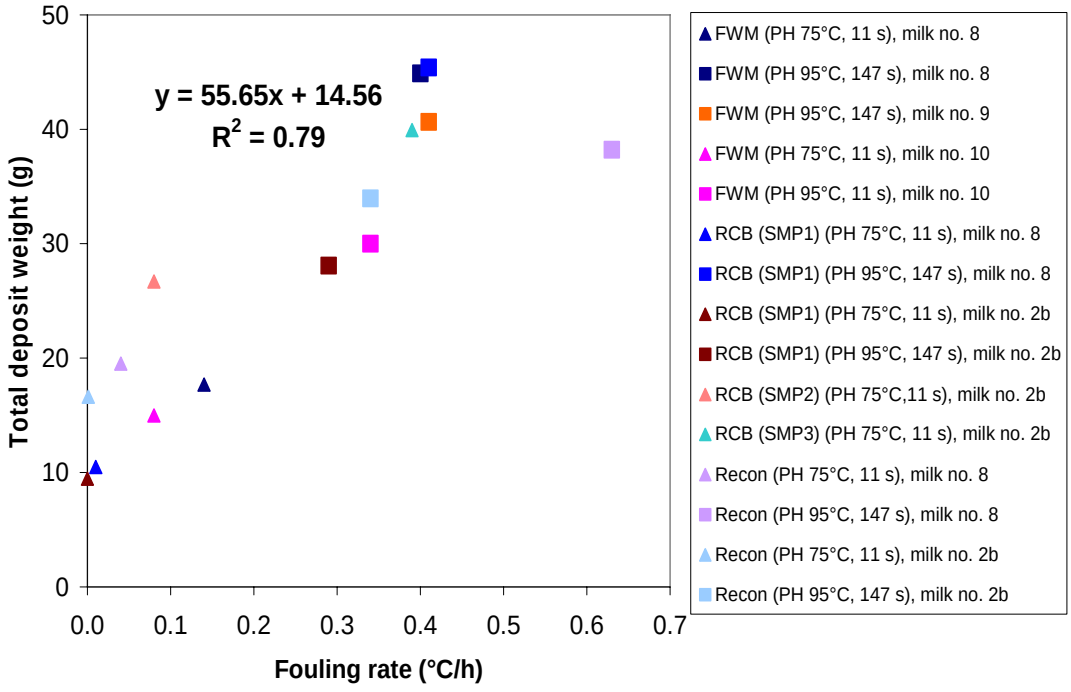
Figure 7.1 (a) and (b) showed that there were negligible amounts of deposit in the preheater and intermediate heater. In Figure 7.1 (c), the thickness of the deposit increased with the temperature of milk in the high-temperature heater. A thin deposit was found at the entrance of the high-temperature heater (126 °C) and a thick deposit was found at the exit (140 °C). Most deposit was located in the last pass of the high temperature heating section.

The nature of deposit in the last pass of the heat exchanger varied with whole milk preparation. The deposits from FWM and Recon were white and greasy, whereas the deposit from RCB was slightly yellowish and greasy. The deposit from reconstituted skim milk was browner, denser and thinner than the deposits from FWM, RCB and Recon.

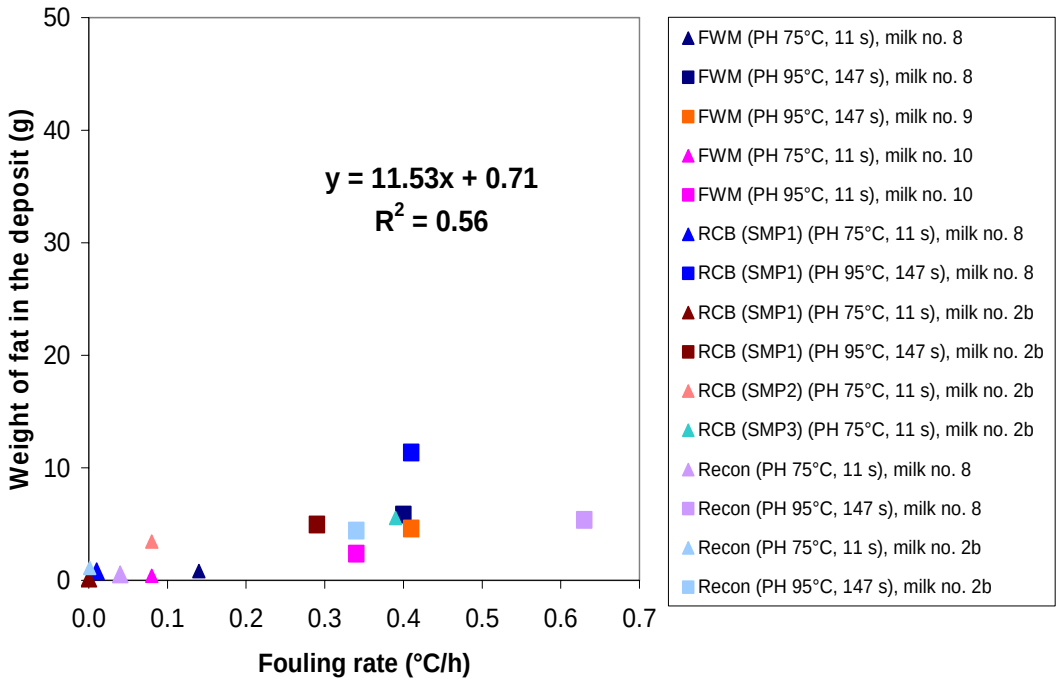
7.3 Fouling rates, and total deposit weight and composition of deposits from homogenized then preheated FWM, RCB and Recon

Total deposit weight (g dry matter) and the weights of each deposit component (g dry matter) are plotted against fouling rate in Figure 7.2 (a)-(d). The weights of the individual components of the deposits from FWM, RCB and Recon as determined by chemical analysis (fat, protein, ash and lactose) sum approximately to the total deposit weight as measured.

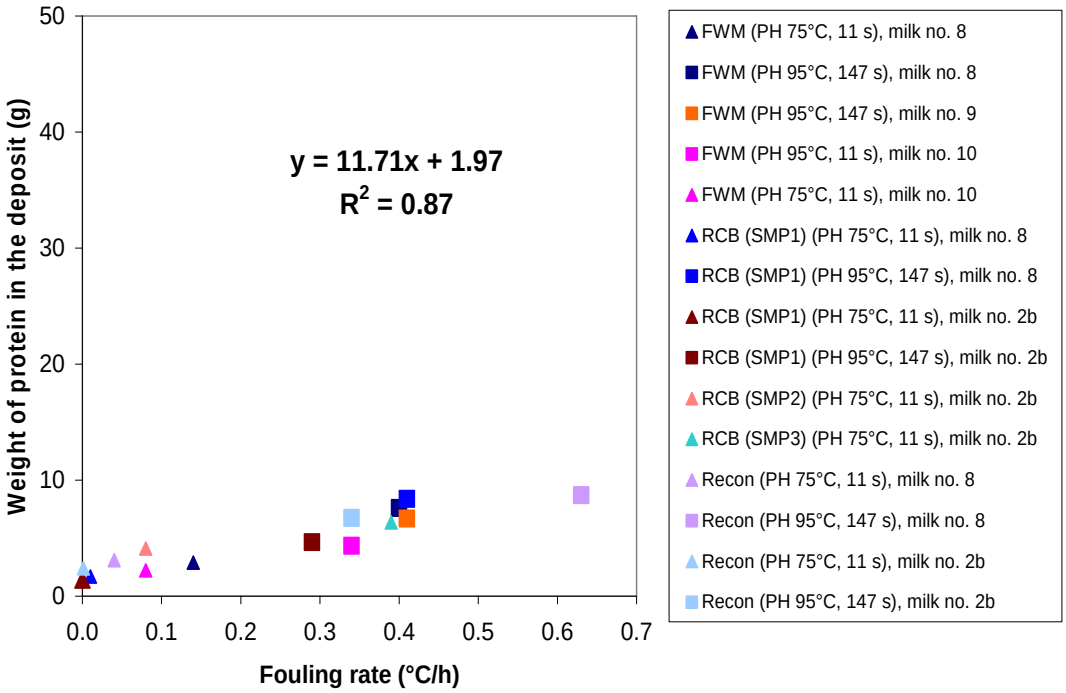
(a)



(b)


y = 11.53x + 0.71
R2 = 0.56
Weight of fat in the deposit (g)
Fouling rate (°C/h)
0
10
20
30
40
50
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
FWM (PH 75°C, 11 s), milk no. 8
FWM (PH 95°C, 147 s), milk no. 8
FWM (PH 95°C, 147 s), milk no. 9
FWM (PH 75°C, 11 s), milk no. 10
FWM (PH 95°C, 11 s), milk no. 10
RCB (SMP1) (PH 75°C, 11 s), milk no. 8
RCB (SMP1) (PH 95°C, 147 s), milk no. 8
RCB (SMP1) (PH 75°C, 11 s), milk no. 2b
RCB (SMP1) (PH 95°C, 147 s), milk no. 2b
RCB (SMP2) (PH 75°C, 11 s), milk no. 2b
RCB (SMP3) (PH 75°C, 11 s), milk no. 2b
Recon (PH 75°C, 11 s), milk no. 8
Recon (PH 95°C, 147 s), milk no. 8
Recon (PH 75°C, 11 s), milk no. 2b
Recon (PH 95°C, 147 s), milk no. 2b

(c)


y = 11.71x + 1.97
R2 = 0.87
Weight of protein in the deposit (g)
Fouling rate (°C/h)
0
10
20
30
40
50
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
FWM (PH 75°C, 11 s), milk no. 8
FWM (PH 95°C, 147 s), milk no. 8
FWM (PH 95°C, 147 s), milk no. 9
FWM (PH 95°C, 11 s), milk no. 10
FWM (PH 75°C, 11 s), milk no. 10
RCB (SMP1) (PH 75°C, 11 s), milk no. 8
RCB (SMP1) (PH 95°C, 147 s), milk no. 8
RCB (SMP1) (PH 75°C, 11 s), milk no. 2b
RCB (SMP1) (PH 95°C, 147 s), milk no. 2b
RCB (SMP2) (PH 75°C, 11 s), milk no. 2b
RCB (SMP3) (PH 75°C, 11 s), milk no. 2b
Recon (PH 75°C, 11 s), milk no. 8
Recon (PH 95°C, 147 s), milk no. 8
Recon (PH 75°C, 11 s), milk no. 2b
Recon (PH 95°C, 147 s), milk no. 2b

(d)

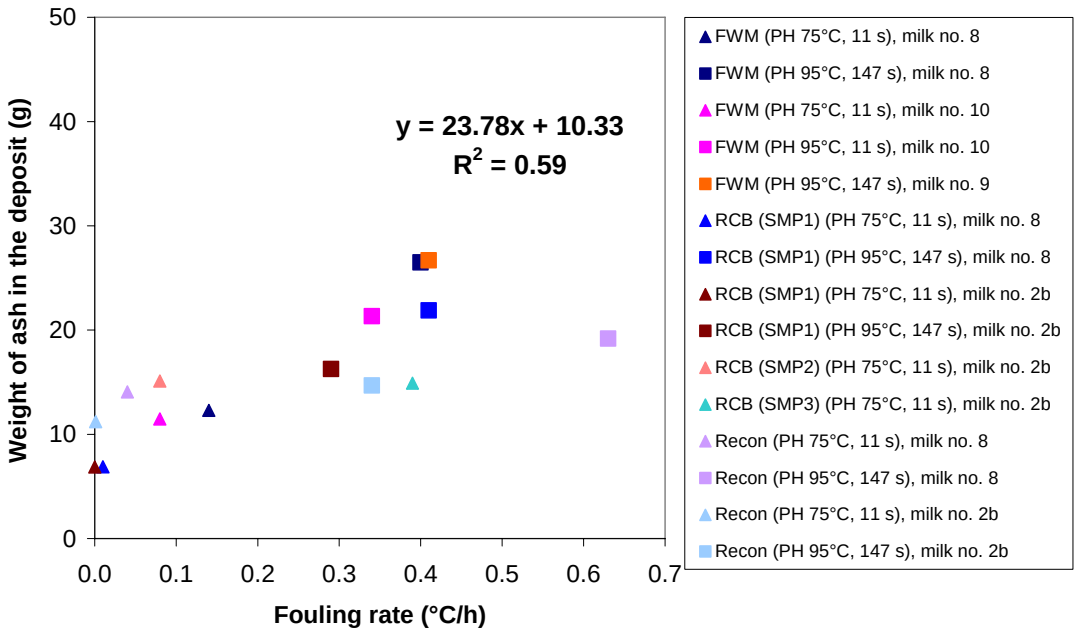


Figure 7.2 Total weight of the deposit, and the weights of fat, protein and ash in the deposits from homogenized then preheated FWM, RCB and Recon versus fouling rate (total weight of deposit (a), fat (b), protein (c) and ash (d)) (Milk Nos. 8-10 and 2b).

Note: - SMP1 (evaporator preheat treatment at 75 °C, 1 s).

- SMP2 (evaporator preheat treatment at 85 °C, 155 s).

- SMP3 (evaporator preheat treatment at 95 °C, 155 s).

- Recon (evaporator preheat treatment at 90 °C, 33 s).

Figure 7.2 (a) shows a good correlation between the fouling rate and the total deposit weight (a, $R^2 = 0.79$). There is also a strong relationship between the fouling rate and the weight of protein in the deposit (c, $R^2 = 0.87$) and moderate relationships between fouling rate and weight of fat in the deposit (b, $R^2 = 0.56$), and weight of ash in the deposit (d, $R^2 = 0.59$).

Figure 7.2 (b)-(d) shows that as fouling rate increases, the amount of ash deposited in the high heater increases (up to 20 g), which is much more than the amount of protein deposited (~ 10 g) and the amount of fat deposited (~ 7-8 g).

As is apparent from Figure 7.2 (a)-(d), these data could be conveniently grouped as low fouling rate (in the range 0-0.13 °C/h), medium fouling rate (in the range 0.28-0.42 °C/h) and high fouling rate (at 0.63 °C/h).

7.4 Effect of preheat treatments on total deposit weight and the weights of fat, protein, ash, phosphate, calcium, phosphorus and lactose in the deposits from homogenized then preheated FWM, RCB and Recon

The data on deposit components (fat, protein, ash, calcium, phosphate, phosphorus and lactose) in the deposits from FWM, RCB and Recon are presented in relation to UHT preheat treatment in section 7.4.1 and in relation to evaporator preheat treatment in the section 7.4.2.

7.4.1 Effect of UHT preheat treatment on the deposits from FWM, RCB and Recon

Homogenized then preheated FWM, RCB and Recon were subjected to two UHT preheat treatments (75 °C, 11 s and 95 °C, 147 s) prior to UHT processing. The drying procedure and analysis are described in Chapter 3. The results are summarised in Table 7.1.

The data in Table 7.1 were log-transformed for statistical analysis because this gave a better balance in the residuals than for the untransformed data. These log-transformed data were analysed using a split-plot design (the design was shown in Chapter 5). The main plot is milk preparation and the sub-plot is UHT preheat treatment within milk preparation. The statistical significance of the results are summarised in Table 7.2. The full results of statistical analysis are reported in Appendix 4 (Appendix A4.1).

Table 7.1 Fouling rate, total deposit weight and the weights of fat, protein, ash, phosphate, phosphorus, calcium and lactose in the deposits from homogenized then preheated FWM, RCB and Recon at the UHT preheat treatments 75 °C, 11 s and 95 °C, 147 s (Milk Nos. 8 -10 and 2b).

Milk No.	Milk	UHT preheat treatment	Fouling rate (°C/h)	Total deposit weight (g DM)	Weight of fat in the deposit (g DM)	Weight of protein in the deposit (g DM)	Weight of ash in the deposit (g DM)	Weight of PO_4 in the deposit (g DM)	Weight of phosphorus in the deposit (g DM)	Weight of calcium in the deposit (g DM)	Weight of lactose in the deposit (g DM)
8	FWM	75 °C, 11 s	0.14	17.7	0.8	2.9	12.3	4.9	2.1	3.6	0.01
		95 °C, 147 s	0.40	44.9	5.8	7.6	26.5	16.5	4.9	8.5	1.8
	RCB	75 °C, 11 s	0.01	10.5	0.8	1.7	6.9	n/a	n/a	n/a	0.001
		95 °C, 147 s	0.41	45.4	11.4	8.4	21.9	11.0	4.3	7.7	0.1
	Recon	75 °C, 11 s	0.04	19.5	0.5	3.1	14.1	4.9	2.6	4.7	0.004
		95 °C, 147 s	0.63	38.2	5.4	8.7	19.2	10.6	3.6	6.3	0.2
9	FWM	95 °C, 147 s	0.41	40.6	4.6	6.7	26.7	13.9	5.1	8.9	0.02
10	FWM	75 °C, 11 s	0.08	15.0	0.4	2.2	11.5	6.1	2.3	4.0	0.01
		95 °C, 147 s	0.34	30.0	2.4	4.3	21.3	12.4	4.3	7.4	0.02
2b	RCB	75 °C, 11 s	0.00	9.5	0.2	1.4	6.9	3.0	1.3	2.4	0.003
		95 °C, 147 s	0.29	28.1	4.9	4.6	16.3	8.1	3.1	5.5	0.03
	Recon	75 °C, 11 s	0.011	16.6	1.1	2.4	11.2	6.0	2.2	4.0	0.002
		95 °C, 147 s	0.34	34.0	4.4	6.7	14.7	9.2	2.9	5.0	0.02

Table 7.2 Probability values (p) for the effect of UHT preheat treatment, milk preparation and milk number on total deposit weight and the weights of fat, protein, ash, phosphate, phosphorus, calcium and lactose in the deposits from homogenized and preheated FWM (Milk Nos. 8-10), RCB (Milk Nos. 8 and 2b) and Recon (Milk Nos. 8 and 2b).

Deposit analyses	p-value		
	UHT preheat within milk preparation	**Milk preparation**	**Milk No.**
Fat	< 0.001	0.971	0.335
Protein	< 0.001	0.037	0.024
Ash	< 0.001	0.021	0.062
Ortho-PO_4	< 0.001	0.250	0.885
Calcium	< 0.001	0.217	0.092
Total phosphorus	< 0.001	0.126	0.079
Lactose	0.008	0.330	0.314
Total deposit weight	< 0.001	0.054	0.070

Main factors: *Milk No., milk preparation and UHT preheat treatment.*

The results in Table 7.1 shows that total deposit weight and the weights of all components in the deposits from FWM, RCB and Recon increased with the intensity of UHT preheat treatment. Table 7.2 shows this trend was highly significant for all components, and it was parallel to the trend of fouling rates as reported in Chapter 5.

The compositions of the deposits from FWM, RCB and Recon at a given (low or high) UHT preheat treatment were not markedly different ; but the deposition of all components was greater at the higher preheat treatment for all three milk preparations. The mineral components showed the greatest increase with preheating intensity for FWM, RCB and Recon.

Lactose (Table 7.1) was a soluble component trapped in the deposit, which was not removed by post-process rinsing prior to opening the heat exchanger. The residual lactose in the deposit was proportional to the amount of deposit.

7.4.2 Effect of evaporator preheat treatment on the deposit from RCB

The skim milk powders used in these trials were prepared using three evaporator preheat treatments before drying; 75 °C, 2 s, 85 °C, 155 s and 95 °C, 155 s. After recombination, RCB was processed for two hours through the UHT plant. The deposit in the high-temperature heater was collected using the procedure described in Chapter 3.

There were no replicates at the 85 °C, 155 s and 95 °C, 155 s evaporator preheat treatments. Thus, there was no statistical analysis of the effect of evaporator preheat treatment on total deposit weight and the weights of fat, protein, ash, PO_4, Ca, P and lactose in the deposit from RCB. However, the weights of fat, protein, ash, PO_4, Ca, P and lactose in the deposit from RCB in the high-temperature heater at three evaporator preheat treatments are reported in Table 7.3.

Table 7.3 shows that increasing the evaporator preheating intensity from 75 °C, 2 s to 85 °C, 155 s caused substantial increases in all weights in the deposit from RCB. On the other hand, changes in all weights when evaporator preheating intensity was increased further from 85 °C, 155 s to 95 °C, 155 s were relatively smaller and were variable; there were increases in the cases of total deposit weight, fat, protein and lactose, but slight decreases in the cases of ash, phosphate, total calcium and total phosphorus.

At this point, it can be concluded that the deposition of all milk components increased with the intensity of both UHT and evaporator preheat treatments, but not necessarily uniformly.

Table 7.3 Fouling rate, total deposit weight and the weights of fat, protein, ash, phosphate, phosphorus, calcium and lactose in the deposit from RCB at the evaporator preheat treatment 75 °C, 2 s, 85 °C, 155 s and 95 °C, 155 s and the constant UHT preheat treatment 75 °C, 11 s (Milk No. 2b).

Milk No.	Evaporator preheat treatment	Fouling rate (°C/h)	Total deposit weight (g DM)	Weight of fat in the deposit (g DM)	Weight of protein in the deposit (g DM)	Weight of ash in the deposit (g DM)	Weight of PO_4 in the deposit (g DM)	Weight of phosphorus in the deposit (g DM)	Weight of calcium in the deposit (g DM)	Weight of lactose in the deposit (g DM)
2b	75 °C, 2 s	0.00	9.5	0.2	1.4	6.9	3.0	1.3	2.4	0.003
	85 °C, 155 s	0.08	26.7	3.5	4.1	15.1	8.0	3.0	5.3	0.01
	95 °C, 155 s	0.39	39.9	5.5	6.4	14.9	6.9	2.4	4.2	0.38

7.5 Comparison of the effects of UHT preheating before or after homogenization on subsequent deposit formation by FWM

FWM was homogenized before or after preheat treatment prior to UHT processing. The results for total deposit weight and the weights of all deposit components from FWM are shown in Table 7.4.

Table 7.4 Total deposit weights and the weights of all deposit components from FWM when the milk was homogenized before or after UHT preheat treatment (Milk Nos. 8-10).

Deposit analyses	**Weight of deposit (g DM)**				
	Homog. then preheated at 75 °C, 11 s (Milk Nos. 8 and 10)	**Homog. then preheated at 95 °C, 147 s**		**Preheated then homog. at 75 °C, 11 s**	**Preheated then homog. at 95 °C, 147 s** (Milk No. 9)
		(Milk Nos. 8 and 10)	(Milk No. 9)		
Fat	0.6[1]	3.7[1]	4.6[3]	n/a[2]	1.3[3]
Protein	2.5	5.7	6.7	n/a	5.6
Ash	11.9	23.8	26.7	n/a	24.7
Ortho-PO_4	5.5	14.3	13.9	n/a	13.1
Calcium	3.8	7.9	8.9	n/a	8.3
Total phosphorus	2.2	4.6	5.1	n/a	4.8
Lactose	0.01	0.2	0.02	n/a	0.5
Total deposit weight	16.3	36.7	40.6	n/a	36.8

[1]Data in this column are geometric means.

[2]No trial was carried out for this combination.

[3]Data in this column are individual values.

These data could not be statistically analysed because of lack of replication. The only comparison that can be made is between homogenization then preheating and preheating then homogenization at the preheating condition of 95 °C, 147 s for Milk No. 9.

At the preheat treatment 95 °C, 147 s, the weight of fat in the deposit from homogenized then preheated FWM is 3-4 times greater than that in the deposit from preheated then homogenized FWM. There were only minor differences between homogenized then preheated FWM and preheated then homogenized FWM for total deposit weight and the weights of protein, ash and the other ash components.

7.6 Effect of the presence of fat globules in the deposit from RCB

A skim milk powder (SMP1), which was subjected to the evaporator preheat treatment of 75 °C, 2 s, was used to prepare reconstituted skim milk and RCB. After reconstitution, reconstituted skim milk and the RCB were preheated at 95 °C, 147 s prior to UHT processing. After 2 h processing, total deposit weight and the weights of fat, protein, ash, PO_4, Ca, P and lactose in the deposits from RCB and reconstituted skim milk were measured, and are reported in Table 7.5.

Table 7.5 Total deposit weight and the weights of deposit components from reconstituted skim milk and RCB at the UHT preheat treatment 95 °C, 147 s (Milk No. 8). Corresponding fouling rates are also shown.

Deposit analyses	**Weight of deposit (g DM)**	
	Reconstituted skim milk	**RCB**
Fat	n/a	11.4
Protein	3.2	8.4
Ash	14.5	21.9
Ortho-PO_4	7.8	11.0
Calcium	4.8	7.7
Total phosphorus	2.7	4.3
Lactose	n/a	0.1
Total deposit weight	19.3	45.4
Fouling rate (°C/h)	0.01	0.41

Table 7.5 shows that the total deposit weight for RCB was double the total deposit weight for reconstituted skim milk. The weights of protein, ash and ash components in the deposit from RCB were also considerably higher than those in reconstituted skim milk.

Next, some losses of milk components from the liquid milk were expected to be detectable on the basis of measurements of milk composition made before and after UHT processing. These losses were compared with the deposit formation in the high-temperature heater. The results are reported in the next section.

7.7 Composition of fouling deposits from FWM, RCB and Recon: effects of UHT and evaporator preheat treatments, and comparisons with changes in whole milk composition during UHT processing

Total deposit weight and the weights of components of the deposits from FWM, RCB and Recon were compared with changes in the composition of these whole milks during 2 h of processing. The effect of UHT preheat treatment in this study for FWM, RCB and Recon is reported in section 7.7.1 and the effect of evaporator preheat treatment for RCB is reported in section 7.7.2.

7.7.1 Effect of UHT preheat treatment

The data on total deposit weight, the weights of fat, protein, ash, PO_4, Ca, P and lactose in the deposits, and losses of milk components during UHT processing are presented in section (a) for FWM, section (b) for RCB and section (c) for Recon.

(a) Homogenized then preheated FWM

FWM was preheated at the UHT preheat treatments 75 °C, 11 s (Milk Nos. 8 and 10) and 95 °C, 147 s (Milk Nos. 8-10). The geometric means of the deposit total weight, the weights of deposit components and losses in milk components are presented in Table 7.6.

(b) Recombined whole milk

RCB was preheated at the UHT preheat treatments 75 °C, 11 s and 95 °C, 147 s prior to UHT processing (Milk Nos. 8 and 2b). The geometric means of deposit total weight, the weights of deposit components and losses of milk components are presented in Table 7.7.

Table 7.6 FWM: effects of UHT preheat treatment on deposit total weight and composition, and on changes in milk composition after 2 h of processing at the UHT preheat treatments 75 °C, 11 s (Milk Nos. 8 and 10) and 95 °C, 147 s (Milk Nos. 8-10).

Milk components	UHT preheat treatment at 75 °C, 11 s (Milk Nos. 8 and 10)				UHT preheat treatment at 95 °C, 147 s (Milk Nos. 8-10)			
	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)
	Feed milk	UHT milk	Losses of milk components (feed milk minus UHT milk)		Feed milk	UHT milk	Losses of milk components (feed milk minus UHT milk)	
Fat	8140[1]	8140[1]	0	0.6[1]	7960[1]	7960[1]	0	4.0[1]
Protein	7940	7920	20	2.5	7750	7730	20	6.0
Ash	1670	1660	10	11.9	1640	1590	50	24.7
Ortho- PO_4	410	400	10	5.5	420	390	30	14.2
Ca	280	270	10	3.8	270	250	20	8.2
P	210	210	0	2.2	210	200	10	4.8
Lactose	11380	11280	100	0.01	11210	11290	-80	0.1
Total deposit weight	-	-	-	16.3	-	-	-	38

[1] Data in this column are geometric means.

Table 7.7 RCB: effects of UHT preheat treatment on deposit total weight and composition, and on changes in milk composition after 2 h of processing (Milk Nos. 8 and 2b).

Milk components	UHT preheat treatment at 75 °C, 11 s (Milk Nos. 8 and 2b)				UHT preheat treatment at 95 °C, 147 s (Milk Nos. 8 and 2b)			
	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)
	Feed milk	UHT milk	Losses of milk components (feed minus UHT)		Feed milk	UHT milk	Losses of milk components (feed minus UHT)	
Fat	8080[1]	8130[1]	-50	0.4[1]	7320[1]	7310[1]	10	7.5[1]
Protein	7920	7980	-60	1.5	7190	6850	340	6.2
Ash	1710	1710	0	6.9	1580	1590	-10	18.9
Ortho- PO_4	420	410	10	3	390	380	10	9.4
Ca	280	280	0	2.4	260	250	10	6.5
P	210	210	0	1.3	200	190	10	3.7
Lactose	11240	11290	-50	0.002	10240	10240	0	0.057
Total deposit weight	-	-	-	10	-	-	-	35.7

[1] Data in this column are geometric means.

Table 7.8 Recon: effects of UHT preheat treatment on deposit total weight and composition, and on changes in milk composition after 2 h of processing (Milk Nos. 8 and 2b).

Milk components	UHT preheat treatment at 75 °C, 11 s (Milk Nos. 8 and 2b)				UHT preheat treatment at 95 °C, 147 s (Milk Nos. 8 and 2b)			
	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)
	Feed milk	UHT milk	Losses of milk components (feed minus UHT)		Feed milk	UHT milk	Losses of milk components (feed minus UHT)	
Fat	8140[1]	8160[1]	-20	0.7[1]	8010[1]	7980[1]	30	4.9[1]
Protein	8030	8010	20	2.7	7750	7770	-20	7.6
Ash	1740	1720	20	12.6	1690	1660	30	16.8
Ortho- PO_4	430	420	10	5.4	430	410	20	9.9
Ca	280	280	0	4.3	280	270	10	5.6
P	210	210	0	2.4	210	200	10	3.2
Lactose	11570	11470	100	0.003	11150	11090	60	0.1
Total deposit weight	-	-	-	18	-	-	-	36

[1] Data in this column are geometric mean.

(c) Reconstituted whole milk

Recon was preheated at the UHT preheat treatments 75 °C, 11 s and 95 °C, 147 s prior to sterilization (Milk Nos. 8 and 2b). The geometric means of deposit total weight, the weights of deposit components, and losses of milk components are presented in Table 7.8.

The results in Tables 7.6-7.8 show that the proportions of Ca and PO_4 in the deposits relative to the total concentrations of these components in the milk were so large that detection of losses of them in the milk was consistently noted when these milks were preheated at 95 °C, 147 s. The weights of ash in these deposits were found to be about half of the corresponding differences between the feed and UHT milk. On the other hand, the weights of protein and fat in the deposits of FWM, RCB and Recon were far too small relative to their total concentrations in the feed milks for depletion to be reliably detectable by the analytical methods used.

An apparent high loss of protein between feed milk and UHT milk for RCB at the UHT preheat treatment 95 °C, 147 s (Table 7.7) is due to an error in the chemical analysis of protein for Milk No. 2b.

7.7.2 Effect of evaporator preheat treatment

Skim milk powder used for the work reported in this study was prepared using three different evaporator preheat treatments, 75 °C, 2 s, 85 °C, 155 s and 95 °C, 155 s, before drying. After recombination, RCB was processed through the UHT plant. There were no replicates for the evaporator preheat treatments 85 °C, 155 s and 95 °C, 155 s. Thus, there was no statistical analysis for the effect of evaporator preheat treatment on total deposit weight and the weights of deposit components for RCB.

The geometric means of total deposit weight, the weights of deposit components and changes in milk composition for RCB are reported in Table 7.9.

Table 7.9 RCB: effect of evaporator preheat treatment on deposit total weight and composition, and on changes in milk composition after 2 h of processing (Milk Nos. 8 and 2b).

Milk components	Evaporator preheat treatment at 75 °C, 11 s (Milk Nos. 8 and 2b)				Evaporator preheat treatment at 85 °C, 155 s (Milk No. 2b)				Evaporator preheat treatment at 95 °C, 155 s (Milk No. 2b)			
	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)	Weight of milk component (g / 240 L milk)			Deposit weight (g DM)
	Feed milk	UHT milk	Losses of milk components (feed minus UHT)		Feed milk	UHT milk	Losses of milk components (feed minus UHT)		Feed milk	UHT milk	Losses of milk components (feed minus UHT)	
Fat	8080[1]	8130[1]	-50	0.4[1]	7790[2]	7890[2]	-100	3.5[2]	7910[2]	7890[2]	20	5.5[2]
Protein	7920	7980	-60	1.5	7880	7940	-60	4.1	7880	7850	30	6.4
Ash	1710	1710	0	6.9	1710	1660	50	15.1	1780	1730	50	14.9
Ortho- PO_4	420	410	10	3	440	420	20	8	430	410	20	6.9
Ca	280	280	0	2.4	290	280	10	5.3	290	280	10	4.2
P	210	210	0	1.3	220	220	0	3	230	220	10	2.4
Lactose	11240	11290	-50	0.002	11560	11720	-160	0.01	11230	11350	-120	0.38
Total deposit weight	-	-	-	10	-	-	-	26.7	-	-	-	39.9

[1]Data in this column are geometric means.

[2] Data in this column are individual values.

The results in Table 7.9 show that increasing evaporator preheating intensity from 75 °C, 2 s to 85 °C, 155 s caused substantial increases in all weights in the deposit from RCB. On the other hand, changes in all weights in the deposit when evaporator preheating intensity was increased from 85 °C, 155 s to 95 °C, 155 s were relatively much smaller, and variable; there were increases in the cases of the total deposit weight and the weights of fat and protein in the deposit from RCB, and a decrease in the case of ash.

Paralleling the deposit results above, losses of ash and its components from the milk at the evaporator preheat treatment of 85 °C, 155 s were much greater than losses at 75 °C, 11 s. However, losses were about the same at 85 °C, 155 s and 95 °C, 155 s. These trends reflect those shown by corresponding deposit weights.

7.8 Comparison of calcium, phosphate and phosphorus, and calcium phosphate in the deposits on a mole basis

Ash is the main milk component in the deposits from FWM, RCB and Recon. Thus, the moles of Ca, PO_4 and P in the deposits from FWM, RCB and Recon was investigated so as to be able to suggest the possible forms of Ca and PO_4. The moles of Ca, PO_4 and P were calculated as indicated in the footnotes to Tables 7.10 and 7.11. The results are reported in Tables 7.10 and 7.11.

Table 7.10 Moles of Ca, PO_4 and P in the deposits from FWM, RCB and Recon at the 75 °C, 11 s and 95 °C, 147 s UHT preheat treatments (Milk Nos. 8-10 and 2b).

Milk	Deposit composition	Molecular weight	Moles[1]		Molar ratio Ca:PO_4	
			UHT preheat treatment		UHT preheat treatment	
			75 °C, 11 s	95 °C, 147 s	75 °C, 11 s	95 °C, 147 s
FWM (Milk Nos. 8-10)	Ca	40	0.10	0.21	1.7	1.4
	Ortho-PO_4	95	0.06	0.15		
	P	31	0.07	0.15		
RCB (Milk Nos. 8 and 2b)	Ca	40	0.06	0.16	2.0	1.6
	Ortho-PO_4	95	0.03	0.10		
	P	31	0.04	0.12		
Recon (Milk Nos. 8 and 2b)	Ca	40	0.11	0.14	1.8	1.4
	Ortho-PO_4	95	0.06	0.10		
	P	31	0.08	0.10		

[1]Moles of each mineral component in the deposit = the weight of each mineral component in the deposit / molecular weight of that component (e.g. Moles of Ca in FWM, PH 75 °C, 11 s = 3.8 / 40 = 0.10 mole).
Note: Total phosphorus (P) in milk is dominated by ortho-PO_4 and the phosphoserine in the caseins. Contributions from the phosphorus-containing components, such as phospholipids, are generally negligible (Walstra & Jenness, 1984).

Table 7.11 Moles of Ca, PO_4 and P in the deposit from RCB at the 75 °C, 2 s and 95 °C, 155 s evaporator preheat treatments (Milk Nos. 8 and 2b).

Milk	Deposit composition	Molecular weight	Moles[1]		Molar ratio Ca:PO_4	
			Evaporator preheat treatment		Evaporator preheat treatment	
			75 °C, 2 s	95 °C, 155 s	75 °C, 2 s	95 °C, 155 s
RCB	Ca	40	0.06	0.11	2.0	1.6
	Ortho-PO_4	95	0.03	0.07		
	P	31	0.04	0.08		

[1]Moles of each mineral component in the deposit = the weight of each mineral component in the deposit / molecular weight of that component (e.g. Moles of Ca in FWM, PH 75 °C, 11 s = 2.4 / 40 = 0.06 mole).
Note: Total phosphorus (P) in milk is dominated by ortho-PO_4 and the phosphoserine in the caseins. Contributions from the phosphorus-containing components, such as phospholipids, are generally negligible (Walstra & Jenness, 1984).

Tables 7.10 and 7.11 show that the moles of Ca in the deposits was greater than the moles of both PO_4 and P in the deposits for both low and high preheat treatments and for all milk

types. The molar ratio of Ca:PO_4 for both UHT and evaporator preheat treatments is in the range of 1.4-2.0.

In most milk preparations, the moles of PO_4 and P for a given preheat treatment were not equal. The moles of PO_4 is always less or equivalent to the moles of P for both low and high preheat treatments.

7.9 Discussion

There were a clear positive relationships between the individual weights of fat, protein and ash in the deposits from FWM, RCB and Recon and fouling rate over than ranges of UHT and evaporator preheat treatments studied (Figures 7.1 and 7.2). This result showed that the measurement of temperature difference (milk outlet temperature - water inlet temperature in the high-temperature heater) reflected the deposit weight despite (totally expected) non-uniform deposition in the high-temperature heater. The increase in the extent of fouling with increasing temperature was very evident when the distribution of deposit was inspected in the high temperature heater (Figure 7.1 (c)).

Effect of UHT preheat treatment on the deposit composition

The total deposit weight and the weights of fat, protein and ash in the deposits from FWM, RCB and Recon generally increased with the intensity of UHT preheat treatment by a factor of two to four (Table 7.1). The variation in total deposit weight corresponded to the variation in fouling rates. The similar trend among the three whole milks suggested that there was a common basic fouling mechanism for FWM, RCB and Recon.

Ash constituted the largest proportion of the deposits from FWM, RCB and Recon for all UHT preheat treatments (Table 7.1). This indicated that the deposit was type B although it was not grey in colour, brittle and gritty as reported by Lyster (1965), Burton (1968), Lalande *et al.* (1984), Tissier *et al.* (1984) and Patil and Reuter (1988). The deposition of minerals can be due to the reverse temperature-dependence of the solubility of calcium phosphate in milk (Burton, 1968; Lalande *et al.*, 1984; Visser & Jeurnink, 1997).

The weight of fat in deposits increased to a greater extent than did the weight of protein for all milk types when UHT preheat treatment became greater (Table 7.1). These deposits were white and greasy because of the high levels of fat.

The deposition of fat in the high-temperature heater (Table 7.1) seemed to have been dependent on in the nature of the proteins covering the fat globules (differences in the MFGMs of FWM, RCB and Recon were described in Chapter 5). When the fat deposited in the high temperature heater, it probably deposited as fat-bound protein rather than as fat; i.e., the fat globules acted as protein particles. The results of Chapter 6 show that the amount of fat-bound whey protein increased with the intensity of UHT preheat treatment.

Increases in the weights of protein and fat in the deposit from RCB, compared with reconstituted skim milk (Table 7.5), showed the deposition of fat-bound protein. Furthermore, the deposition of protein from the milk plasma of reconstituted skim milk was lower than the deposition of protein and fat-bound protein for RCB.

The level of fat in the deposit from homogenization then UHT preheated FWM was greater than that in the deposit from UHT preheated then homogenized FWM (Table 7.4). From the results of Chapters 5 and 6, the thicker covering of protein around the fat globules of homogenized then UHT preheated FWM corresponded to a greater fouling rate. Thus, the thicker membrane of fat globules gave a greater level of fat in the deposit and a higher fouling rate.

Effect of evaporator preheat treatment on deposit composition

The increasing weight of protein in the deposit from RCB with increasing intensity of evaporator preheat treatment (Table 7.3) showed that the deposition of protein in the high temperature heater was related to evaporator preheating intensity. On the other hand, the weights of fat and ash in the deposit did not show a similar trend to the weight of protein when evaporator preheat treatment was increased from 85 °C, 155 s to 95 °C, 155 s. This result suggests that only protein has a role in the deposition toward an increase of total deposit weight and of fouling rate (Chapter 5).

Comparison of fouling deposits with changes in milk composition during UHT processing

Depletion in the weights of ash, PO_4, calcium, and total phosphorus between the feed and the UHT milk were consistently 1-2 times greater than the corresponding amounts recovered in the deposits in the high-temperature heater for all milk types (Tables 7.6-7.9). Although the ash constituted the greatest proportion of the deposit in the high-temperature heater, not all ash lost from the milk was recovered in the deposit. This may be due to the deposition of ash in other parts of the milk plant: preheater, holding tube, UHT holding tube, or cooling section (Lalande *et al.*, 1984; Tissier *et al.*, 1984; Burton, 1988; Patil & Reuter, 1988; Visser & Jeurnink, 1997), which this was not covered in this present study.

Depletion of fat and protein between the feed and the UHT milk for FWM, RCB and Recon was very low and was not be detectable, compared with the deposition of fat and protein in the high-temperature heater (Tables 7.6-7.9).

Molar ratio of Ca:PO_4

The molar ratio of Ca:PO_4 in the deposits of FWM, RCB and Recon varied in the range 1.4-2.0 and decreased with the intensity of UHT and evaporator preheat treatments (Tables 7.10 and 7.11). The forms of minerals in the deposit from fresh whole milk were reported by Burton (1968) and Lyster (1965) to be in the form of β-$Ca_3(PO_4)_2$, in which the ratios of Ca:P and Ca:PO_4 are 1.5:1. This was transformed into hydroxyapatite ($Ca_5OH(PO_4)_3$), giving a ratio of Ca/P of 1.6, when the precipitate was subjected to prolonged heating in the heat exchanger. Visser *et al.* (1997) reported that the deposit from FWM was a mixture of calcium phosphate dihydrate ($CaHPO_4.2H_2O$), which has a ratio of Ca:P of 1:1, and octacalcium phosphate ($Ca_8H_2(PO_4)_6.5H_2O$), which has a ratio of Ca:P of 1.3:1. The range of the molar ratio of Ca:PO_4 found in this study suggests that the ash in the deposits was a mixture of different forms of calcium phosphate.

7.10 Conclusions

The deposition of fat globules in the high-temperature heater is due to the level of protein covering the fat globules. Fat-bound whey protein seems to be involved in the fouling mechanism. The deposition of minerals in the high-temperature heater is due to the reverse solubility of calcium phosphate, which is present in the casein micelles and the milk serum. Homogenization of FWM either before or after UHT preheat treatment results in the deposition of fat in the high temperature heater. The basic fouling mechanisms for FWM, RCB and Recon are similar as are the effects of UHT and evaporator preheat treatments.

Chapter 8
Overall discussion

In all cases, for all milk preparations, fouling increased with increasing preheat treatment intensity (Chapters 5 and 7). Furthermore, the response of fouling rate to increasing heat treatment applied during milk powder manufacture was greater than that to preheat treatment applied prior to UHT processing.

This is the same trend as that found by Newstead *et al.* (1999); thus the apparent conflict with the results of other workers (Bell & Sanders, 1944; Burton, 1968; Patil & Reuter, 1986a, 1988; Mottar & Moermans, 1988) still remains. It is unclear whether the milks used in the comparisons made by these workers were derived from the same batches of original fresh milk. In the present study, to minimise variation within a set of comparisons, SMPs, WMPs and FWMs were derived from the same batch of original fresh milk (as described in Chapter 3). Most trials were replicated using different batches of commercial milk to cover any effects of natural variation in milk composition. The results were always the same, the more intense preheat treatments giving higher fouling rates.

Fouling rate for FWM increased with increasing UHT preheat treatment intensity for all process variations investigated; homogenized then preheated FWM, preheated then homogenized FWM, or unhomogenized FWM (the last condition was the case for other workers cited above except Mottar & Moermans (1988) where homogenization was applied after preheating). There was no instance in which fouling rate decreased with increasing intensity of UHT preheat treatment (Chapter 5).

In the case of RCB and Recon, fouling rate increased with increasing evaporator preheat treatment intensity when a constant low preheat treatment was applied prior to UHT processing (Chapters 5 and 7). The same results were found by Smith (1992), Harnett *et al.* (1997), Lean *et al.* (1996), Hill *et al.* (1997a) and Armstrong *et al.* (1998).

Casein and whey protein were found to be the common constituents of the membranes covering the surface of fat globules in the cream layer of FWM, RCB and Recon. It was

reasonable to assume that any processing-induced changes in the composition of fat-bound protein in the cream layer would reflect the same trends in the fat-bound protein from the unrecovered portion (top-serum, serum and sediment). Relative progressive changes in the average level of total fat-bound protein in the cream layers of these three whole milks with the severity of heating of successive process stages did not relate to the relative magnitudes of the fouling rates of these milks.

In the case of FWM, homogenization before UHT preheat treatment resulted in greater deposition of fat than homogenization after UHT preheat treatment (Chapter 7) and in a greater fouling rate (Chapter 5). The greater association of casein with the surface of fat globules, resulting from homogenization, evidently provided more binding sites for the association of whey protein during subsequent preheating (Chapter 6). Preheating non-homogenized FWM possibly resulted only in heat-induced changes in the milk plasma rather than the association of whey protein with the surface of fat globules.

Homogenization of preheated FWM resulted in less fat-bound protein, which possibly indicated a greater efficiency of surface coverage. The association of molecules with the membrane covering the surface of fat globules for preheated then homogenized FWM could be casein-whey protein complexes and small molecules (whey protein, molecules of casein from disintegrated micelles or small casein-whey protein complexes). These molecules gave greater interfacial spreading than in the case of FWM homogenized before preheat treatment.

In most cases, the ratio of protein to fat in the deposit was considerably higher than estimates made from the composition of fat-bound protein. This indicated that there was deposition of both fat-bound protein and non-fat-bound protein. The deposition of fat-bound protein was likely to have paralleled to the deposition of non-fat-bound protein from the milk plasma (i.e. aggregates of whey protein, casein micelles[1], sub-casein micelles[1], or casein-whey protein complexes). Although it is still unknown what sizes of fat globules and protein complexes preferentially deposit from the three whole milks studied, increasing preheat treatment resulted in an increase in the deposition of both fat-bound protein and non-fat-bound protein.

[1] The deposition of casein micelles and sub-micelles were unlikely to deposit alone unless they complexed with whey protein.

Various suggestions about the fouling mechanism have been put forward by different workers, differing mainly in whether the mineral or the denatured whey protein initiates the fouling process. Three basic processes appear to contribute to the fouling mechanism in the high-temperature heater of a UHT plant.

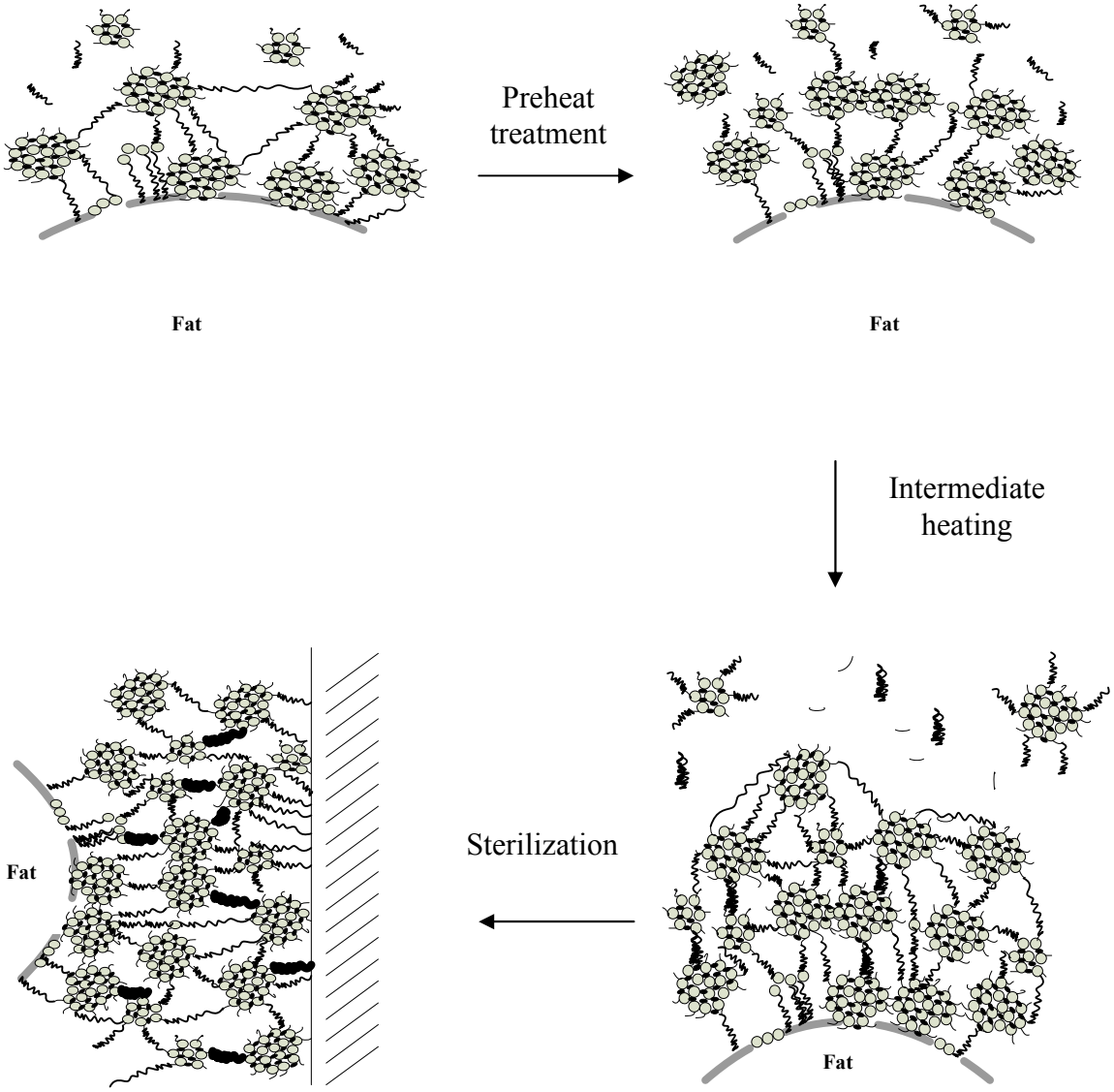
- Mineral appears to derive from the decreased solubility of calcium and phosphate at high temperature (Burton, 1968; Visser *et al.*, 1997; Visser & Jeurnink, 1997). The result implies that higher preheat treatment leads to a greater overall amount of insolubilized calcium phosphate complexes available for deposition in the high- temperature heater.
- Protein in the deposition appears to be mediated by denatured whey protein but involves also casein, probably including intact casein micelles. These proteins are possibly bound into the deposit through heat-induced linkages between κ-casein and β-lg.
- Fat in the deposit appears to be in the form of intact fat globules, covered by membranes comprising mainly milk proteins, both whey protein and caseins. Fat was simply complexed with the milk protein and was a "passenger" as the fat globules deposited in the high-temperature heater.

The deposition of insoluble calcium phosphate was likely to be the crucial step in the deposition for whole milks in the high-temperature heater because it constituted the largest proportion of the total deposit weight. It is still difficult to indicate the source of calcium phosphate in milk that is deposited, i.e. whether it is from the salts forming part of the complex in the casein micelle or more directly from the non-micellar portion. The concentrations of calcium phosphate in the structure of the casein micelles and in milk serum are similar (Walstra & Jenness, 1984).

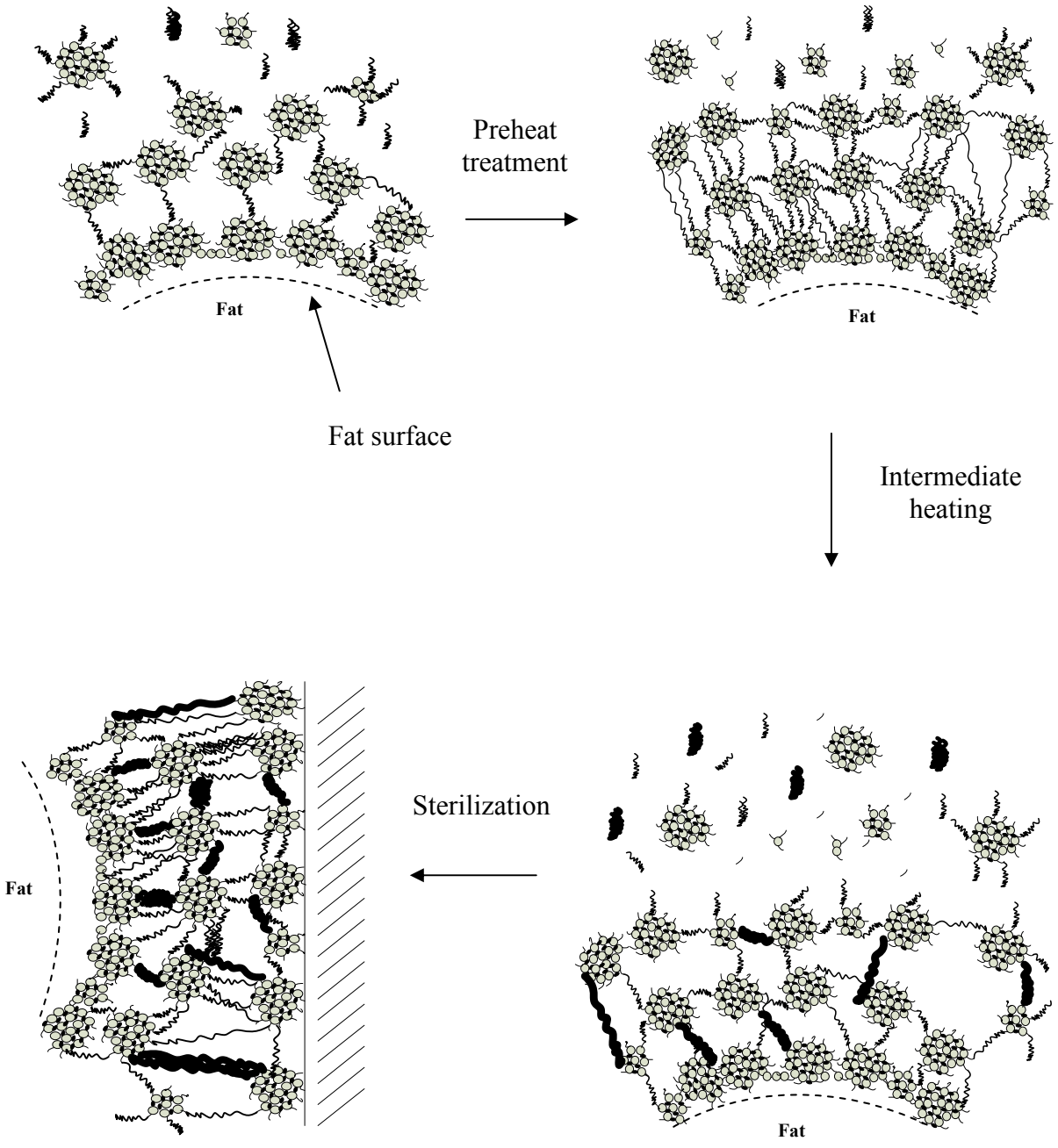
Mineral may deposit itself as crystallites or interact with protein (fat-bound protein or non fat-bound protein). These proteins could be absorbed onto the surface of mineral crystallites or the denatured proteins act as nuclei for crystal growth as suggested by Lyster (1965) and Patil & Reuter (1988). Although it is difficult to suggest whether mineral or protein deposited first on the heating surface in the high-temperature heater, both definitely increased with increasing preheat treatment.

Schematic diagrams of presumptive fouling mechanisms for FWM, RCB and Recon as described above are presented in Figures 8.1 (a), (b) and (c), respectively.

(a) FWM



(b) RCB


Preheat
treatment
Fat
Fat
Fat surface
Intermediate
heating
Sterilization
Fat
Fat

(c) Recon

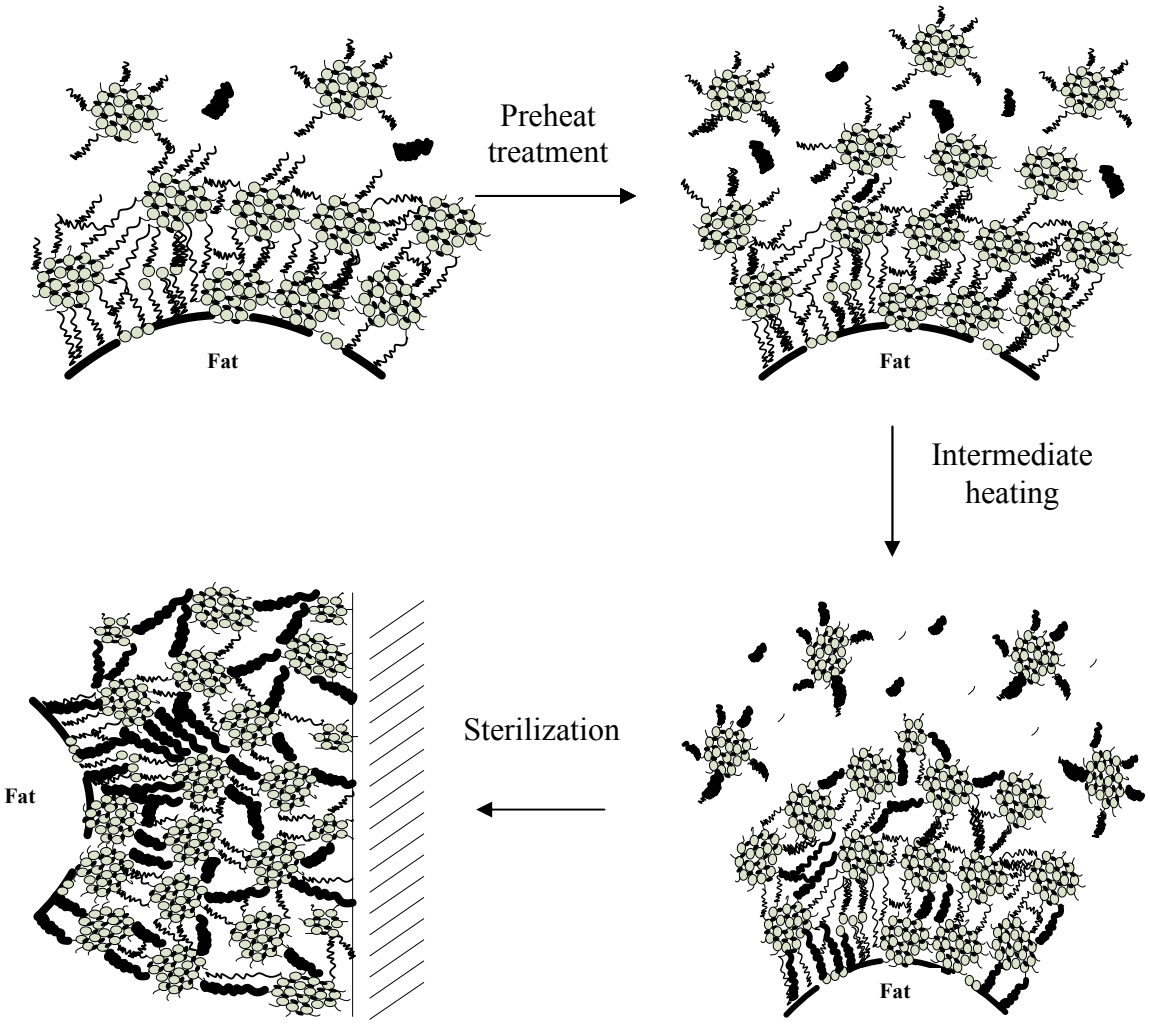


Figure 8.1 Schematic diagram for changes in casein and whey protein, both covering the fat globules and in milk plasma, and their deposition for homogenized then preheated FWM (a), RCB (b) and Recon (c) on the surface of stainless steel in the high-temperature heater of the heat exchanger.

= κ-casein

= Denatured whey protein

= Aggregates of whey protein

= Casein micelles

= Whey protein-casein complexes

 = Natural MFGM

 = Heated natural MFGM

Changes of both casein and whey protein in the MFGM of homogenized then preheated FWM in Figure 8.1 (a) showed that whey protein associated with the natural MFGM post pasteurisation. Casein micelles and fragments of casein micelles associated with the MFGM of FWM during homogenization. When FWM was preheated, the association of both whey protein and casein micelles in the MFGM of preheated FWM (Table 6.10) increased. During intermediate heating, the aggregates of whey protein, denatured whey protein or the complexes of whey protein-casein micelles from milk plasma further associated with casein micelles already in the MFGM through κ-casein and there was the dissociation of κ-casein. During sterilization, the fat globules covered with the casein micelles, whey protein, whey protein-casein micelle complexes and the whey protein aggregates from milk plasma deposited on the surface of the stainless steel in the high-temperature heater along with proteins from milk plasma. With the greater intensity of UHT preheat treatment, these components deposited to a greater extent on the surface of stainless steel in the high-temperature heater.

As indicated in RCB (Figure 8.1 (b)), there was no natural MFGM. After recombination (including homogenization), each droplet of anhydrous milk fat was covered by casein micelles, fragments of casein micelles and whey protein-casein micelle complexes. When RCB was preheated, the association of whey protein and casein micelles in the MFGM of preheated RCB increased (Table 6.12). During intermediate heating, there was further association of whey protein-casein micelle complexes and aggregates of whey protein from milk plasma with the MFGM of RCB, and there was the dissociation of κ-casein. During sterilization, there was the deposition of the fat globules, whey protein-casein complexes, whey protein aggregates, casein micelles and sub-casein micelles. The mechanism of deposition is speculated to be similar to that in the case of FWM.

As shown for Recon (Figure 8.1 (c)), the natural MFGM was heated during milk powder manufacture. There were whey protein-casein micelle complexes, whey protein aggregates and denatured whey protein in milk plasma of Recon. This differed from milk plasma of FWM. The reactivity of already heated natural MFGM for Recon was expected to not be

similar to the natural MFGM for homogenized then preheated FWM. Table 6.14 shows that the MFGM of Recon was very reactive to the association of whey protein during reconstitution, compared with the association of whey protein in the MFGM of FWM (Table 6.10). When Recon was preheated, there was the greater association of whey protein to the MFGM. During intermediate heating, the association of fat-bound total whey protein in Recon slightly changed and there was the dissociation of κ-casein. During sterilization, the deposition of fat globules for Recon, whey protein-casein micelles, whey protein aggregates, casein micelles, sub-casein micelles was speculated to be similar to the deposition occurring in the cases of FWM and RCB.

For all three whole milks, more severe preheating resulted in faster fouling. It is thought that this could be due to greater extents of whey protein denaturation, whey protein aggregation and whey protein-casein interaction.

The different genetic variants of β-lg have a major effect on fouling of UHT plants. Hill *et al.* (1997a) found that milk with only the A variant fouled about 10 times faster than the milk with only the B variant only. Milk with both variants, with approximately equal proportion of variants A and B, fouled about at least 2 times faster than milk with variant B only. With such spectacular differences in the behaviour of milk with different variants of β-lg, a major role for β-lg in the fouling mechanism is seemingly inescapable.

However, work beyond the scope of the data presented here will require further investigations to explain differences between the trends for the effect of preheat treatment on UHT fouling found in this study and the trends found by the workers cited above.

Future work on UHT fouling may include:

- The effect of pasteurization on the extent and sensitivity to preheat treatment intensity of fouling by whole milk.
- The effect of homogenization pressure on UHT fouling by FWM, RCB and Recon (This may suggest whether the deposition of fat globules depends on the particle size distribution of fat globules).
- The structure of deposits from FWM, RCB and Recon in the high-temperature heater, and how it varied with preheat treatment and milk type.

- Refinement of the method used in this work for the estimation of total fat-bound protein. This might involve calculating the density distribution of the fat globules in the milk sample using existing or measured data on the relationship between globule size and MFGM thickness. This would enable accurate identification of the "zero-buoyancy" globule size (see section 6.2.4). It might also lead to the possibility of calculating the distribution of fat globule size ranges, of fat globule volume fractions and fat-bound protein fractions in the serum top serum and cream layers after laboratory centrifugation.

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Appendix 1

Table A1.1 Comparison of an induction period of each fouling trial predicted from Method 1, Method 2 and linear regression (Milk No. 1-11, 2a and 2b).

Milk no.	Milk month	Process	Milk	Milk powder	Evaporator preheat treatment	UHT preheat treatment	Method 1			Method 2		Fouling rate (°C/h) from linear regression
							Induction period (h)	Fouling rate (°C/h)	RSS	Fouling rate (°C/h)	RSS	
1	Dec-03	Homog. --> preheat	FWM			75 °C, 1 s A	0.97	0.28	64.32	0.016	65.01	0.005
2	Dec-03	Homog. --> preheat	FWM			75 °C, 1 s B	0.00	0.16	30.88	0.16	30.88	0.15
1	Dec-03	Homog. --> preheat	FWM			75 °C, 123 s	0.47	0.36	11.28	0.26	12.65	0.27
1	Dec-03	Homog. --> preheat	FWM			95 °C, 123 s	0.58	0.48	14.28	0.33	14.93	0.32
1	Dec-03		RCB	SMP1	75 °C, 2 s	75 °C, 1 s A	0.58	0.00	30.65	0.00	30.65	-0.05
	Dec-03		RCB	SMP1	75 °C, 2 s	75 °C, 1 s B	0.56	0.34	33.58	0.23	34.13	0.23
1	Dec-03		RCB	SMP1	75 °C, 2 s	75 °C, 123 s	0.00	0.64	41.51	0.64	41.51	0.35
1	Dec-03		RCB	SMP1	75 °C, 2 s	95 °C, 123 s	0.00	0.17	6.59	0.17	6.59	0.16
1	Dec-03		RCB	SMP2	75 °C, 155 s	75 °C, 1 s	0.68	0.41	16.53	0.19	18.90	0.18
1	Dec-03		RCB	SMP3	95 °C, 155 s	75 °C, 1 s	0.58	0.68	14.42	0.43	18.38	0.43
1	Dec-03		Recon	WMP	95 °C, 33 s	75 °C, 1 s A	0.75	0.96	15.73	0.46	21.53	0.46
	Dec-03		Recon	WMP	95 °C, 33 s	75 °C, 1 s B	0.48	0.49	6.49	0.39	6.52	0.39
1	Dec-03		Recon	WMP	95 °C, 33 s	75 °C, 123 s	0.49	0.49	0.51	12.12	0.37	13.95
1	Dec-03		Recon	WMP	95 °C, 33 s	95 °C, 123 s	0.10	0.78	35.24	0.77	35.19	0.78
2	Oct-04	Homog. --> preheat	FWM			75 °C, 11 s	0.018	0.14	4.90	0.14	4.90	0.14
2	Oct-04	Homog. --> preheat	FWM			85 °C, 147 s	0.26	0.15	4.52	0.14	4.58	0.14
2	Oct-04	Homog. --> preheat	FWM			95 °C, 147 s A	0.29	0.23	4.99	0.21	5.07	0.21
2	Oct-04	Homog. --> preheat	FWM			95 °C, 147 s B	0.22	0.28	4.39	0.26	4.44	0.26

2	Oct-04		RCB	SMP1	75 °C, 2 s	75 °C, 11 s	0.19	0.020	3.68	0.018	3.69	0.021
2	Oct-04		RCB	SMP1	75 °C, 2 s	85 °C, 147 s	0.70	0.10	3.32	0.039	3.40	0.039
2	Oct-04		RCB	SMP1	75 °C, 2 s	95 °C, 147 s	0.083	0.20	3.10	0.19	3.10	0.19
2	Oct-04		RCB	SMP2	85 °C, 155 s	75 °C, 11 s	0.27	0.12	3.59	0.11	3.65	0.11
2	Oct-04		RCB	SMP3	95 °C, 155 s	75 °C, 11 s	0.023	0.46	3.49	0.46	3.49	0.46
2	Oct-04		Recon	WMP	95 °C, 33 s	75 °C, 11 s	0.33	0.077	5.38	0.064	5.49	0.064
2	Oct-04		Recon	WMP	95 °C, 33 s	85 °C, 147 s	0.16	0.23	3.93	0.23	3.98	0.22
2	Oct-04		Recon	WMP	95 °C, 33 s	95 °C, 147 s	0.11	0.59	2.15	0.57	2.14	0.57
3	Nov-04	Homog. --> preheat	FWM	FM		75 °C, 11 s	0.29	0.040	3.79	0.036	3.80	0.036
3	Nov-04	Homog. --> preheat	FWM	FM		85 °C, 147 s	0.94	0.20	3.92	0.070	4.10	0.070
3	Nov-04	Homog. --> preheat	FWM	FM		95 °C, 147 s A	0.042	0.16	4.48	0.16	4.49	0.16
3	Nov-04	Homog. --> preheat	FWM	FM		95 °C, 147 s B	0.00	0.16	4.71	0.15	4.71	0.15
3	Nov-04		RCB	SMP1	75 °C, 2 s	75 °C, 11 s	0.00	0.012	2.94	0.013	2.94	0.013
3	Nov-04		RCB	SMP1	75 °C, 2 s	85 °C, 147 s	0.66	0.12	3.62	0.050	3.79	0.050
3	Nov-04		RCB	SMP1	75 °C, 2 s	95 °C, 147 s	0.13	0.13	3.37	0.12	3.38	0.12
3	Nov-04		RCB	SMP2	85 °C, 155 s	75 °C, 11 s	0.72	0.016	3.67	0.0050	3.68	0.0050
3	Nov-04		RCB	SMP3	95 °C, 155 s	75 °C, 11 s	0.086	0.36	3.96	0.35	3.99	0.35
3	Nov-04		Recon	WMP	95 °C, 33 s	75 °C, 11 s	0.18	0.045	3.04	0.043	3.04	0.043
3	Nov-04		Recon	WMP	95 °C, 33 s	85 °C, 147 s	0.36	0.074	3.56	0.061	3.59	0.062
3	Nov-04		Recon	WMP	95 °C, 33 s	95 °C, 147 s	0.31	0.27	4.21	0.24	4.35	0.24
2a	Oct-04	Homog.	Reconst. skim milk	SMP1	75 °C, 2 s	75 °C, 11 s	1.46	0.88	3.52	-0.037	3.27	0.00
2a	Oct-04	No homog.	Reconst. skim milk	SMP1	75 °C, 2 s	75 °C, 11 s	1.46	0.88	3.49	-0.022	3.41	0.00
2a	Oct-04	Homog.	Reconst. skim milk	SMP1	75 °C, 2 s	95 °C, 147 s	1.43	0.83	3.78	0.0050	3.79	0.0047
2a	Oct-04	No homog.	Reconst. skim milk	SMP1	75 °C, 2 s	95 °C, 147 s	1.46	0.88	5.27	0.083	4.08	0.083
3a	Nov-04	Homog.	Reconst. skim milk	SMP1	75 °C, 2 s	75 °C, 11 s	0.28	-0.062	3.44	-0.045	3.50	0.000
3a	Nov-04	No homog.	Reconst. skim milk	SMP1	75 °C, 2 s	75 °C, 11 s	0.00	-0.028	3.86	-0.028	3.86	0.000
3a	Nov-04	Homog.	Reconst. skim milk	SMP1	75 °C, 2 s	95 °C, 147 s	0.30	0.061	3.49	0.030	3.66	0.030
3a	Nov-04	No homog.	Reconst. skim milk	SMP1	75 °C, 2 s	95 °C, 147 s	0.32	0.048	3.83	0.037	3.80	0.037
4	Jun-05		Recon	LHWMP	75 °C, 2 s	75 °C, 11 s	0.080	0.060	3.44	0.030	3.44	0.030
4	Jun-05		Recon	LHWMP	75 °C, 2 s	95 °C, 147 s	0.11	0.90	4.46	0.88	4.50	0.88
4	Jun-05		Recon	HHWMP	95 °C, 155 s	75 °C, 11 s	0.26	0.24	3.60	0.21	3.70	0.21

4	Jun-05		Recon	HHWMP	95 °C, 155 s	95 °C, 147 s	0.060	1.71	4.57	1.69	4.64	1.69
5	Jul-05		Recon	LHWMP	75 °C, 2 s	75 °C, 11 s	0.00	0.00	3.70	-0.049	3.37	0.00
5	Jul-05		Recon	LHWMP	75 °C, 2 s	95 °C, 147 s	0.00	0.17	3.78	0.17	3.78	0.17
5	Jul-05		Recon	HHWMP	95 °C, 155 s	75 °C, 11 s	0.25	0.15	3.27	0.13	3.26	0.13
5	Jul-05		Recon	HHWMP	95 °C, 155 s	95 °C, 147 s	0.00	0.52	3.94	0.52	3.94	0.52
6	AugA-05	Homog. --> preheat	FWM			75 °C, 11 s	0.013	0.15	4.75	0.15	4.75	0.15
6	AugA-05	Homog. --> preheat	FWM			95 °C, 147 s	0.22	0.58	4.33	0.55	4.66	0.55
6	AugA-05	Preheat --> homog.	FWM			75 °C, 11 s	0.25	0.20	3.59	0.18	3.59	0.18
6	AugA-05	Preheat --> homog.	FWM			95 °C, 147 s	0.29	0.21	4.11	0.19	4.17	0.19
7	AugB-05	Homog. --> preheat	FWM			75 °C, 11 s	1.21	0.25	3.12	0.0070	3.30	0.0073
7	AugB-05	Homog. --> preheat	FWM			95 °C, 147 s	0.25	0.23	4.06	0.23	4.06	0.21
7	AugB-05	Preheat --> homog.	FWM			75 °C, 11 s	0.44	0.068	3.23	0.051	3.32	0.051
7	AugB-05	Preheat --> homog.	FWM			95 °C, 147 s	0.49	0.16	3.93	0.12	4.19	0.12
8	Feb-06	Homog. --> preheat	FWM			75 °C, 11 s	0.10	0.14	8.25	0.14	8.25	0.14
8	Feb-06	Homog. --> preheat	FWM			95 °C, 147 s	0.28	0.43	6.91	0.40	7.47	0.40
8	Feb-06		RCB	SMP1	75 °C, 2 s	75 °C, 11 s	1.49	0.17	15.25	0.0050	15.77	0.0049
8	Feb-06		RCB	SMP1	75 °C, 2 s	95 °C, 147 s	0.0010	0.41	7.68	0.41	7.68	0.41
8	Feb-06		Recon	WMP	95 °C, 33 s	75 °C, 11 s	0.61	0.048	19.72	0.036	19.83	0.035
8	Feb-06		Recon	WMP	95 °C, 33 s	95 °C, 147 s	0.43	0.73	7.36	0.63	12.57	0.63
9	Jun-06	Homog. --> preheat	FWM			95 °C, 147 s	0.91	0.72	12.99	0.42	18.31	0.42
9	Jun-06	Preheat --> homog.	FWM			95 °C, 147 s	0.33	0.26	4.85	0.22	5.17	0.27
10	Oct-06	Homog. --> preheat	FWM			75 °C, 11 s	0.16	0.092	3.75	0.088	3.75	0.081
10	Oct-06	Homog. --> preheat	FWM			95 °C, 147 s	0.074	0.35	3.51	0.34	3.51	0.34
10	Oct-06	No homog.	FWM			75 °C, 11 s	0.15	0.00	5.02	0.00	5.02	0.00
10	Oct-06	No homog.	FWM			95 °C, 147 s	0.49	0.067	4.83	0.043	4.88	0.043
11	Apr-07	Homog. --> preheat	FWM			75 °C, 11 s	0.57	0.20	3.68	0.14	3.99	0.15
11	Apr-07	Homog. --> preheat	FWM			95 °C, 147 s	0.16	0.62	5.72	0.60	5.93	0.59
11	Apr-07	No homog.	FWM			75 °C, 11 s	0.017	0.076	3.81	0.075	3.81	0.070
11	Apr-07	No homog.	FWM			95 °C, 147 s	0.90	0.29	5.44	0.13	5.16	0.19
2b	Oct-04		RCB	SMP1	75 °C, 2 s	75 °C, 11 s	0.870	0.00	17.03	0.00	17.03	0.00
2b	Oct-04		RCB	SMP1	75 °C, 2 s	95 °C, 147 s	0.050	0.30	5.83	0.29	5.83	0.29

2b	Oct-04		RCB	SMP2	85 °C, 155 s	75 °C, 11 s	0.57	0.086	3.30	0.055	3.43	0.083
2b	Oct-04		RCB	SMP3	95 °C, 155 s	75 °C, 11 s	0.016	0.40	5.35	0.39	5.33	0.39
2b	Oct-04		Recon	WMP	95 °C, 33 s	75 °C, 11 s	2.00	0.26	4.27	0.0090	4.26	0.0011
2b	Oct-04		Recon	WMP	95 °C, 33 s	95 °C, 147 s	0.12	0.33	7.06	0.32	7.09	0.33

Table A1.2 Results for: Induction period (FWM)
General Linear Method: log (induction period versus UHT preheat treatment)

```
Factor                    Type   Levels  Values
UHT preheat treatment     fixed       3  75 11, 85147, 95147

Analysis of Variance for log (induction period+1), using Adjusted SS for Tests

Source                    DF   Seq SS   Adj SS   Adj MS     F      P
UHT preheat treatment      2  0.01877  0.01877  0.00939  0.92  0.418
Error                     16  0.16271  0.16271  0.01017
Total                     18  0.18148

S = 0.100843   R-Sq = 10.34%   R-Sq(adj) = 0.00%
```

Table A1.3 Results for: Induction period (RCB, prepared from SMP1)
General Linear Method: log (induction period versus UHT preheat treatment)

```
Factor                    Type   Levels  Values
UHT preheat treatment     fixed       3  75 11, 85147, 95147

Analysis of Variance for log (induction period+1), using Adjusted SS for Tests

Source                    DF   Seq SS   Adj SS   Adj MS     F      P
UHT preheat treatment      2  0.07282  0.07282  0.03641  2.55  0.147
Error                      7  0.09988  0.09988  0.01427
Total                      9  0.17269

S = 0.119451   R-Sq = 42.16%   R-Sq(adj) = 25.64%
```

Table A1.4 Results for: Induction period (Recon)
General Linear Method: log (induction period versus UHT preheat treatment)

```
Factor                    Type   Levels  Values
UHT preheat treatment    fixed       3  75 11, 85147, 95147

Analysis of Variance for log (induction period+1), using Adjusted SS for Tests

Source                    DF   Seq SS   Adj SS   Adj MS     F      P
UHT preheat treatment      2  0.03801  0.03801  0.01900  1.23  0.350
Error                      7  0.10851  0.10851  0.01550
Total                      9  0.14652

S = 0.124507   R-Sq = 25.94%   R-Sq(adj) = 4.78%
```

Table A1.5 Results for: Induction period (RCB, prepared from SMP1, SMP2 and SMP3)
General Linear Method: log (induction period versus evaporator preheat treatment)

```
Factor                          Type   Levels  Values
Evaporator preheat treatment    fixed       3  752, 85155, 95155

Analysis of Variance for log (induction period+1), using Adjusted SS for Tests

Source                          DF   Seq SS   Adj SS   Adj MS     F      P
Evaporator preheat treatment     2  0.04275  0.04275  0.02138  1.52  0.254
Error                           13  0.18234  0.18234  0.01403
Total                           15  0.22509

S = 0.118431   R-Sq = 18.99%   R-Sq(adj) = 6.53%
```

Appendix 2

Table A2.1 Results for: FWM, RCB and Recon
General Linear Model: log (FR+1) versus Milk No., Milk preparation and UHT PH

```
Factor            Type   Levels  Values
Milk No.          fixed       2  2, 3
Milk preparation  fixed       3  comWMP, FM, SMP1
UHT PH            fixed       3  75 11, 85147, 95147

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source                     DF     Seq SS     Adj SS     Adj MS      F      P
Milk No.                    1  0.0052239  0.0046646  0.0046646  14.67  0.009
Milk preparation            2  0.0062629  0.0060673  0.0030336   9.54  0.014
Milk No.*Milk preparation   2  0.0017826  0.0018127  0.0009063   2.85  0.135
UHT PH                      2  0.0178385  0.0193488  0.0096744  30.42  0.001
Milk preparation*UHT PH     4  0.0042958  0.0042958  0.0010739   3.38  0.090
Milk No.*UHT PH             2  0.0007831  0.0007831  0.0003915   1.23  0.356
Error                       6  0.0019079  0.0019079  0.0003180
Total                      19  0.0380947


S = 0.0178323   R-Sq = 94.99%   R-Sq(adj) = 84.14%
```

The effects of Milk No., milk preparation and the effect of UHT preheat treatment within milk preparation in Table 5.3 are individually adjusted by following the principle of split-plot analysis as described in Chapter 3. The results are shown below.

a) Effect of Milk No.

$$F = \frac{MS\ of\ the\ effect\ of\ Milk\ No.}{MS\ of\ the\ interaction\ of\ Milk\ No.*milk\ preparation}$$

$$F = \frac{0.0046646}{0.0009063}$$

$$F_{calculated} = 5.15$$

The degrees of freedom for the effect of Milk No. is 1 and the degree of freedom for the "interaction" of Milk No. and milk preparation is 2.

Thus, the p-value for the effect of Milk No. is 0.151.

b) Effect of milk preparation

$$F = \frac{MS\ of\ the\ effect\ of\ milk\ preparation}{MS\ of\ the\ interaction\ of\ Milk\ No.*milk\ preparation}$$

$$F = \frac{0.0030336}{0.0009063}$$

$$F = 3.35$$

The degrees of freedom for the effect of milk preparation is 2 and the degrees of freedom for the "interaction" of Milk No. and milk preparation is 2.

Thus, the p-value for the effect of milk preparation is 0.230.

c) Effect of UHT preheat treatment within milk preparation

$$F = \frac{MS\ of\ the\ effect\ of\ UHT\ PH + MS\ of\ the\ interaction\ of\ UHT\ PH * milk\ preparation}{MS\ of\ the\ interaction\ of\ Milk\ No.\ and\ UHT\ PH + MS\ of\ error}$$

$$F = \frac{0.0096744 + 0.0010739}{0.0003915 + 0.0003180}$$

$$F_{calaulated} = 15.15$$

The degree of freedom for this effect is more complicated than the other two effects. It involves the combination of the effects for the nominator and denominator. The degrees of freedom for the nominator are 6, which is the sum of two degrees of freedom for the effect of UHT PH and another four degrees of freedom for the "interaction" of UHT PH and milk preparation. The degrees of freedom of the denominator are eight, which is the sum of two degrees of freedom for the "interaction" of Milk No. and UHT PH and another six degrees of freedom for the error.

Thus, the p-value for the effect of UHT PH within milk preparation is 0.001.

Table A2.2 Results for: FWM, RCB and Recon
General Linear Model: log (FR+1) versus Milk No., Milk preparation and UHT PH

```
Factor            Type   Levels  Values
Milk No.          fixed       8  2, 3, 6, 7, 8, 9, 10, 11
Milk preparation  fixed       3  comWMP, FM, SMP1
UHT PH            fixed       3  75 11, 85147, 95147

Analysis of Variance for log(FR+1), using Adjusted SS for Tests

Source                   DF     Seq SS     Adj SS     Adj MS      F      P
Milk No.                  7  0.0358864  0.0277568  0.0039653   5.13  0.002
Milk preparation          2  0.0077224  0.0069939  0.0034969   4.52  0.025
UHT PH                    2  0.0671189  0.0642076  0.0321038  41.51  0.000
Milk preparation*UHT PH   4  0.0049085  0.0049085  0.0012271   1.59  0.219
```

```
Error                    19  0.0146961  0.0146961  0.0007735
Total                    34  0.1303322

S = 0.0278115   R-Sq = 88.72%   R-Sq(adj) = 79.82%
```

The effects of Milk No., milk preparation and the effect of UHT preheat treatment within milk preparation in Table 5.3 are adjusted by following the Split-plot analysis as shown below.

a) Effect of Milk No.

There is no “interaction” between Milk No. and milk preparation, which is the denominator for the effect of Milk No. Thus, the p-value for the effect of Milk No. is 0.002.

b) Effect of milk preparation

There is no “interaction” between Milk No. and milk preparation, which is the denominator for the effect of milk preparation. Thus, the p-value for the effect of milk preparation is 0.025.

c) Effect of UHT preheat treatment within milk preparation

$$F = \frac{MS\ of\ the\ effect\ of\ UHT\ PH + MS\ of\ the\ "interaction"\ of\ UHT\ PH * milk\ preparation}{MS\ of\ error}$$

$$F = \frac{0.0321038 + 0.0012271}{0.0007735}$$

$$F_{calaulated} = 43.1$$

The degrees of freedom of the nominator are six, which is the sum addition of two degrees of freedom from the effect of UHT preheat treatment and four degrees of freedom for the “interaction” between UHT preheat treatment and milk preparation. The degrees of freedom of the denominator are 19, which is from the degrees of freedom of error.

Thus, the p-value for the effect of UHT preheat treatment within milk preparation is < 0.001.

Table A2.3 Results for: RCB, prepared from SMP1, SMP2 and SMP3 (at constant UHT preheat treatment 75°C, 11 s)
General Linear Model: log (FR+1) versus Milk No. and evaporator preheat treatment

```
Factor              Type   Levels  Values
Milk No.            fixed       2  Nov-04, Oct-04
Evaporator PH       fixed       3  75 2, 85 155, 95 155

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source              DF     Seq SS     Adj SS     Adj MS      F      P
Milk No.             1  0.0010825  0.0010825  0.0010825   5.00  0.155
Evaporator PH        2  0.0234358  0.0234358  0.0117179  54.10  0.018
Error                2  0.0004332  0.0004332  0.0002166
Total                5  0.0249515

S = 0.0147178   R-Sq = 98.26%   R-Sq(adj) = 95.66%
```

Table A2.4 Results for: LH and HH WMP
General Linear Model: log (FR+1) versus Milk No., Evaporator PH and UHT PH

```
Factor         Type   Levels  Values
Milk No.       fixed       2  4, 5
Evaporator PH  fixed       2  95 155, 75 2
UHT PH         fixed       2  75 11, 95147

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source                  DF    Seq SS    Adj SS    Adj MS        F      P
Milk No.                 1  0.030805  0.030805  0.030805   392.20  0.032
Evaporator PH            1  0.019235  0.019235  0.019235   244.90  0.041
Milk No.*Evaporator PH   1  0.000432  0.000432  0.000432     5.50  0.257
UHT PH                   1  0.081051  0.081051  0.081051  1031.92  0.020
Evaporator PH*UHT PH     1  0.002673  0.002673  0.002673    34.03  0.108
Milk No.*UHT PH          1  0.021150  0.021150  0.021150   269.27  0.039
Error                    1  0.000079  0.000079  0.000079
Total                    7  0.155424

S = 0.00886252   R-Sq = 99.95%   R-Sq(adj) = 99.65%
```

With the split-plot analysis, the effects of Milk No., evaporator preheat treatment and UHT preheat treatment within evaporator preheat treatment are adjusted as shown below.

a) Effect of Milk No.

$$F = \frac{MS\ of\ the\ effect\ of\ Milk\ No.}{MS\ of\ the\ "interaction"\ of\ Milk\ No.*evaporator\ PH}$$

$$F = \frac{0.030805}{0.000432}$$

$$F_{calculated} = 71.31$$

The degree of freedom for the effect of Milk No. is one and the degree of freedom for the "interaction" of Milk No. and milk preparation is also one.

Thus, the p-value for the effect of Milk No. is 0.075.

b) Effect of evaporator preheat treatment

$$F = \frac{MS\ of\ the\ effect\ of\ evaporator\ preheat\ treatment}{MS\ of\ the\ "interaction"\ of\ Milk\ No.*evaporator\ PH}$$

$$F = \frac{0.019235}{0.000432}$$

$$F = 44.53$$

The degree of freedom for the effect of milk preparation is one and the degree of freedom for the "interaction" between Milk No. and milk preparation is also one.

Thus, the p-value for the effect of evaporator preheat treatment is 0.095.

c) Effect of UHT preheat treatment within evaporator preheat treatment

$$F = \frac{MS\ of\ the\ effect\ of\ UHT\ PH + MS\ of\ the\ "interaction"\ of\ UHT\ PH * evaporator\ PH}{MS\ of\ the\ "interaction"\ of\ Milk\ No.\ and\ UHT\ PH + MS\ of\ error}$$

$$F = \frac{0.081051 + 0.002673}{0.021150 + 0.000079}$$

$$F_{calaulated} = 3.96$$

The overall degrees of freedom for the nominator is two, which is the sum of one degree of freedom for the effect of UHT PH and another one degree of freedom for the "interaction" between UHT PH and evaporator preheat treatment. The degree of freedom of the denominator is two, which is the sum of one degree of freedom for the "interaction" between Milk No. and UHT PH and another one degree of freedom for the error.

Thus, the p-value for the effect of UHT preheat treatment within evaporator preheat treatment is 0.202.

Table A2.5 Results for: homog.-preheat FWM
General Linear Model: log (FR+1) versus Milk No. and UHT PH

```
Factor                  Type   Levels  Values
Milk No.                fixed       3  6, 7, 9
UHT PH                  fixed       2  75 11, 95147

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source                  DF     Seq SS     Adj SS     Adj MS      F      P
Milk No.                 2  0.0105316  0.0069889  0.0034945   5.62  0.286
UHT PH                   1  0.0109610  0.0109610  0.0109610  17.62  0.149
Error                    1  0.0006220  0.0006220  0.0006220
Total                    4  0.0221146

S = 0.0249390   R-Sq = 97.19%   R-Sq(adj) = 88.75%
```

Results for: Preheat-homog. FWM
General Linear Model: log (FR+1) versus Milk No. and UHT PH

```
Factor                   Type   Levels  Values
Milk No.                 fixed       3  AugA-05, AugB[05, Jun-06
UHT PH                   fixed       2  75 11, 95147

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source                   DF     Seq SS     Adj SS     Adj MS     F      P
Milk No.                  2  0.0034070  0.0026110  0.0013055  9.10  0.228
UHT PH                    1  0.0002446  0.0002446  0.0002446  1.71  0.416
Error                     1  0.0001434  0.0001434  0.0001434
Total                     4  0.0037950

S = 0.0119752   R-Sq = 96.22%   R-Sq(adj) = 84.88%
```

Table A2.6 Results for: Homog.-preheat FWM
General Linear Model: log(FR+1) versus Milk No. and UHT PH

```
Factor                   Type   Levels  Values
Milk No.                 fixed       2  Apr-07, Oct-06
UHT PH                   fixed       2  75 11, 95147

Analysis of Variance for log(FR+1), using Adjusted SS for Tests

Source                   DF     Seq SS     Adj SS     Adj MS      F      P
Milk No.                  1  0.0025586  0.0025586  0.0025586   4.55  0.279
UHT PH                    1  0.0136865  0.0136865  0.0136865  24.35  0.127
Error                     1  0.0005622  0.0005622  0.0005622
Total                     3  0.0168072

S = 0.0237101   R-Sq = 96.66%   R-Sq(adj) = 89.97%
```

Results for: No homog. FWM
General Linear Model: log (FR+1) versus Milk No. and UHT PH

```
Factor                   Type   Levels  Values
Milk No.                 fixed       2  Apr-07, Oct-06
UHT PH                   fixed       2  75 11, 95147

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source                   DF     Seq SS     Adj SS     Adj MS     F      P
Milk No.                  1  0.0018769  0.0018769  0.0018769  9.66  0.198
UHT PH                    1  0.0010384  0.0010384  0.0010384  5.34  0.260
Error                     1  0.0001943  0.0001943  0.0001943
Total                     3  0.0031096

S = 0.0139394   R-Sq = 93.75%   R-Sq(adj) = 81.25%
```

Table A2.7 Results for: Homog.-preheat FWM & no homog.-preheat FWM (UHT PH 75 11)
General Linear Model: log(FR+1) versus Milk No. and homogenization

```
Factor           Type   Levels  Values
Milk No.         fixed       2  10, 11
Homogenization   fixed       2  homog.-preheat, no homog.-preheat
```

```
Analysis of Variance for log(FR+1), using Adjusted SS for Tests

Source            DF     Seq SS     Adj SS     Adj MS       F      P
Milk No.           1  0.0007912  0.0007912  0.0007912  501.68  0.028
Homogenization     1  0.0010608  0.0010608  0.0010608  672.64  0.025
Error              1  0.0000016  0.0000016  0.0000016
Total              3  0.0018536

S = 0.00125582   R-Sq = 99.91%   R-Sq(adj) = 99.74%
```

Results for: Homog.-preheat FWM & no homog.-preheat FWM (UHT PH 95147)
General Linear Model: log(FR+1) versus Milk No. and homogenization

```
Factor            Type   Levels  Values
Milk No.          fixed       2  10, 11
Homogenization    fixed       2  homog.-preheat, no homog.-preheat

Analysis of Variance for log(FR+1), using Adjusted SS for Tests

Source            DF     Seq SS     Adj SS     Adj MS       F      P
Milk No.           1  0.0043267  0.0043267  0.0043267   59.68  0.082
Homogenization     1  0.0137676  0.0137676  0.0137676  189.89  0.046
Error              1  0.0000725  0.0000725  0.0000725
Total              3  0.0181668

S = 0.00851484   R-Sq = 99.60%   R-Sq(adj) = 98.80%
```

Table A2.8 Results for: Homo reconstituted skim milk and RCB
General Linear Model: log (FR+1) versus combination of milk preparation-UHT PH

```
Factor                   Type   Levels  Values
Milk preparation-UHT PH  fixed       4  recombined milk*75 11, recombined
                                        milk*95147, SMP1Homopreheat75 11,
                                        SMP1Homopreheat95147

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source                   DF     Seq SS     Adj SS     Adj MS     F      P
Milk preparation-UHT PH   3  0.0158784  0.0158784  0.0052928  5.82  0.033
Error                     6  0.0054541  0.0054541  0.0009090
Total                     9  0.0213325

S = 0.0301500   R-Sq = 74.43%   R-Sq(adj) = 61.65%
```

Table A2.9 Results for: Reconstituted skim milk (unhomog.-preheat and homog.-preheat)
General Linear Model: log (FR+1) versus combination of homogenization-UHT PH

```
Factor                 Type   Levels  Values
Homogenization-UHT PH  fixed       4  Homopreheat75 11, Homopreheat95147,
                                      Unhomopreheat75 11, Unhomopreheat95147

Analysis of Variance for log (FR+1), using Adjusted SS for Tests

Source                 DF     Seq SS     Adj SS     Adj MS     F      P
Homogenization-UHT PH   3  0.0008482  0.0008482  0.0002827  4.82  0.081
Error                   4  0.0002346  0.0002346  0.0000586
Total                   7  0.0010828

S = 0.00765821   R-Sq = 78.33%   R-Sq(adj) = 62.08%
```

Table A2.10 Results for: Ageing effect of RCB
General Linear Model: log FR+1 versus Milk No.

```
Factor    Type   Levels  Values
Milk No.  fixed       2  2, 2b

Analysis of Variance for log FR+1, using Adjusted SS for Tests

Source    DF    Seq SS    Adj SS    Adj MS     F      P
Milk No.   1  0.000006  0.000006  0.000006  0.00  0.972
Error      6  0.026469  0.026469  0.004411
Total      7  0.026474

S = 0.0664185   R-Sq = 0.02%   R-Sq(adj) = 0.00%
```

Fouling rates of RCB for Milk No. 2 were measured when the skim milk powder used in the RCB preparation was freshly made. SMP used for Milk No. 2b was aged for 24 months before recombination. Three batches of RCB from each Milk No. were subjected to three evaporator preheat treatments; 75 °C, 2 s, 85 °C, 155 s and 95 °C, 155 s. The measured fouling rates are shown in Figure A2.1.

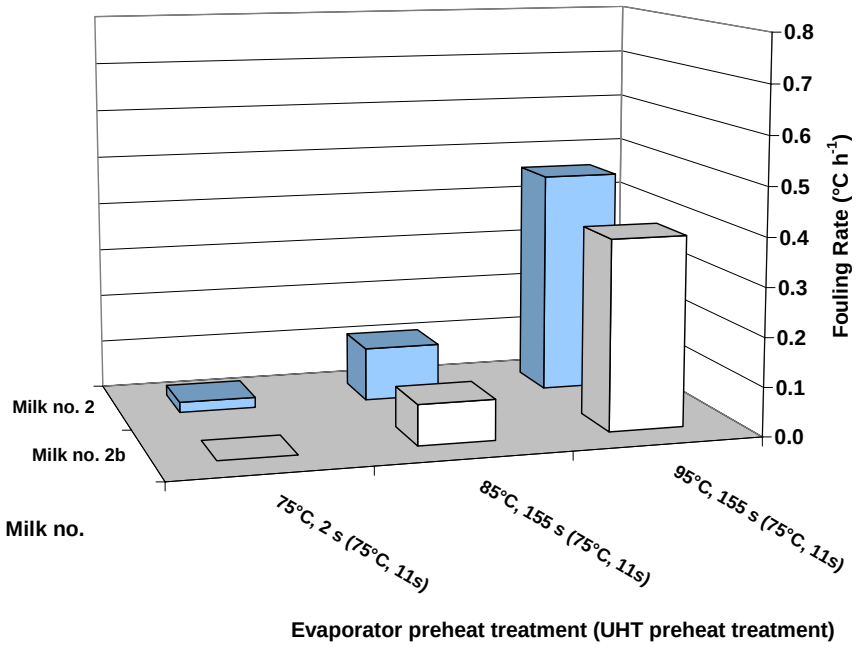

Figure A2.1 Fouling rate of RCB (made using different evaporator preheat treatments) and then subjected to the UHT preheat treatment 75 °C, 11 s (Milk Nos. 2 and 2b).

Statistical analysis of the data shown in Figure A2.1 provided no evidence that the fouling rate of derived RCB varied with milk number, i.e. milk powder age (p = 0.972).

Three batches of Recon for Milk No. 2 and two batches for Milk No. 2b (prepared using evaporator preheat treatment at 95 °C, 33 s). Fouling rates of Recon for Milk No. 2 were measured when the milk powder was freshly made. Fouling rates of Recon for Milk No. 2b were measured on Recon made from WMP from Milk No. 2 that had been aged for 24 months. The measured fouling rates are shown in Figure A2.2.

Table A2.11 Results for: Ageing effect of Recon
General Linear Model: log (FR +1) versus Milk No.

```
Factor    Type   Levels  Values
Milk No.  fixed       2  2, 2b

Analysis of Variance for log (FR +1), using Adjusted SS for Tests

Source    DF    Seq SS    Adj SS    Adj MS     F      P
Milk No.   1  0.001935  0.001935  0.001935  0.25  0.649
Error      3  0.022915  0.022915  0.007638
Total      4  0.024849

S = 0.0873967   R-Sq = 7.79%   R-Sq(adj) = 0.00%
```

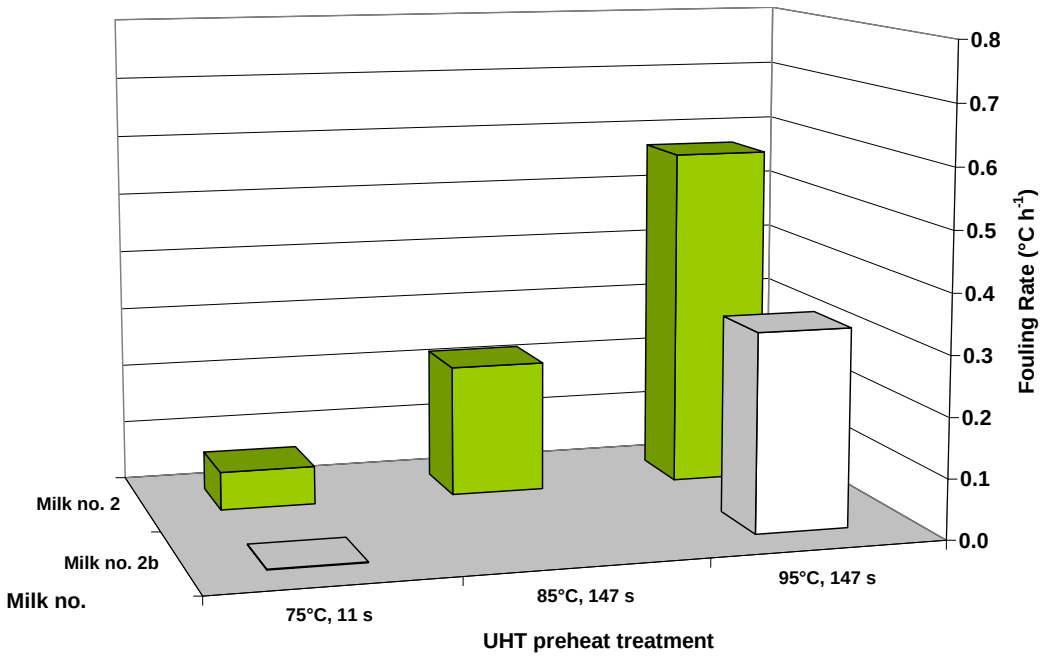

Figure A2.2 Fouling rate of Recon at UHT preheat treatments 75 °C, 11 s, 85 °C, 147 s and 95 °C, 147 s (Milk Nos. 2 and 2b).

Statistical analysis of the data shown in Figure A2.2 provided no evidence that the fouling rate of derived Recon varied with milk number, i.e. with the age of the whole milk powder ($p = 0.649$).

Thomas *et al.* (2004) reported that the effect of milk powder ageing related to changes caused by lactosylation (condensed of β-lg and lactose). Temperature is the main factor that causes lactosylation of milk protein, which can be avoided if the milk powder is kept at temperatures below 20 °C. In this study, skim milk powder and whole milk powder were kept at a controlled temperature of 14 °C to 16 °C. Therefore, it is likely that there was no lactosylation in either aged powder in this study.

Appendix 3

Table A3.1 The percentage of fat and TN in the cream layer, top-serum, serum and sediment for FWM, RCB and Recon (Milk No. 10).

Milk	Preheat treatment	Cream layer			Top-serum			Serum			Sediment			Milk liquid		
		% fat	% TN	weight (g)	% fat	% TN	weight (g)	% fat	% TN	weight (g)	% fat	% TN	weight (g)	% fat	% TN	weight (g)
Past. & homog. FWM	75 °C, 11 s	47.20	0.99	13.48	0.51	0.19	17.73	1.42	0.40	239	2.76	2.98	17.92	3.41	0.536	288
UHT FWM		44.60	1.08	13.08	0.46	0.16	18.18	1.43	0.39	237	3.59	2.56	19.45	3.41	0.539	288
Past. & homog. FWM	95 °C, 147 s	48.00	0.96	13.56	0.57	0.18	17.22	1.32	0.39	239	2.34	2.85	17.94	3.38	0.532	288
UHT FWM		46.80	1.05	13.13	0.44	0.16	17.27	1.40	0.36	236	3.65	2.43	21.91	3.37	0.530	288
RCB	SMP1, 75 °C, 11 s	46.20	1.06	13.96	0.35	0.16	17.25	1.28	0.30	225	2.32	2.17	31.85	3.37	0.525	288
UHT RCB		33.40	1.19	14.34	0.26	0.17	17.35	0.87	0.23	224	4.36	2.71	32.47	3.40	0.529	288
RCB	SMP1, 95 °C, 147 s	44.60	0.97	14.41	0.30	0.16	16.90	1.15	0.30	229	2.21	2.26	27.88	3.25	0.499	288
UHT RCB		43.50	1.11	14.55	0.22	0.15	17.31	1.02	0.25	228	2.97	2.76	28.38	3.25	0.500	288
RCB	SMP2, 75 °C, 11 s	41.70	1.03	14.82	0.37	0.14	17.44	1.38	0.31	227	2.18	2.34	28.49	3.33	0.528	288
UHT RCB		36.10	1.14	14.87	0.29	0.17	17.25	0.73	0.33	226	3.13	2.79	30.00	3.37	0.532	288
RCB	SMP3, 75 °C, 11 s	39.70	1.06	13.42	0.46	0.15	17.22	1.54	0.32	221	3.40	2.17	36.75	3.38	0.528	288
UHT RCB		33.40	1.36	15.40	0.27	0.17	17.11	0.76	0.26	219	7.49	2.54	36.49	3.37	0.526	288
Recon.	75 °C, 11 s	45.80	0.81	13.99	0.58	0.14	17.59	1.38	0.32	230	1.73	2.80	26.35	3.31	0.523	288
UHT Recon.		46.40	0.91	13.90	0.42	0.16	17.57	1.35	0.31	230	1.73	2.62	26.81	3.34	0.523	288
Recon.	95 °C, 147 s	46.20	0.83	13.99	0.64	0.14	17.10	1.46	0.35	233	1.76	2.66	24.44	3.41	0.524	288
UHT Recon.		45.10	1.00	13.26	0.53	0.16	17.72	1.48	0.28	230	2.90	2.67	27.16	3.39	0.525	288

Table A3.2 Fat content of, and proportion of total sample fat in the cream layer of homogenized then preheated FWM (Milk No. 7).

Milk	Evap. preheat	UHT preheat treatment	% concentration of fat in cream layer as recovered				% proportion of total fat recovered in the cream layer			
			After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)	After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)
FWM	n/a	75 °C, 11 s	45.70	45.30	46.00	44.70	56	54	53	55
		95 °C, 147 s	47.00	45.00	44.00	42.90	52	50	53	54

Table A3.3 Fat content of, and proportion of total sample fat in the cream layer of homogenized then preheated FWM, RCB and Recon (Milk No. 8).

Milk	Evap. preheat	UHT preheat treatment	% concentration of fat in cream layer as recovered				% proportion of total fat recovered in the cream layer			
			After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)	After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)
FWM	n/a	75 °C, 11 s	45.5	44.4	45.0	44.7	55.3	54.5	53.6	50.0
		95 °C, 147 s	46.0	44.2	43.8	43.5	56.1	51.3	49.5	51.9
RCB	75 °C, 2 s	75 °C, 11 s	43.2	40.9	33.3	39.2	56.7	53.8	43.6	49.8
		95 °C, 147 s	44.5	42.1	35.7	43.4	54.2	54.3	45.5	52.5
Recon	95 °C, 33 s	75 °C, 11 s	45.3	44.5	37.7	45.9	53.5	53.1	45.1	54.2
		95 °C, 147 s	44.6	43.9	43.9	46.3	51.2	51.8	54.2	51.6

Table A3.4 Fat content of, and proportion of total sample fat in the cream layer of homogenized then preheated FWM (Milk No. 9).

Milk	UHT preheat treatment	% concentration of fat in cream layer as recovered				% proportion of total fat recovered in the cream layer			
		After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)	After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)
FWM	95 °C, 147 s	48.8	46.1	48.6	46.5	56.5	53.3	53.7	52.6

Table A3.5 Fat content of, and proportion of total sample fat in the cream layer of homogenized then preheated FWM (Milk No. 10).

Milk	Evap. preheat	UHT preheat treatment	% concentration of fat in cream layer as recovered				% proportion of total fat recovered in the cream layer			
			After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)	After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)
FWM	n/a	75 °C, 11 s	47.2	46.8	44.1	44.6	54.6	53.6	52.2	53.6
		95 °C, 147 s	48.0	46.6	46.8	46.8	56.8	54.1	52.4	53.3

Table A3.6 Fat content of, and proportion of total sample fat in the cream layer of homogenized then preheated RCB and Recon (Milk No. 2b).

Milk	Evap. preheat	UHT preheat treatment	% concentration of fat in cream layer as recovered				% proportion of total fat recovered in the cream layer			
			After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)	After prepa-ration (4 °C)	After preheat (75 or 95 °C)	After inter. heating (126 °C)	After UHT (140 °C)
RCB	75 °C, 2 s	75 °C, 11 s	46.2	46.0	45.0	33.4	56.4	57.2	57.1	42.0
		95 °C, 147 s	44.6	44.6	44.9	43.5	56.6	57.5	56.1	56.1
	85 °C, 155 s	75 °C, 11 s	41.7	30.9	40.2	36.1	57.0	41.9	55.1	53.0
	95 °C, 155 s	75 °C, 11 s	39.7	37.9	43.4	33.4	46.8	44.4	48.3	50.4
Recon	95 °C, 33 s	75 °C, 11 s	45.8	45.2	43.6	46.4	55.6	55.8	45.0	57.2
		95 °C, 147 s	46.2	47.6	47.4	45.1	56.9	57.8	47.7	53.2

Table A3.7 Estimation of surface area fraction and volume fraction from different sizes of fat globules in whole milks (Milk No. 2).

Fat globule size (μm)	Surface area (m^2)	Volume (m^3)	Surface area / volume	Volume fraction (from particle size distribution)			Volume fraction for specific size range[1]			Surface area fraction[2]			Cumulative surface area fraction[3]			Surface area fraction[4]		
				RCB (UHT PH (75 °C, 11 s)	FWM (UHT PH 95 °C, 147 s)	Recon (UHT PH 95 °C, 147 s)	RCB (UHT PH (75 °C, 11 s)	FWM (UHT PH 95 °C, 147 s)	Recon (UHT PH 95 °C, 147 s)	RCB (UHT PH (75 °C, 11 s)	FWM (UHT PH 95 °C, 147 s)	Recon (UHT PH 95 °C, 147 s)	RCB (UHT PH (75 °C, 11 s)	FWM (UHT PH 95 °C, 147 s)	Recon (UHT PH 95 °C, 147 s)	RCB (UHT PH (75°C, 11 s)	FWM (UHT PH 95°C, 147 s)	Recon (UHT PH 95°C, 147 s)
0.040	0.005	0.000	150	0.0012	0.0014	0.0010	0.0060	0.0069	0.0056	0.900	1.035	0.840	0.900	1.035	0.840	0.024	0.025	0.022
0.045	0.006	0.000	133	0.0072	0.0083	0.0066	0.0096	0.0110	0.0089	1.280	1.467	1.187	2.180	2.502	2.027	0.057	0.060	0.054
0.050	0.008	0.000	120	0.0168	0.0193	0.0155	0.0139	0.0158	0.0128	1.668	1.896	1.536	3.848	4.398	3.563	0.101	0.106	0.094
0.056	0.010	0.000	107	0.0307	0.0351	0.0283	0.0186	0.0213	0.0174	1.993	2.282	1.864	5.841	6.680	5.427	0.154	0.160	0.143
0.063	0.012	0.000	95	0.0493	0.0564	0.0457	0.0251	0.0285	0.0233	2.390	2.714	2.219	8.231	9.394	7.646	0.216	0.226	0.202
0.071	0.016	0.000	85	0.0744	0.0849	0.0690	0.0317	0.0361	0.0297	2.679	3.051	2.510	10.910	12.445	10.156	0.287	0.299	0.268
0.080	0.020	0.000	75	0.1061	0.1210	0.0987	0.0379	0.0432	0.0357	2.843	3.240	2.678	13.753	15.685	12.833	0.361	0.377	0.339
0.089	0.025	0.000	67	0.1440	0.1642	0.1344	0.0434	0.0492	0.0411	2.926	3.317	2.771	16.679	19.002	15.604	0.438	0.456	0.412
0.100	0.031	0.001	60	0.1874	0.2134	0.1755	0.0475	0.0539	0.0456	2.850	3.234	2.736	19.529	22.236	18.340	0.513	0.534	0.485
0.112	0.039	0.001	54	0.2349	0.2673	0.2211	0.0506	0.0572	0.0490	2.711	3.064	2.625	22.239	25.300	20.965	0.584	0.607	0.554
0.126	0.050	0.001	48	0.2855	0.3245	0.2701	0.0523	0.0587	0.0515	2.490	2.795	2.452	24.730	28.095	23.418	0.650	0.674	0.619
0.142	0.063	0.001	42	0.3378	0.3832	0.3216	0.0524	0.0586	0.0526	2.214	2.476	2.223	26.944	30.571	25.640	0.708	0.734	0.678
0.159	0.079	0.002	38	0.3902	0.4418	0.3742	0.0508	0.0564	0.0524	1.917	2.128	1.977	28.861	32.700	27.617	0.759	0.785	0.730
0.178	0.100	0.003	34	**0.4410**	0.4982	0.4266	0.0480	0.0526	0.0510	1.618	1.773	1.719	30.479	34.473	29.337	**0.801**	0.828	0.775
0.200	0.126	0.004	30	**0.4890**	0.5508	0.4776	0.0436	0.0471	0.0483	1.308	1.413	1.449	31.787	35.886	30.786	**0.835**	0.861	0.813
0.224	0.158	0.006	27	0.5326	0.5979	**0.5259**	0.0385	0.0408	0.0450	1.031	1.093	1.205	32.818	36.978	31.991	0.863	0.888	**0.845**
0.252	0.200	0.008	24	0.5711	0.6387	0.5709	0.0335	0.0343	0.0413	0.798	0.817	0.983	33.616	37.795	32.974	0.883	0.907	0.871
0.283	0.252	0.012	21	0.6046	0.6730	0.6122	0.0292	0.0288	0.0381	0.619	0.611	0.808	34.235	38.406	33.782	0.900	0.922	0.893
0.317	0.316	0.017	19	0.6338	0.7018	0.6503	0.0266	0.0251	0.0357	0.503	0.475	0.676	34.738	38.881	34.458	0.913	0.933	0.911
0.356	0.398	0.024	17	0.6604	0.7269	0.6860	0.0261	0.0237	0.0345	0.440	0.399	0.581	35.178	39.280	35.039	0.925	0.943	0.926
0.399	0.500	0.033	15	0.6865	0.7506	0.7205	0.0271	0.0243	0.0345	0.408	0.365	0.519	35.586	39.646	35.558	0.935	0.952	0.940
0.448	0.631	0.047	13	0.7136	0.7749	0.7550	0.0294	0.0258	0.0347	0.394	0.346	0.465	35.979	39.991	36.023	0.946	0.960	0.952
0.502	0.792	0.066	12	0.7430	0.8007	0.7897	0.0315	0.0277	0.0347	0.376	0.331	0.415	36.356	40.322	36.437	0.955	0.968	0.963
0.564	0.999	0.094	11	0.7745	0.8284	0.8244	0.0331	0.0288	0.0337	0.352	0.306	0.359	36.708	40.629	36.796	0.965	0.975	0.972
0.632	1.255	0.132	9	0.8076	0.8572	0.8581	0.0332	0.0284	0.0315	0.315	0.270	0.299	37.023	40.898	37.095	0.973	0.982	0.980
0.710	1.584	0.187	8	0.8408	0.8856	0.8896	0.0316	0.0262	0.0279	0.267	0.221	0.236	37.290	41.120	37.331	0.980	0.987	0.986
0.796	1.991	0.264	8	0.8724	0.9118	0.9175	0.0288	0.0228	0.0234	0.217	0.172	0.176	37.507	41.292	37.507	0.986	0.991	0.991
0.893	2.505	0.373	7	0.9012	0.9346	0.9409	0.0253	0.0189	0.0189	0.170	0.127	0.127	37.677	41.418	37.634	0.990	0.994	0.994

1.002	3.154	0.527	6	0.9265	0.9535	0.9598	0.0218	0.0152	0.0146	0.131	0.091	0.087	37.808	41.510	37.722	0.994	0.996	0.997
1.125	3.976	0.746	5	0.9483	0.9687	0.9744	0.0182	0.0118	0.0109	0.097	0.063	0.058	37.905	41.572	37.780	0.996	0.998	0.998
1.262	5.003	1.052	5	0.9665	0.9805	0.9853	0.0142	0.0084	0.0074	0.068	0.040	0.035	37.972	41.612	37.815	0.998	0.999	0.999
1.416	6.299	1.487	4	0.9807	0.9889	0.9927	0.0105	0.0054	0.0044	0.044	0.023	0.019	38.017	41.635	37.834	0.999	1.000	1.000
1.589	7.932	2.101	4	0.9912	0.9943	0.9971	0.0073	0.0032	0.0025	0.028	0.012	0.009	38.044	41.647	37.843	1.000	1.000	1.000
1.783	9.987	2.968	3	0.9985	0.9975	0.9996	0.0015	0.0017	0.0004	0.005	0.006	0.001	38.050	41.653	37.844	1.000	1.000	1.000
2.000	12.566	4.189	3	1.0000	0.9992	1.0000		0.0008			0.002			41.655		1.000	1.000	1.000
2.244			3		1.0000												1.000	

[1]Volume fraction for specific size range = Volume fraction for a larger size of fat globule - volume fraction for a smaller size of fat globule (e.g. 0.0072 – 0.0012 = 0.0060), where 0.006 is the volume fraction represented by fat globules between 0.040 and 0.045 μm.

[2]Surface area fraction = (Surface area / volume) × volume fraction for specific size range (e.g. 150 × 0.0060 = 0.900).

[3]Cumulative surface area fraction = Surface area fraction for a larger size of fat globule – surface area fraction for a smaller size of fat globule (e.g. 0.900 + 1.28 = 2.180).

[4]Surface area fraction = Cumulative surface area at a given fat globule size range / total cumulative surface area (e.g. 0.900 / 38.05 = 0.024).

The recovered fat in the cream layer, which ranged from 0.42 to 0.58 of the total fat in the milk sample, comprises the largest globules in the sample. Therefore, the unrecovered fat in the top-serum, serum and sediment, which constitutes 0.58-0.42 of the total fat, comprises the smaller fat globules. Three critical fat globule sizes, within the volume fraction range 0.58-0.42, were chosen to represent the unrecovered fat. These sizes were 0.18, 0.20 and 0.22 μm.

For each size, the total fat-bound protein in the original milk sample was estimated as in the following sample:

Critical size = 0.20 μm

Fraction of total fat that was in the cream layer = 0.51

Therefore, fraction of total fat that was unrecovered = 1 – 0.51 = 0.49

From Figure 6.3, the fraction of total fat surface area corresponding to a volume fraction of 0.49 is 0.84. Therefore, the fraction of total surface area corresponding to the recovered fat = 1 – 0.84 = 0.16.

Thus, for a measured fat-bound protein in the cream layer of 0.1 g / g fat, the estimated total fat-bound protein in the milk sample, using equation (6-1).

= (0.51 × 0.1) / 0.16

= 0.32 g / g fat

Table A3.8: Results for Past. and raw FWM
General Linear Model: log (FBP +1) versus Milk No. and fresh milk

```
Factor      Type   Levels  Values
Milk No.    fixed       2  9, 10
Fresh milk  fixed       2  Past. FM, Raw FM

Analysis of Variance for log (FBP +1), using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS     F      P
Milk No.     1  0.0006541  0.0006541  0.0006541  1.00  0.500
Fresh milk   1  0.0050885  0.0050885  0.0050885  7.78  0.219
Error        1  0.0006541  0.0006541  0.0006541
Total        3  0.0063968

S = 0.0255763   R-Sq = 89.77%   R-Sq(adj) = 69.32%
```

Table A3.9: Analysis of Split-plot design for the effects of milk preparation, UHT preheat treatment and milk batch

The statistical result below is the result of the Split-plot analysis for the total fat-bound protein in FWM, RCB and Recon.

Results for: homog.-preheat FWM SMP1 WMP
General Linear Model: log (FBP +1) versus Milk No. , Milk preparation and UHT PH

```
Factor            Type   Levels  Values
Milk No.          fixed       5  7, 8, 9, 10, 2b
Milk preparation  fixed       3  comWMP, FM, SMP1
UHT PH            fixed       2  75 11, 95147


Analysis of Variance for log (FBP +1), using Adjusted SS for Tests

Source                   DF    Seq SS    Adj SS    Adj MS      F      P
Milk No.                  4  0.015734  0.015625  0.003906   2.29  0.073
Milk preparation          2  0.103635  0.103635  0.051817  30.36  0.000
UHT PH                    1  0.000464  0.000813  0.000813   0.48  0.493
Milk preparation*UHT PH   2  0.001903  0.001903  0.000952   0.56  0.576
Error                    50  0.085330  0.085330  0.001707
Total                    59  0.207067

S = 0.0413111   R-Sq = 58.79%   R-Sq(adj) = 51.37%
```

P-values for the effects of Milk No. and milk preparation are 0.073 and <0.001, respectively. They are directly reported as shown in Table 6.5. P-value of the effect of UHT preheat treatment within milk preparation can be calculated from the combined effect of UHT PH and the effect of an "interaction" of milk preparation * UHT PH for a new F-value.

$$F = \frac{(MS\ of\ UHT\ PH) + (MS\ of\ the\ "interaction"\ of\ milk\ preparation \times UHT\ PH)}{MS\ of\ error}$$

$$F = \frac{0.000813 + 0.000952}{0.001707}$$

$$F_{calculated} = 1.03$$

The new degree of freedom was calculated by the addition of the degree of freedom from the effect of UHT preheat treatment (1) and the interaction between the milk preparation and the UHT preheat treatment (2). Thus, the new degree of freedom is equal to 3.

From the table of F-distribution,

$$F_{table}\ (3, 50) = 2.79$$

Thus, $F_{calculated}$ (1.03) < F_{table} (2.79)

From the F-Table, the p-value of this new $F_{calculated}$ is 0.387, which represents the p-value of the effect of UHT preheat treatment within milk preparations.

Table A3.10: Results for FWM, RCB and Recon
General Linear Model: log (Alphas c, log (β-casein, ... versus Milk No., Milk preparation and UHT PH

```
Factor            Type   Levels  Values
Milk No.          fixed       5  7, 8, 9, 10, 2b
Milk preparation  fixed       3  comWMP, FM, SMP1
UHT PH            fixed       2  75 11, 95147

Analysis of Variance for log(Alphas casein/fat fract.+1), using Adjusted SS for
     Tests

Source                   DF   Seq SS   Adj SS   Adj MS     F      P
Milk No.                  4  0.78753  0.64577  0.16144  2.29  0.072
Milk preparation          2  0.23602  0.23602  0.11801  1.68  0.197
UHT PH                    1  0.00943  0.01252  0.01252  0.18  0.675
Milk preparation*UHT PH   2  0.01343  0.01343  0.00672  0.10  0.909
Error                    50  3.52077  3.52077  0.07042
Total                    59  4.56718

S = 0.265359   R-Sq = 22.91%   R-Sq(adj) = 9.04%
Analysis of Variance for log(β-casein/fat fract.+1), using Adjusted SS for
     Tests

Source                   DF    Seq SS    Adj SS    Adj MS      F      P
Milk No.                  4  0.396817  0.384564  0.096141  19.85  0.000
Milk preparation          2  0.201118  0.201118  0.100559  20.76  0.000
UHT PH                    1  0.015666  0.021939  0.021939   4.53  0.038
Milk preparation*UHT PH   2  0.019047  0.019047  0.009524   1.97  0.151
Error                    50  0.242171  0.242171  0.004843
Total                    59  0.874819

S = 0.0695947   R-Sq = 72.32%   R-Sq(adj) = 67.33%

Analysis of Variance for log(κ-casein/fat fract.+1), using Adjusted SS for
     Tests

Source                   DF    Seq SS    Adj SS    Adj MS      F      P
Milk No.                  4  0.547793  0.506832  0.126708  41.16  0.000
Milk preparation          2  0.001105  0.001105  0.000552   0.18  0.836
UHT PH                    1  0.003058  0.006074  0.006074   1.97  0.166
Milk preparation*UHT PH   2  0.017642  0.017642  0.008821   2.87  0.066
Error                    50  0.153910  0.153910  0.003078
Total                    59  0.723508

S = 0.0554816   R-Sq = 78.73%   R-Sq(adj) = 74.90%

Analysis of Variance for log(γ-casein/fat fract.+1), using Adjusted SS for
     Tests

Source                   DF   Seq SS   Adj SS   Adj MS      F      P
Milk No.                  4  0.46563  0.27916  0.06979   2.71  0.041
Milk preparation          2  0.71440  0.71440  0.35720  13.86  0.000
UHT PH                    1  0.03704  0.05880  0.05880   2.28  0.137
Milk preparation*UHT PH   2  0.14626  0.14626  0.07313   2.84  0.068
Error                    50  1.28896  1.28896  0.02578
Total                    59  2.65228

S = 0.160559   R-Sq = 51.40%   R-Sq(adj) = 42.65%
```

Analysis of Variance for log(Total casein/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	4	0.118300	0.117777	0.029444	8.12	0.000
Milk preparation	2	0.172137	0.172137	0.086069	23.74	0.000
UHT PH	1	0.009093	0.013440	0.013440	3.71	0.060
Milk preparation*UHT PH	2	0.017388	0.017388	0.008694	2.40	0.101
Error	50	0.181305	0.181305	0.003626		
Total	59	0.498223				

S = 0.0602171 R-Sq = 63.61% R-Sq(adj) = 57.06%

Analysis of Variance for log(β-lg/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	4	0.13995	0.06102	0.01525	0.31	0.873
Milk preparation	2	0.33156	0.33156	0.16578	3.32	0.044
UHT PH	1	0.56394	0.57344	0.57344	11.48	0.001
Milk preparation*UHT PH	2	0.09207	0.09207	0.04604	0.92	0.404
Error	50	2.49722	2.49722	0.04994		
Total	59	3.62474				

S = 0.223482 R-Sq = 31.11% R-Sq(adj) = 18.71%

Analysis of Variance for log(α-la/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	4	0.51770	0.41102	0.10275	2.31	0.071
Milk preparation	2	0.64314	0.64314	0.32157	7.22	0.002
UHT PH	1	0.50291	0.49067	0.49067	11.02	0.002
Milk preparation*UHT PH	2	0.01035	0.01035	0.00517	0.12	0.891
Error	50	2.22645	2.22645	0.04453		
Total	59	3.90055				

S = 0.211019 R-Sq = 42.92% R-Sq(adj) = 32.65%

Analysis of Variance for log(Total WP/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	4	0.18549	0.09958	0.02490	0.52	0.721
Milk preparation	2	0.37788	0.37788	0.18894	3.95	0.026
UHT PH	1	0.55602	0.56168	0.56168	11.74	0.001
Milk preparation*UHT PH	2	0.07047	0.07047	0.03523	0.74	0.484
Error	50	2.39235	2.39235	0.04785		
Total	59	3.58220				

S = 0.218739 R-Sq = 33.22% R-Sq(adj) = 21.19%

Analysis of Variance for log(Total protein/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	4	0.082669	0.086157	0.021539	5.81	0.001
Milk preparation	2	0.090325	0.090325	0.045162	12.18	0.000
UHT PH	1	0.032071	0.038300	0.038300	10.33	0.002
Milk preparation*UHT PH	2	0.012282	0.012282	0.006141	1.66	0.201
Error	50	0.185426	0.185426	0.003709		
Total	59	0.402773				

S = 0.0608976 R-Sq = 53.96% R-Sq(adj) = 45.68%

Table A3.11: Results for homogenized then preheated FWM
General Linear Model: log (FBP +1) versus Milk No., UHT PH and process stage

```
Factor         Type   Levels  Values
Milk No.       fixed       4  7, 8, 9, 10
UHT PH         fixed       2  75 11, 95147
Process stage  fixed       4  Homopreheated fresh milk, Inter heated fresh
                              milk, Past&homo fresh milk, UHT fresh milk

Analysis of Variance for log (FBP +1), using Adjusted SS for Tests

Source                DF     Seq SS     Adj SS     Adj MS      F      P
Milk No.               3  0.0064730  0.0065162  0.0021721  10.33  0.000
UHT PH                 1  0.0002353  0.0002353  0.0002353   1.12  0.305
Process stage          3  0.0101031  0.0100008  0.0033336  15.86  0.000
UHT PH*process stage   3  0.0004163  0.0004163  0.0001388   0.66  0.588
Error                 17  0.0035743  0.0035743  0.0002103
Total                 27  0.0208019

S = 0.0145001   R-Sq = 82.82%   R-Sq(adj) = 72.71%
```

P-values for the effect of Milk No. and UHT preheat treatment are < 0.001 and 0.305. The p-value for the effect of heating process stage within UHT preheat treatment is combined between the effect of heating process stage and the "interaction" of heating process stage × UHT preheat treatment.

Thus, the new F-value can be calculated from;

$$F = \frac{(MS\ of\ UHT\ PH) + (MS\ of\ the\ "interaction"\ of\ milk\ preparation \times UHT\ PH)}{MS\ of\ error}$$

$$F = \frac{0.0033336 + 0.0001388}{0.0002103}$$

$$F_{calculated} = 16.52$$

The new degree of freedom for the effect of heating process stage within UHT preheat treatment is the addition of the degree of freedom from the effect of heating process stage (3) and the degree of freedom from an interaction of UHT PH and heating process stage (3). Thus, the new degree of freedom for the effect UHT PH within heating process stage is 6. The degree of freedom of the error term is 17.

Thus, the F_{table} is

$$F_{table}\ (6,17) = 2.699$$

$$F_{calculated}\ (16.52) > F_{table}\ (2.699)$$

From the table of F-distribution, the new p-value is < 0.001.

Table A3.12: Results for homog.-preheated FWM
General Linear Model: log (Alphas c, log (β-casein, ... versus Milk No., UHT PH and process stage

```
Factor         Type   Levels  Values
Milk No.       fixed       4  7, 8, 9, 10
UHT PH         fixed       2  75 11, 95147
Process stage  fixed       4  FL of homopreheated fresh milk, FL of inter
                              heated fresh milk, FL of past and homo. fresh
                              milk, FL of UHT fresh milk

Analysis of Variance for log(Alphas casein/fat fract.+1), using Adjusted SS for
     Tests

Source                DF  Seq SS  Adj SS  Adj MS     F      P
Milk No.               3  0.6009  0.5973  0.1991  1.41  0.274
UHT PH                 1  0.0000  0.0000  0.0000  0.00  0.995
Process stage          3  0.9739  1.0000  0.3333  2.36  0.107
UHT PH*Process stage   3  0.0275  0.0275  0.0092  0.07  0.978
Error                 17  2.3982  2.3982  0.1411
Total                 27  4.0005

S = 0.375595   R-Sq = 40.05%   R-Sq(adj) = 4.79%

Analysis of Variance for log(β-casein/fat fract.+1), using Adjusted SS for
     Tests

Source                DF    Seq SS    Adj SS    Adj MS     F      P
Milk No.               3  0.116610  0.102094  0.034031  7.81  0.002
UHT PH                 1  0.000357  0.000357  0.000357  0.08  0.778
Process stage          3  0.034085  0.031589  0.010530  2.42  0.102
UHT PH*Process stage   3  0.006963  0.006963  0.002321  0.53  0.666
Error                 17  0.074054  0.074054  0.004356
Total                 27  0.232069

S = 0.0660008   R-Sq = 68.09%   R-Sq(adj) = 49.32%

Analysis of Variance for log(κ-casein/fat fract.+1), using Adjusted SS for
     Tests

Source                DF    Seq SS    Adj SS    Adj MS       F      P
Milk No.               3  0.502095  0.505391  0.168464  195.15  0.000
UHT PH                 1  0.003404  0.003404  0.003404    3.94  0.063
Process stage          3  0.036533  0.036983  0.012328   14.28  0.000
UHT PH*Process stage   3  0.015053  0.015053  0.005018    5.81  0.006
Error                 17  0.014675  0.014675  0.000863
Total                 27  0.571760

S = 0.0293808   R-Sq = 97.43%   R-Sq(adj) = 95.92%

Analysis of Variance for log(γ-casein/fat fract.+1), using Adjusted SS for
     Tests

Source                DF   Seq SS   Adj SS   Adj MS      F      P
Milk No.               3  0.25174  0.25455  0.08485  31.92  0.000
UHT PH                 1  0.00774  0.00774  0.00774   2.91  0.106
Process stage          3  0.60889  0.59402  0.19801  74.49  0.000
UHT PH*Process stage   3  0.15719  0.15719  0.05240  19.71  0.000
Error                 17  0.04519  0.04519  0.00266
Total                 27  1.07075

S = 0.0515583   R-Sq = 95.78%   R-Sq(adj) = 93.30%
```

Analysis of Variance for log(Total casein/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	3	0.020857	0.019086	0.006362	1.77	0.191
UHT PH	1	0.000715	0.000715	0.000715	0.20	0.661
Process stage	3	0.019266	0.018911	0.006304	1.75	0.194
UHT PH*Process stage	3	0.000775	0.000775	0.000258	0.07	0.974
Error	17	0.061138	0.061138	0.003596		
Total	27	0.102751				

S = 0.0599694 R-Sq = 40.50% R-Sq(adj) = 5.50%

Analysis of Variance for log(β-lg/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	3	0.12204	0.06095	0.02032	7.80	0.002
UHT PH	1	0.16999	0.16999	0.16999	65.31	0.000
Process stage	3	1.21698	1.15003	0.38334	147.27	0.000
UHT PH*Process stage	3	0.20974	0.20974	0.06991	26.86	0.000
Error	17	0.04425	0.04425	0.00260		
Total	27	1.76301				

S = 0.0510196 R-Sq = 97.49% R-Sq(adj) = 96.01%

Analysis of Variance for log(α-la/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	3	0.41333	0.30770	0.10257	20.33	0.000
UHT PH	1	0.20061	0.20061	0.20061	39.75	0.000
Process stage	3	1.08601	1.08656	0.36219	71.77	0.000
UHT PH*Process stage	3	0.18738	0.18738	0.06246	12.38	0.000
Error	17	0.08579	0.08579	0.00505		
Total	27	1.97311				

S = 0.0710365 R-Sq = 95.65% R-Sq(adj) = 93.09%

Analysis of Variance for log(Total WP/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	3	0.16922	0.09683	0.03228	15.02	0.000
UHT PH	1	0.17576	0.17576	0.17576	81.77	0.000
Process stage	3	1.18671	1.13453	0.37818	175.95	0.000
UHT PH*Process stage	3	0.20345	0.20345	0.06782	31.55	0.000
Error	17	0.03654	0.03654	0.00215		
Total	27	1.77168				

S = 0.0463615 R-Sq = 97.94% R-Sq(adj) = 96.72%

Analysis of Variance for log(Total protein/fat fract.+1), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	3	0.013901	0.014467	0.004822	1.81	0.183
UHT PH	1	0.001432	0.001432	0.001432	0.54	0.473
Process stage	3	0.024771	0.022770	0.007590	2.85	0.068
UHT PH*Process stage	3	0.003795	0.003795	0.001265	0.48	0.703
Error	17	0.045218	0.045218	0.002660		
Total	27	0.089118				

S = 0.0515741 R-Sq = 49.26% R-Sq(adj) = 19.41%

Appendix 4

Appendix A4.1 Results for homog. then preheat FWM RCB Recon

The main plot is milk preparation and the sub plot is UHT preheat treatment.

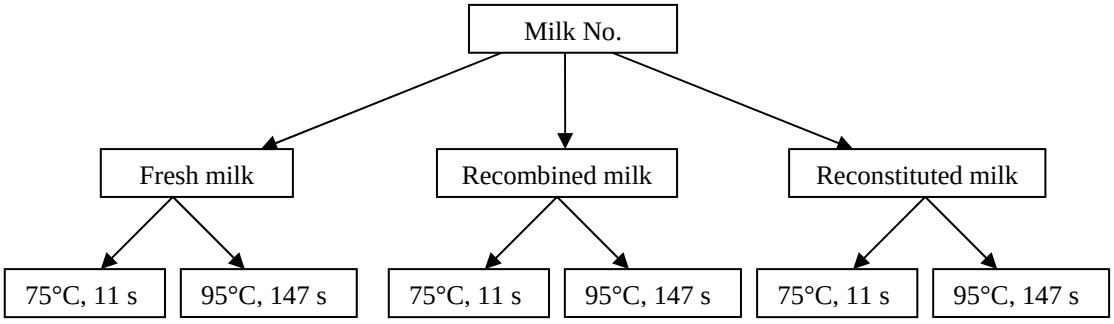

Figure A4.1 Split-plot design with replicates for analysing the effects of milk preparation and UHT preheat treatment on total deposit weight and deposit component weights for FWM, RCB and Recon. (Milk Nos. 8-10 and 2b).

The statistical result below is the result of the split-plot analysis for the total weight of deposit for FWM, RCB and Recon.

General Linear Model: Log deposit , log fat, ... versus Milk No., Milk preparation and UHT PH

```
Factor            Type   Levels  Values
Milk No.          fixed       3  8, 10, 2b
Milk preparation  fixed       3  Fresh milk, Recom. milk, Reconst. milk
UHT PH            fixed       2  75 11, 95147

Analysis of Variance for Log deposit weight, using Adjusted SS for Tests

Source                   DF    Seq SS    Adj SS    Adj MS       F      P
Milk No.                  2  0.033339  0.032581  0.016291    5.56  0.070
Milk preparation          2  0.038884  0.038884  0.019442    6.64  0.054
UHT PH                    1  0.486241  0.486241  0.486241  166.09  0.000
Milk preparation*UHT PH   2  0.035424  0.035424  0.017712    6.05  0.062
Error                     4  0.011710  0.011710  0.002928
Total                    11  0.605597

S = 0.0541066   R-Sq = 98.07%   R-Sq(adj) = 94.68%
```

P-values for the effects of Milk No. and milk preparation are 0.070 and 0.054, respectively. They are directly reported as shown in Table 7.2. The p-value for the effect of UHT preheat treatment within milk preparation can be calculated from the combined effect of UHT PH and the effect of the “interaction” milk preparation * UHT PH, to give a new F-value.

$$F = \frac{(MS\ of\ UHT\ PH) + (MS\ of\ the\ "interaction"\ of\ milk\ preparation * UHT\ PH)}{MS\ of\ error}$$

$$F = \frac{0.486241 + 0.017712}{0.002928}$$

$$F_{calculated} = 172.12$$

The new degrees of freedom were calculated by summing the degrees of freedom of the effect of UHT preheat treatment (1) and of the interaction between milk preparation and UHT preheat treatment (2). Thus, the new degrees of freedom are equal to 3.

From the table of F-distribution,

$$F_{table}\ (3, 4) = 6.59$$

Thus, $F_{calculated}\ (172.12) > F_{table}\ (6.59)$

The p-value of this new $F_{calculated}$ is < 0.001, which represents the p-value of the effect of UHT preheat treatment within milk preparation.

```
Analysis of Variance for log fat, using Adjusted SS for Tests

Source                   DF   Seq SS   Adj SS   Adj MS      F      P
Milk No.                  2  0.20541  0.18100  0.09050   1.46  0.335
Milk preparation          2  0.00369  0.00369  0.00184   0.03  0.971
UHT PH                    1  2.81975  2.81975  2.81975  45.34  0.003
Milk preparation*UHT PH   2  0.13681  0.13681  0.06840   1.10  0.416
Error                     4  0.24879  0.24879  0.06220
Total                    11  3.41444

S = 0.249395   R-Sq = 92.71%   R-Sq(adj) = 79.96%

Analysis of Variance for log protein, using Adjusted SS for Tests

Source                   DF   Seq SS   Adj SS   Adj MS       F      P
Milk No.                  2  0.07362  0.07441  0.03720   10.83  0.024
Milk preparation          2  0.05756  0.05756  0.02878    8.38  0.037
UHT PH                    1  0.65986  0.65986  0.65986  192.03  0.000
Milk preparation*UHT PH   2  0.03210  0.03210  0.01605    4.67  0.090
Error                     4  0.01374  0.01374  0.00344
Total                    11  0.83687

S = 0.0586189   R-Sq = 98.36%   R-Sq(adj) = 95.48%

Analysis of Variance for log ash, using Adjusted SS for Tests

Source                   DF    Seq SS    Adj SS    Adj MS       F      P
Milk No.                  2  0.040001  0.018661  0.009331    6.00  0.062
Milk preparation          2  0.036233  0.036233  0.018117   11.66  0.021
UHT PH                    1  0.248872  0.248872  0.248872  160.15  0.000
Milk preparation*UHT PH   2  0.048711  0.048711  0.024356   15.67  0.013
Error                     4  0.006216  0.006216  0.001554
Total                    11  0.380033

S = 0.0394211   R-Sq = 98.36%   R-Sq(adj) = 95.50%
```

Analysis of Variance for log Lactose, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	2	0.9968	1.1800	0.5900	1.57	0.314
Milk preparation	2	1.1140	1.1140	0.5570	1.48	0.330
UHT PH	1	5.6731	5.6731	5.6731	15.10	0.018
Milk preparation*UHT PH	2	0.0329	0.0329	0.0165	0.04	0.958
Error	4	1.5031	1.5031	0.3758		
Total	11	9.3200				

S = 0.613010 R-Sq = 83.87% R-Sq(adj) = 55.65%

General Linear Model: log PO4, log TP, ... versus Milk No., Milk preparation and UHT PH

Factor	Type	Levels	Values
Milk No.	fixed	3	8, 10, 2b
Milk preparation	fixed	3	Fresh milk, Recom. milk, Reconst. milk
UHT PH	fixed	2	75 11, 95147

Analysis of Variance for log PO4, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	2	0.059548	0.002099	0.001049	0.13	0.885
Milk preparation	2	0.003324	0.037524	0.018762	2.28	0.250
UHT PH	1	0.358985	0.365803	0.365803	44.40	0.007
Milk preparation*UHT PH	2	0.021555	0.021555	0.010778	1.31	0.390
Error	3	0.024715	0.024715	0.008238		
Total	10	0.468128				

S = 0.0907651 R-Sq = 94.72% R-Sq(adj) = 82.40%

Analysis of Variance for log TP, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	2	0.069015	0.015943	0.007971	6.67	0.079
Milk preparation	2	0.000990	0.010715	0.005357	4.48	0.126
UHT PH	1	0.182446	0.195914	0.195914	163.95	0.001
Milk preparation*UHT PH	2	0.031520	0.031520	0.015760	13.19	0.033
Error	3	0.003585	0.003585	0.001195		
Total	10	0.287556				

S = 0.0345684 R-Sq = 98.75% R-Sq(adj) = 95.84%

Analysis of Variance for log TCa, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Milk No.	2	0.061835	0.016751	0.008376	5.88	0.092
Milk preparation	2	0.002294	0.007572	0.003786	2.66	0.217
UHT PH	1	0.167790	0.180122	0.180122	126.45	0.002
Milk preparation*UHT PH	2	0.034113	0.034113	0.017056	11.97	0.037
Error	3	0.004273	0.004273	0.001424		
Total	10	0.270305				

S = 0.0377418 R-Sq = 98.42% R-Sq(adj) = 94.73%

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EFFECT OF PREHEATING ON FOULING OF A PILOT SCALE UHT STERILIZING PLANT BY RECOMBINED, RECONSTITUTED AND FRESH WHOLE MILKS

A. SRICHANTRA[1]*, D. F. NEWSTEAD[2], O. J. MCCARTHY[1] and A. H. J. PATERSON[3]

[1]*Institute of Food, Nutrition and Human Health, Massey University, Palmerston North, New Zealand*
[2]*Fonterra Co-operative Group Ltd, New Zealand*
[3]*Institute of Technology and Engineering, Massey University, Palmerston North, New Zealand*

The principal aim of this study was to confirm and extend previous findings on the effects of milk preheating on fouling of UHT (ultra-high temperature) sterilizing plants by recombined whole milks. A comprehensive replicated experiment was carried out to examine the effects of preheat treatment on fouling by milk recombined from skim milk powder (SMP) and milk fat, milk reconstituted from whole milk powder (WMP), and fresh milk. Within each replicate the SMP, the WMP and the fresh milk were all derived from the same batch of milk. Fouling rates were measured in the high-temperature section of a pilot-scale indirect UHT plant. The effects of preheating conditions were compared as follows: UHT preheating conditions (recombined and fresh milk): 75°C × 11 s, 85°C × 147 s, and 95°C × 147 s; evaporator preheating conditions for skim milk powder manufacture: 75°C × 2 s, 85°C × 155 s, and 95°C × 155 s. The evaporator preheating conditions in whole milk powder manufacture (95°C × 33 s) were not varied. In all cases it was found that the more severe was a preheat treatment, whether evaporator preheating or UHT preheating, the higher was the fouling rate. The results, which are discussed in relation to previous findings for both recombined milks and fresh milk, suggest that the preheating of previously homogenized whole milks exacerbates fouling.

Keywords: preheat; UHT; fouling; temperature difference; heat exchanger; whole milk.

INTRODUCTION

Fouling of heat transfer surfaces by milk, especially under the high temperature conditions existing in the final heating sections of indirectly heated ultra-high temperature (UHT) sterilizing plants, is an ongoing problem in the dairy industry. A number of researchers have reported that preheating fresh milk prior to UHT processing results in lower extents of fouling (Burton, 1968; Lalande *et al*., 1984; Patil and Reuter, 1986; Mottar and Moermans, 1988). In contrast, Newstead *et al*. (1999) found that preheating, both during powder manufacture (evaporator preheating) and immediately prior to UHT processing (UHT preheating), resulted in increased fouling rates for recombined whole milk (made from skim milk powder (SMP) and milk fat) and reconstituted whole milk [made from whole milk powder (WMP)]. Further, Newstead *et al*. (1999) found that the effects of UHT preheating on the rate of fouling by fresh milk were variable, and there was evidence of variation in behaviour between batches of milk.

The objectives of the work described here were to confirm and extend the findings of Newstead *et al*. (1999), to compare the relative effects of preheat treatment on the fouling rates of recombined, reconstituted and fresh whole milks, and to assess the relative impacts of evaporator and UHT preheating.

METHODS AND MATERIALS

Experimental Design

The experiment design (Figure 1) comprised three parts. The first concerned the effects of heat treating (i.e., preheating) fresh milk prior to UHT processing. The second concerned the effects of preheating reconstituted WMP prior to UHT processing. The third concerned the effects of both pre-evaporation heat treatment (evaporator preheating) during the manufacture of SMP, and preheating the recombined milk derived from it prior to UHT processing.

**Correspondence to*: Ms A. Srichantra, Institute of Food, Nutrition, and Human Health, Massey University, Private bag 11222, Palmerston North 5301, New Zealand.
E-mail: A.Srichantra@massey.ac.nz.

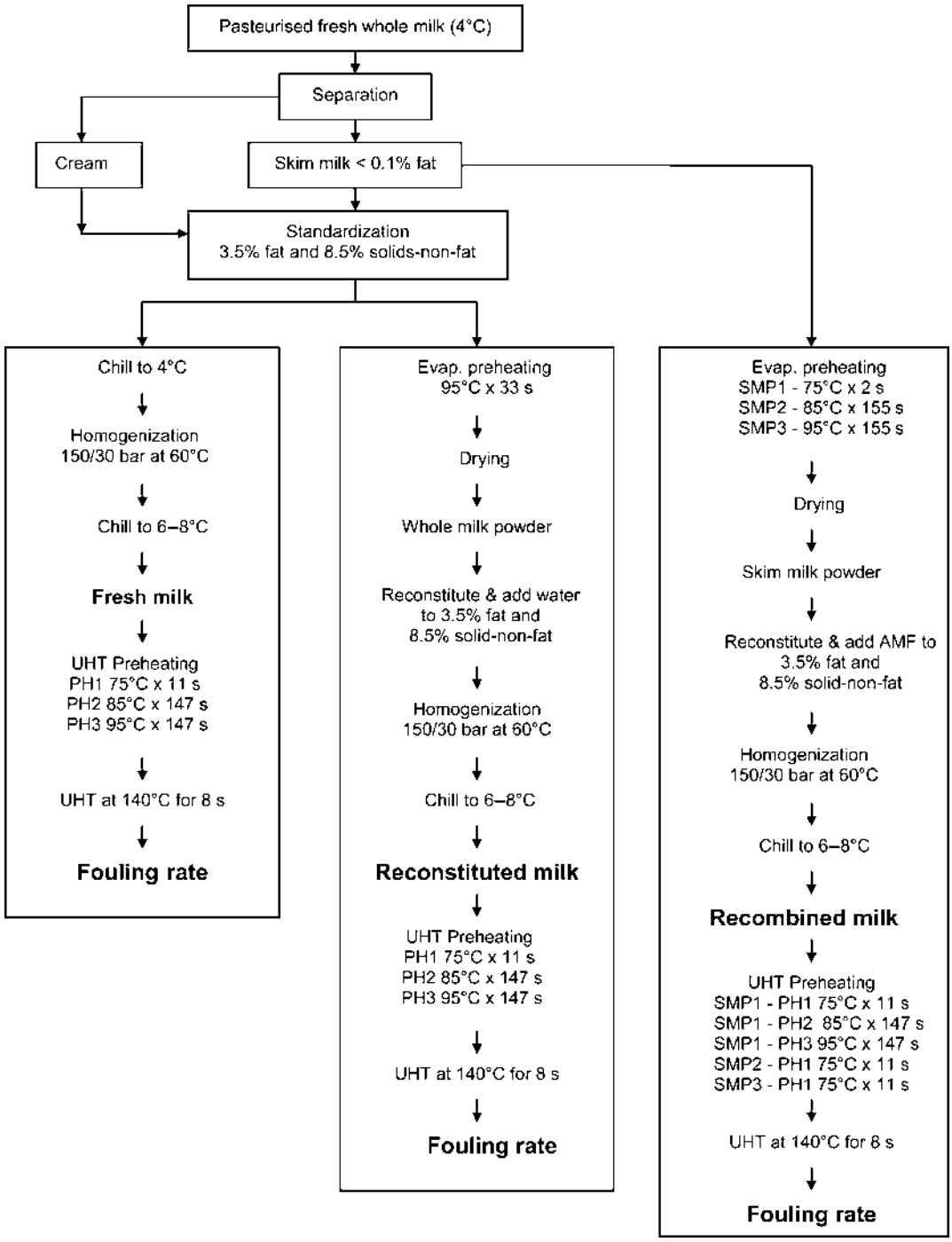

Figure 1. Experiment design.

Two complete replicates of this design (Figure 1) were carried out a month apart (replicate 1 in October and replicate 2 in November 2004). In each of the two replicate experiments the fresh milk, the SMP and the WMP were all derived from the same batch of milk. In each experiment, starting from a single batch of fresh pasteurised whole milk, UHT fouling measurements were made on the fresh milk using three different preheating conditions (PH1, 75°C × 11 s; PH2, 85°C × 147 s; PH3, 95°C × 147 s) prior to UHT sterilization, SMP was manufactured using three different evaporator preheating conditions (SMP1, 75°C × 2 s; SMP2, 85°C × 155 s; SMP3, 95°C × 155 s), and WMP was manufactured with one evaporator preheating condition only:[1] WMP, 95°C × 33 s. UHT-plant fouling rates were determined for recombined milk prepared from the SMP1 and milk fat, and for reconstituted milk made from the WMP, in each case for each of the three UHT preheating conditions.

[1]A moderate preheat treatment is always used in the manufacture of WMP to ensure adequate flavour stability. The preheat treatment applied in the present study was typical of commercial practice.

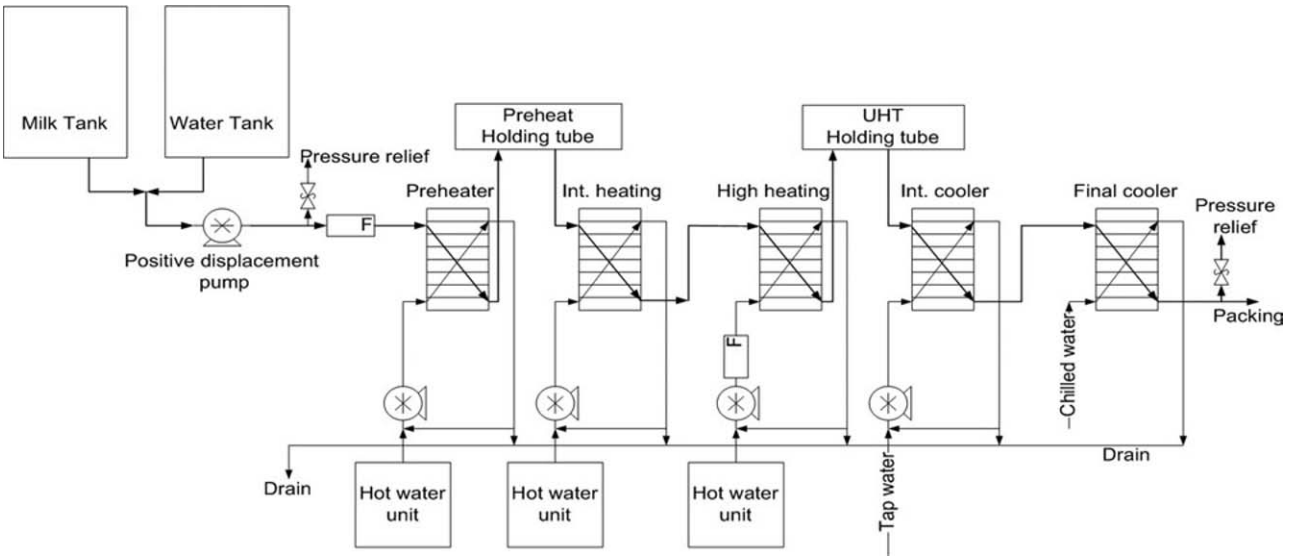

Figure 2. Simplified UHT plant diagram (F = flow meter).

Fouling rates were determined for recombined milk prepared from SMP2 and SMP3 for the UHT preheating condition PH1 only.

Fouling rate was evaluated as the rate of increase in the difference between the heating medium (hot water) inlet temperature and the milk outlet temperature (controlled to 140°C) in the high-temperature section of the UHT plant.

Milk Reception, Preparatory Processing and Fouling Experiments

Fresh raw whole milk was obtained from a single road-tanker collection area of the Fonterra Co-operative Group Ltd, New Zealand (Manawatu region). For each replicate (October and November), about 7000 L of raw fresh milk was pasteurized at 72°C for 15 s, separated into skim milk and cream, and chilled to 4°C. About 3400 L of skim milk was manufactured into (three batches of) SMP using the three evaporator preheating conditions listed above. Cream was added to the remaining 3600 L of skim milk to give standardized fresh whole milk with 3.5% fat and 8.5% solids non-fat. About 2200 L of this standardized milk was converted into whole milk powder. The remainder was used to determine UHT-plant fouling rates for each of the three UHT preheating conditions given above. Two fouling measurements were made immediately (on the same day) and two further measurements on the following day after overnight storage of the milk (at 4°C). On the second day, one of the preheating conditions was a repeat of one of the conditions used on the first day. Fouling measurements were made on the recombined SMP and reconstituted WMP, as outlined above, at later dates.

UHT Pilot Plant

The UHT pilot plant used, purpose-built for sub-commercial sample production and experimental work, had the following characteristics: working temperature of up to 150°C, temperature independently controllable in all three heating sections, variable holding tubes for preheating and for sterilization, and full instrumentation with data logging. The milk flow rate was controlled to 120 L h^{-1}. A schematic flow diagram is shown in Figure 2.

The heat exchanger (Alfa-Laval type Clip3 R, TetraPak, Sweden) comprised five sections: preheater, intermediate heater, high-heater, intermediate cooler and final cooler. In the first four sections, milk temperature was automatically controlled via dedicated heating or cooling water circuits. The final cooling section was supplied with chilled water. A detailed flow diagram of the first three sections is shown in Figure 3. Flow within the high temperature section of the plate heat exchanger was fully counter-current. Each section was equipped with temperature sensors to measure the inlet and outlet temperatures of the milk and water.

The length of the 120 s holding tube plus connecting pipework, located between the preheater and intermediate heater, was 48.32 m and the tube diameter was 0.9 cm. The milk Reynolds number in the holding tube was approximately 5279 and 6597 at 75°C and 95°C, respectively, and approximately 1694 in the flow passages of the high-temperature section of the plate heat exchanger.[2]

The plant was started on water, and switched to milk once temperature control had been established. The start of the fouling run was taken as the time (t_0) at which the milk, displacing the water, reached full concentration in the high-heater. Temperature and flow rate data were logged at 5 s intervals. The UHT preheat treatment under study was applied in the first, preheating, section and preheating holding tube. The milk temperature was then raised to 126°C by the intermediate heater, and fouling measured in the high-heater, in which the temperature

[2] $Re = D_e V\rho/\mu$, where D_e = equivalent diameter of plate flow passage = 0.003 m (from manufacturer); V = mean velocity in plate flow passage = [milk volumetric flow rate ($m^3\ s^{-1}$)/cross-sectional area for flow (flow manufacturer) (m^2)] = [$3.5 \times 10^{-5}\ m^3\ s^{-1}/0.00015\ m^2$] = 0.233 m s^{-1}; ρ and μ = milk density and viscosity, respectively, evaluated at the milk bulk mean temperature (133°C) in the high-heat section = 962 kg m^{-3} and 3.97×10^{-4} Pa s, respectively (data from Kessler, 2002).

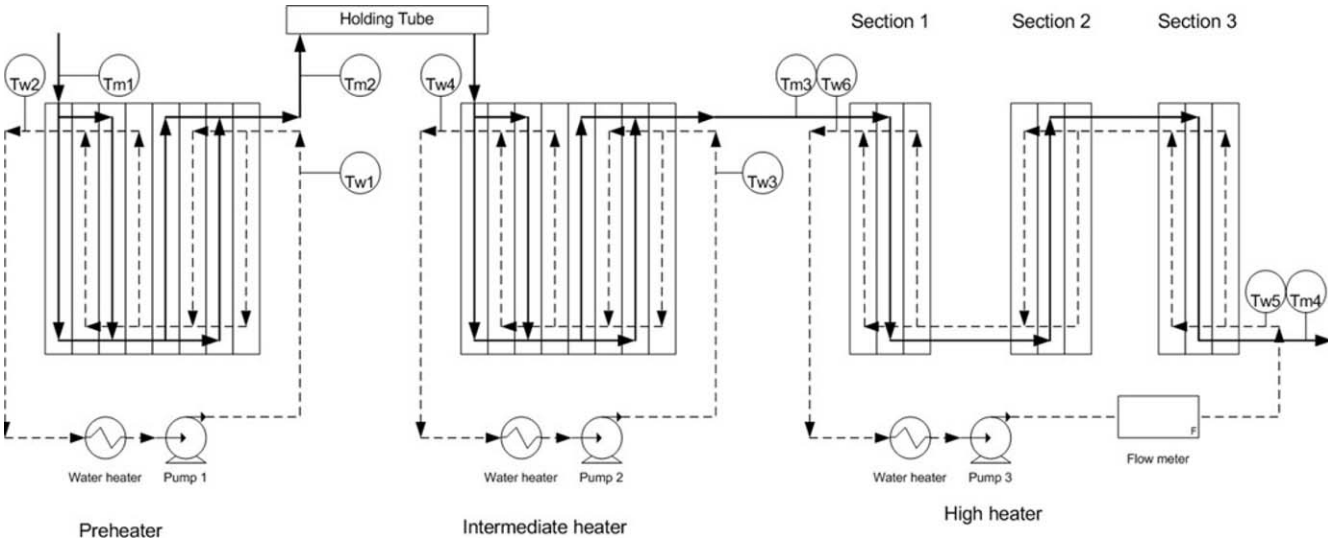

Figure 3. Flow patterns in the first three sections of the plate heat exchanger (– – – –water; —— milk).

was raised from 126°C to the final sterilisation value of 140°C. The milk was held at sterilisation temperature for 8 s prior to cooling in the final two sections.

The UHT plant was thoroughly cleaned-in-place prior to every run. The temperature difference (water-in minus milk-out in the high-temperature section) that existed for a perfectly clean plant (checked by direct inspection of plate surfaces) was established at the beginning of the work. No fouling run was carried out if the initial temperature difference was higher than this value, until the plant had been (re)cleaned-in-place.

The milk contact surfaces of the heat exchanger plates were periodically inspected (by temporarily dismantling the heat exchanger) to check that the temperature difference (water-in minus milk-out in the high-temperature section) that existed for a perfectly clean plant remained a valid indication of pre-run cleanliness. The heat exchanger was not dismantled regularly, i.e., after every fouling measurement run, owing to time constraints. However, it was occasionally dismantled after a fouling run and before cleaning-in-place in order to check visually the extent of fouling, if any, in the preheating, intermediate heating and high heating sections.

Calculation of Fouling Rate

The fouling rate in the high-heater was determined as the rate of change in the heat-transfer characteristics of the high-heater as indicated by the rate of change in the temperature difference, ΔT, at the product outlet [where ΔT = hot water inlet temperature (T_{w5}, Figure 3) minus the milk outlet temperature ($T_{m4,}$ Figure 3)]. As the milk and hot water flow rates, and the milk inlet and outlet temperatures (126 and 140°C, respectively) were automatically controlled to constant values, the rate of change of ΔT was a direct reflection of the rate of change of (rate of decrease in) the overall heat transfer coefficient in the high-heater, and can thus be called a 'fouling rate'.

The fouling rate in the high-heater, FR, for a run was thus calculated as the slope of the linear regression of ΔT against time over the period of the run as follows:

$$\Delta T = T_{w5} - T_{m4} \tag{1}$$

$$FR = \frac{(\Delta T - \Delta T_{t_0})}{t} \quad (^{\circ}\mathrm{C\ h^{-1}}) \tag{2}$$

Data Analysis

The fouling rates were converted to their logarithms and subjected to analysis of variance according to the factorial design described above (three preheat treatments × five milk preparations) replicated in two blocks (replicate 1, October; replicate 2, November). (The three UHT preheating combinations and the three evaporator preheating combinations were assumed to be essentially equivalent.)

RESULTS

The results, summarized in Table 1, show that the fouling rate in the UHT high-heater increased with the intensity (temperature × holding time) of preheating whether preheating was evaporator preheating prior to drying to make milk powder or UHT preheating applied to the recombined, reconstituted or fresh milk prior to UHT sterilization. (The preheating section and intermediate heating section did not show measurable fouling rates—as determined in a way similar to that described for the high-temperature section—and negligible amounts of deposit were found when these sections were opened.)

Analysis of variance showed a difference in overall fouling rates between the two replicates: replicate one fouling rates were greater than those of replicate two (significance, $P = 0.025$).

All milk preparations (reconstituted WMP, recombined SMP + fat and fresh milk) showed an increase in fouling rate as the intensity of preheat treatment was increased (significance, $P = 0.002$). In the case of recombined SMP + fat, the effects were similar whether the preheat treatment was applied to the milk during manufacture of the SMP

Table 1. Measured fouling rates for the October and November replicates.

Milk	Evaporator preheat treatment	UHT preheat treatment (milk powder)	Replicate 1, Oct		Replicate 2, Nov	
			Fouling rate ($^\circ$C h^{-1})	Standard error	Fouling rate ($^\circ$C h^{-1})	Standard error
Recombined milk (SMP + milk fat)	75°C, 2 s	75°C, 11 s (SMP1)	0.02	0.004	0.01	0.005
		85°C, 147 s (SMP1)	0.07	0.006	0.05	0.006
		95°C, 147 s (SMP1)	0.19	0.007	0.12	0.006
	85°C, 155 s	75°C, 11 s (SMP2)	0.11	0.004	0.01	0.004
	95°C, 155 s	75°C, 11 s (SMP3)	0.46	0.004	0.35	0.005
Reconstituted milk (WMP)	95°C, 33 s	75°C, 11 s (WMP)	0.06	0.005	0.04	0.004
		85°C, 147 s (WMP)	0.23	0.005	0.06	0.005
		95°C, 147 s (WMP)	0.57	0.009	0.23	0.005
Fresh milk	n/a	75°C, 11 s	0.14	0.005	0.04	0.004
		85°C, 147 s	0.14	0.005	0.07	0.004
		95°C, 147 s	0.23	0.005	0.15	0.005

or to the recombined milk immediately prior to the UHT process.

Mean fouling rates (across preheat treatments) varied among the different milk preparation methods (recombined SMP + fat < fresh milk < reconstituted WMP; significance, $P = 0.003$).

There were no significant differences among the degrees of response of the different milk preparations (reconstituted WMP, recombined SMP + fat and fresh milk) to the different intensities of preheat treatment (significance, $P > 0.1$).

The data, as shown in Figure 4, indicate a relatively small increase in fouling for the 85°C × 147 s preheat treatment compared with the lowest treatment (75°C × 11 s) whereas the increase in fouling resulting from the 95°C × 147 s heat treatment was considerably greater; this appears to be a common pattern for all three types of whole milk [recombined SMP + fat (a), reconstituted (b) and fresh (c)].

The effects of evaporator preheat treatment during SMP manufacture on the fouling rate of the derived recombined milk (Figure 5) showed a very similar pattern to that observed for the effects of the UHT-plant preheat treatment, i.e., only a small increase when progressing from 75°C × 2 s to 85°C × 155 s, but a considerable increase for 95°C × 155 s. The effects on the fouling rate of evaporator preheating and UHT preheating of recombined SMP + fat are shown in direct comparison in Figure 6.

DISCUSSION AND CONCLUSIONS

In the present work, higher preheat treatment of all milk preparations resulted in higher fouling rates in the high-temperature section of the UHT plant. This finding on the effect of preheating strongly supports that of Newstead *et al*. (1999), who found that relatively severe preheating (90°C × 120 s) almost doubled UHT-plant fouling rates in the case of recombined milk, and increased fouling rates by almost one and a half times in the case of reconstituted milk. However, in the case of fresh milk, our results are in contrast to those of other workers, who reported that the extent of fouling by fresh milk decreased with increasing preheating intensity (Lalande *et al*., 1984; Patil and Reuter, 1986; Mottar and Moermans, 1988). Although this discrepancy in findings presents a disturbing paradox, an explanation may lie in the differences in processing procedures used by the different groups. In the present investigation the milk was homogenized prior to preheating, whereas Lalande *et al.* and Mottar and Moermans homogenized the milk after preheating, and Patil and Reuter appear not to have homogenized the milk at all. Further, the starting material was pasteurized milk in our work, but raw milk in each of the earlier studies just cited. Newstead *et al*. (1999) reported that the effects of preheat treatment on fouling by fresh milk, that is whether preheating increased or decreased fouling rate, was influenced by homogenization, and whether this was applied before or after preheat treatment or not at all, and these effects appeared to vary from batch to batch of milk. Newstead *et al.* found that when preheating preceded homogenization, preheating increased fouling rate for one batch, but decreased it for two others; the latter effect is in agreement with the results of both Lalande *et al.* and Mottar and Moermans. Clearly, the effects of preheating and homogenization, and the order in which these are applied, need further investigation for fresh milk in particular. Such investigations are in progress in our laboratory.

In the work reported here, fouling rate increased with preheating intensity for all three whole milk types for UHT preheating, and for recombined milk for evaporator preheating. In the case of fresh milk, all constituents of the milk (including milk fat globules and their membranes, casein micelles and whey proteins) were preheated just prior to high-temperature UHT treatment. In the case of recombined milk, fat globules and their membranes were not subjected to (evaporator) preheating, as this was applied before recombination (though plasma components destined to form membranes on recombination were so subjected). In the case of reconstituted milk, all milk constituents, including fat globules and their membranes, were preheated twice—once prior to evaporation, and again prior to UHT treatment. Elucidation of the mechanisms underlying the results reported here will thus depend on studies of fouling deposit composition, milk fat globule membrane protein composition and protein changes. Such studies are currently in progress in our laboratory.

These studies will aim also at understanding the effects of drying. Fouling rates for the least intense preheat treatment were lower for recombined and reconstituted milks than for fresh milk. Drying thus appears to have conferred

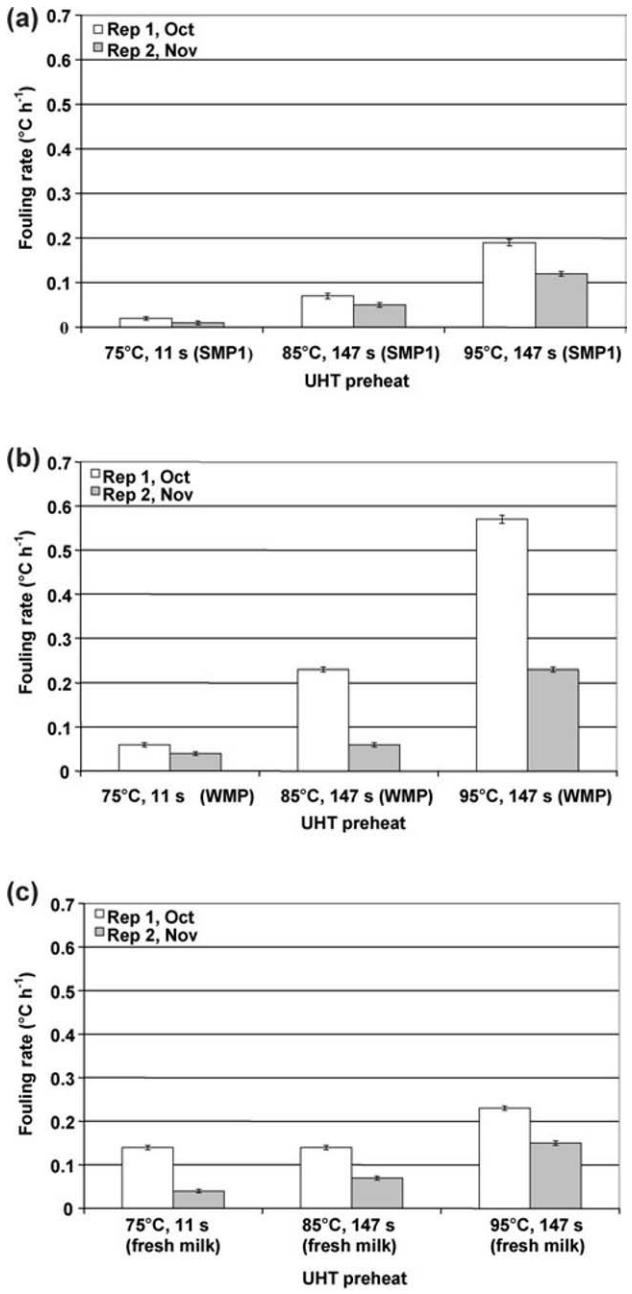

Figure 4. Fouling rates versus preheat treatment for replicates 1 and 2: (a) recombined SMP1 + fat; (b) reconstituted WMP; (c) fresh milk. (Bars show standard error of estimate from linear regression.)

some stability on these milks. This was also the case for recombined milk for the medium and high UHT preheat treatments, although the effect was small in the latter case. For these same preheat treatments, drying tended to exacerbate the fouling propensity of reconstituted milk.

A noteworthy feature of our results is the fact that although fouling rates were significantly higher in one replicate experiment than in the other, the relative effects of preheating were found to be the same in each experiment. Differences in fouling rates between replicates were presumably due to differences in composition between the two batches of milk involved. Further

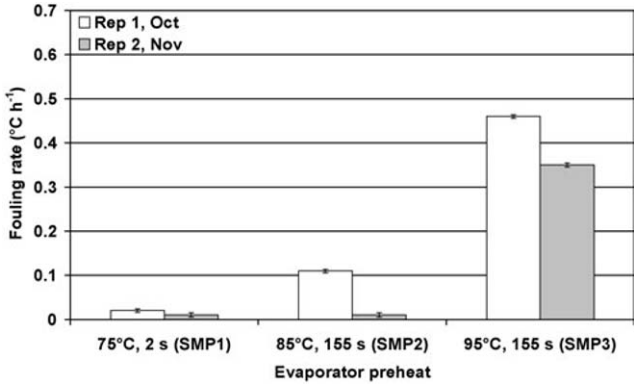

Figure 5. Fouling rates of recombined milk (SMP + fat) versus evaporator preheat treatment, in each case with a preheat treatment of the recombined milk prior to UHT processing of 75°C × 11 s. (Bars show standard error of estimate from linear regression.)

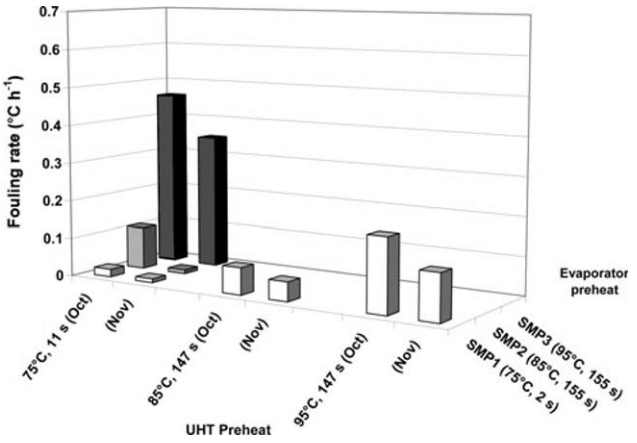

Figure 6. Fouling rates of recombined milk (SMP + fat) versus UHT-plant preheat treatment (SMP1 only) and versus evaporator preheat treatment (SMP1, SMP2, and SMP3, with UHT-plant preheat of 75°C × 11 s only).

investigation is required to determine what these differences are, and the mechanisms of their effects.

In conclusion, our results suggest that the practice of preheating previously homogenized recombined, reconstituted and fresh milks prior to UHT sterilization should be avoided.

NOMENCLATURE

t	time, h
t_0	time at start of run
T_{m4}	milk outlet temperature, °C
T_{w5}	hot water inlet temperature, °C
ΔT	temperature difference, °C
ΔT_{t_0}	temperature difference at start of run (clean plant), °C
FR	fouling rate, °C h^{-1}

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