Comparison of two ultrafiltration membrane systems for whole milk feta cheese production.

A thesis presented in partial fulfilment of the requirements for the degree of

Master of Technology

In

Food Technology

At Massey University, Auckland,

New Zealand.

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2009

Abstract

Cheese is one of the most well known food products in the world dating back to the 8th century B.C. There are more than 2000 varieties of cheese that are manufactured all over the world. Feta cheese is a soft white cheese with a salty and slightly acidic taste, which has originated from Greece. Most of the feta cheese manufactured in Greece is consumed locally, the migration of greeks to other parts of the world led to a demand for feta cheese outside of Greece. The spreading of the popularity of feta cheese to other ethnic groups in different parts of the world resulted in the high demand for feta cheese worldwide.

The modern and most efficient method of feta cheese production involves a membrane filtration method, known as ultrafiltration. The ultrafiltration process utilises pressure as a driving force to concentrate milk by removal of water and small dissolved molecules. Hollow fibre and spiral wound ultrafiltration membranes are the two types of membranes that are commonly used for cheese production. An extensive amount of research exists on the implementation of ultrafiltration to improve the efficiency of the cheese making process and the performance of the membranes. However, limited research has been conducted on the comparison of the hollow fibre and spiral wound membrane performance in the cheese making process.

The objective of the research was to determine if the hollow fibre membranes used at Puhoi Valley Cheese can be replaced with spiral wound membranes without compromising the quality of cheese produced. In order to achieve the objective, feta cheese was produced using hollow fibre and spiral wound ultrafiltration pilot plants. The operating performances of the hollow fibre and spiral wound membrane units were compared. To ensure that the quality of cheese is maintained, the cheese manufactured on the pilot plant units was analysed in terms of composition, microbiology, texture and sensory properties.

The cheese made using the hollow fibre membrane pilot plant was compared with the reference sample from Puhoi Valley Cheese as they use hollow fibre membranes to produce feta cheese. The cheese made from the spiral wound membrane unit was also compared to that made by the hollow fibre membrane pilot plant unit. The operating parameters such as the inlet and outlet pressure, pressure difference along the membrane, transmembrane pressure, flow rate, recycle rate (bleed off rate),

temperature and the run time were recorded. The operating parameters of the hollow fibre and spiral wound runs were compared with the data from Puhoi Valley Cheese.

The quality of cheese made on the hollow fibre and spiral wound pilot plant units were evaluated in terms of composition, texture, microbiology and sensory properties. The composition was defined by the fat, protein, total solids and salt contents. The fat content was determined by utilising the modified Schmid-Bondzynski-Ratzlaff method, protein by the Kjeldahl method, total solids by using the air drying oven and salt percentage by the volhard method. The texture of the cheese was determined by the fracturability and hardness from the compression curve generated using the single bite compression test. The microbiological testing was performed according to New Zealand testing methods for *E.Coli*, *Staphylococcus aureus*, coliforms and yeast and mould. The difference from the control method was utilised for sensory evaluation. The acid degree value method was used to determine the lipase activity in feta cheese.

It was found from the composition, texture and sensory analysis that the cheese from the hollow fibre pilot plant was different from the cheese manufactured at Puhoi Valley Cheeses (PVC). The spiral wound cheeses were also found to be different to PVC cheese, however the spiral wound cheeses and the pilot plant hollow fibre cheese were the same. The differences between both the pilot plant cheeses and PVC cheese were in terms of the fat, salt, moisture contents and the lipase activity in the cheeses. The fat content in the hollow fibre and spiral wound pilot plant cheeses are lower in comparison to the PVC cheese. This difference in fat content is considered to be due to the difference in the fat to protein ratio of the milk concentrated on the pilot plant and the PVC ultrafiltration system. The lower fat content resulted in firmer cheese than PVC due to more cross linking between the protein strands in cheese.

The salt content in the cheeses made using the hollow fibre and spiral wound pilot plants was lower than Puhoi Valley Cheese. This is considered to be due to the low ratio of brine volume to cheese volume used for salting the cheese. The salt content of brine decreases during brining; hence a low ratio of brine volume to cheese volume causes a significant decrease in brine concentration. The decrease in brine concentration decreases the salt intake of the cheese. As salt diffuses in the moisture diffuses out, lower salt content results in higher moisture content in the cheese. As mentioned, the moisture content of the hollow fibre pilot plant cheese was higher than

the PVC cheese. The moisture content is inversely proportional to the total solids, hence higher moisture in pilot plant cheeses implies lower total solids than the PVC cheese.

The lipase activity results showed that the hollow fibre and spiral wound pilot plant cheeses had higher lipase activity than the Puhoi valley cheese. The differences in lipase activity of the pilot plant cheeses and Puhoi Valley cheese were considered to be due to the incomplete inactivation of lipase present in milk during pasteurisation. The results from texture and sensory evaluation support the above mentioned differences. The microbiology results for all pilot plant cheeses were within the trigger limits set by Puhoi valley cheeses.

The results from monitoring the operating parameters of both the pilot plant data show that the permeate flux decreases while the total solids in milk increase with time, which was also observed from the Puhoi Valley Cheese data. However, the rate of decrease of the permeate flux and the increase of the total solids in milk are dependent on the membrane area, feed volume, transmembrane pressure, pressure drop across the membrane and the flow characteristics.

The rate of decrease in permeate flux and the rate of increase in the total solids of the hollow fibre runs and spiral wound runs are slightly different. The difference is due to the availability of larger membrane surface area and processing of larger feed volume of milk in the spiral wound runs. The transmembrane pressure and the pressure drop across the membrane were maintained as close as possible to Puhoi Valley Cheese.

In conclusion, spiral wound membranes can be used to achieve the desired total solids concentration and successfully make the same feta cheese as the hollow fibre pilot plant. In order to make the same quality of feta cheese as Puhoi Valley Cheese using the spiral wound membrane pilot plant, the same composition of milk used for concentration at Puhoi Valley Cheese needs to be used on the spiral wound pilot plant unit. It is recommended that Puhoi Valley Cheeses should be replaced with spiral wound membranes if they are more economical in terms of cost than the hollow fibre membranes.

Acknowledgements

I would like to acknowledge the following people for their assistance

- My supervisors Rod Bennett, Dr. Marie Wong from Massey University and Hamish Conway from Goodman Fielder.

- Kay McMath for sensory advice.

- All operators in the ultrafiltration plant, Meagan, Bex, Olive and Chris from the Micro lab at Puhoi Valley Cheeses.

- Anthony Light and Caroline Edge from Puhoi Valley Cheeses.

- Garry Radford, Steve Glasgow, Michelle Tamahera and Helen Matthews for technical assistance in the labs at Massey University.

- Anne Wilson, Mike Dunn and Sonja Lincoln from Goodman Fielder (Longburn division) for milk supply and microbiological analysis of samples.

- Byron McKillop, Chris Hall, Warwick from the product development lab at Massey University.

- Hugh Morton for statistical advice.

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1.0 Introduction

Cheese is the generic named used for a group of fermented milk based food products which date back to the 8th century B.C. (Fox, 2004; Polychroniadou-Alichanidou, 2004). The primary objective of cheese-making is to preserve the main constituents of milk. The retention of the principal constituents of milk makes cheese a highly nutritious food product. Cheese-making has evolved as a result of various developments such as stability in terms of storage, ease of transport and provision of variety in the human diet (Fox, 2004).

It is estimated that more than 2000 varieties of cheese exist to date (Gunasekaran and Mehmet, 2003). Cheese is most commonly classified into hard, semi hard, semi soft and soft based on the moisture content in the cheese (Polychroniadou-Alichanidou, 2004). In 2007, the total cheese production in New Zealand was 308 thousand metric tons, 90% of the cheese manufactured in New Zealand is exported. The New Zealand cheese export returns were $US 830 million in 2007 (Lee-Jones, 2007). The world wide annual production of cheese was greater than the combined yearly production of coffee beans, tea leaves, cocoa beans and tobacco.

Feta cheese is a soft white cheese, ripened and kept in brine for storage, which gives it a salty, slightly acidic taste (Robinson and Tamime, 1991). Feta cheese has traditionally been made from sheep or goat's milk or a mixture of both (Robinson and Tamime, 1991; Polychroniadou-Alichanidou, 2004). Feta cheese has been produced in Greece since ancient times, it has been part of the Greek diet for centuries and still remains a significant element in their diet to date. Greece is a small country with an extremely high annual production of cheeses in brine, 95% of which is feta cheese (Robinson and Tamime, 1991). Most of the annual feta production in Greece is consumed locally, very small quantities are exported (Polychroniadou-Alichanidou, 2004).

For many centuries feta cheese had been known only to the Balkan countries; migration of Greeks to various countries around the world resulted in large Greek ethnic groups in many countries (Robinson and Tamime, 1991; Polychroniadou-Alichanidou, 2004). The large Greek communities outside of Greece retained their traditional dietary habits, therefore new markets for feta cheese emerged and international trade of feta cheese was established (Robinson and Tamime, 1991). As

most of the feta cheese made in Greece was consumed locally, the availability of traditional feta cheese to meet the international demand was insufficient. In order to meet the demands for feta cheese, cow's milk was used with the feta cheese manufacturing procedure (Robinson and Tamime, 1991).

Traditionally feta cheese is made from thermal heating of milk, addition of yoghurt culture and rennet from a calf's stomach, and ripening in a wooden barrel (Georgala et al., 2005). Ultrafiltration is a pressure driven membrane filtration process, which was introduced to the dairy industry around 1970 (Robinson and Tamime, 1991). Modern day feta cheese production involves ultrafiltration which is used in cheese-making to concentrate the milk without thermal heating, and using mesophilic starter cultures and commercially available bovine rennet (Georgala et al., 2005). Today, large quantities of brined cheeses are made successfully using the ultrafiltration technology. The main advantage of utilizing ultrafiltration in cheese-making is the increase in the yield due to inclusion of the whey proteins in the cheese curd.

Ultrafiltration in cheese-making can be achieved using either hollow fibre or spiral wound membranes. Both membranes have high area to volume ratio and are used in the production of feta cheese (Cheryan, 1998). A wide range of research has been conducted on the improvement of the performance and efficiency of the membranes. However, very little research exists on comparing the performance of the hollow fibre and spiral wound membranes directly.

The objective of this research was to make same quality of feta cheese utilising both hollow fibre and spiral wound membranes, so that the hollow fibre membranes at Puhoi Valley Cheeses could be replaced by spiral wound membranes. The cheeses made were to be analysed in terms of chemical, microbiological and sensory methods to evaluate the quality of the cheeses. The performance of both the membranes were compared to determine if the same or better quality of feta cheese could be made using spiral wound membranes compared to the existing hollow fibre membrane process at Puhoi Valley Cheeses.

2.0 Literature review

2.1 Introduction

The main objective is to compare the production of feta made using ultrafiltration with spiral wound and hollow fibre membranes. In order to achieve the objective a good understanding is required of the principles of ultrafiltration, cheese-making in general, feta making using ultrafiltration and chemical, microbiology and sensory test methods to evaluate quality of cheese is required.

An extensive amount of research has been conducted and reported on the use of ultrafiltration in the dairy industry, specifically towards implementation of ultrafiltration in the cheese making process. The majority of the research looks at the effect of ultrafiltration on the quality of cheese made or the efficiency of the cheese making process.

The cheese making process is essentially the removal of moisture through controlling acidity and temperature (Mistry, 2002). Increase in acidity and temperature results in a firmer gel hence less moisture in the cheese made. The type of the cheese made and its structure is defined by the amount of moisture removed.

The efficiency of the cheese making process determines the cost of production. The efficiency is dependent on the retention of casein, fat, insoluble minerals and water. The membrane filtration methods were introduced into cheese making in order to improve the quality of cheese and make the process more efficient (Mistry, 2002). Membrane filtration methods result in retention of whey proteins which have high nutritional benefits associated with them hence better quality of cheese can be made using membrane filtration methods.

2.2 Membrane Filtration

2.2.1 What is membrane filtration?

Membrane filtration is a separation process used to separate different molecules based on size (Nielsen, 2000). A membrane filtration process can be viewed as a selective sieving mechanism, where certain molecules permeate through the pores in the membrane (Winston and Sirkar, 1992; Cuperus and Nijhuis, 1993). Permeation of certain molecules from a solution results in an increase in the concentration of the remaining particles in the retained solution (Winston and

Sirkar, 1992). The concentrated feed retained by the membrane is known as the retentate or concentrate. The rate of permeation of the molecules through the membrane is defined by the driving force (Strathmann, 1987).

2.2.2 Types of membrane filtration

Membrane separation processes can be split into different categories based on the driving force used for filtration (Nielsen 2000). The pressure driven filtration processes are reverse osmosis also known as hyperfiltration, nanofiltration, ultrafiltration and microfiltration (Mistry, 2002; Kuriyel, 2000). The concentration driven membrane filtration process is known as dialysis and the electrode potential difference driven filtration is called the electrodialysis process (Nielsen, 2000).

The different pressure driven processes can be classified on the basis of the size of the components passing thought the membrane, which in turn relates to the required differential pressure needed to operate the system.

The required operating pressure for reverse osmosis is higher in comparison to the other pressure driven membrane processes. The high pressure is required to overcome the osmotic pressure difference to separate water from the feed solution (Winston and Sirkar, 1992). The transmembrane pressure achieved during reverse osmosis filtration ranges from 10-100 bar (Mulder, 1991). In reverse osmosis, ideally, water is the only material that passes through the membrane, all the dissolved molecules (solutes) and suspended material is rejected (Nielsen, 2000; Wagner, 2001). An application of reverse osmosis in the dairy industry is the removal of water from milk and whey (Wagner, 2001). In the food and beverage industry reverse osmosis is used to recover substances that are temperature or pH sensitive and solids from wastewater to reduce waste treatment costs (Meyer et al., 1995).

Nanofiltration is very similar to the reverse osmosis filtration process (Winston and Sirkar, 1992). In nanofiltration small monovalent ions such as sodium and chloride pass through the membrane (Mistry, 2002). As the osmotic pressure to overcome is not as great as that of reverse osmosis, the required transmembrane pressures are in the range of 5-35 bar (Nielsen, 2000).

Nanofiltration is used in the environmental industry to purify and reuse caustic cleaning solutions (Nielsen, 2000). In the dairy industry nanofiltration can be utilised to remove water and small ions such as sodium, potassium and chloride from a feed solutions. It is also used to remove salt and water from whey to produce desalted whey concentrate (Wagner, 2001). As nanofiltration is still new in terms of cheese making, it is still in the development stages (Mistry 2002).

In ultrafiltration, water, minerals, sugars, organic acids and vitamins pass through the membrane (Nielsen, 2000), while large molecules such as proteins, fat and carbohydrates are fully or partially retained depending on the pore size of the membrane (Mulder, 1991). The transmembrane pressures attained in ultrafiltration range between 1-10 bar (Wagner, 2001; Nielsen, 2000).

Microfiltration is utilised to retain larger components of feed solutions such as proteins in milk (Mistry 2002). The transmembrane pressure of microfiltration is lower than that of ultrafiltration, usually less than 2 bar (Wagner, 2001). Ultrafiltration and microfiltration are most commonly used in the dairy industry. Microfiltration in the dairy industry is used to improve the quality of milk for cheese-making and extend the shelf life of market milk (Nielsen, 2000). It can also be used for protein separation and for the removal of fat, bacteria or suspended solids. Applications of microfiltration in other industries include clarification and biological stabilisation in the beverage industry (Winston and Sirkar, 1992). Figure 1 shows the above mentioned four different pressure driven membrane filtration processes.

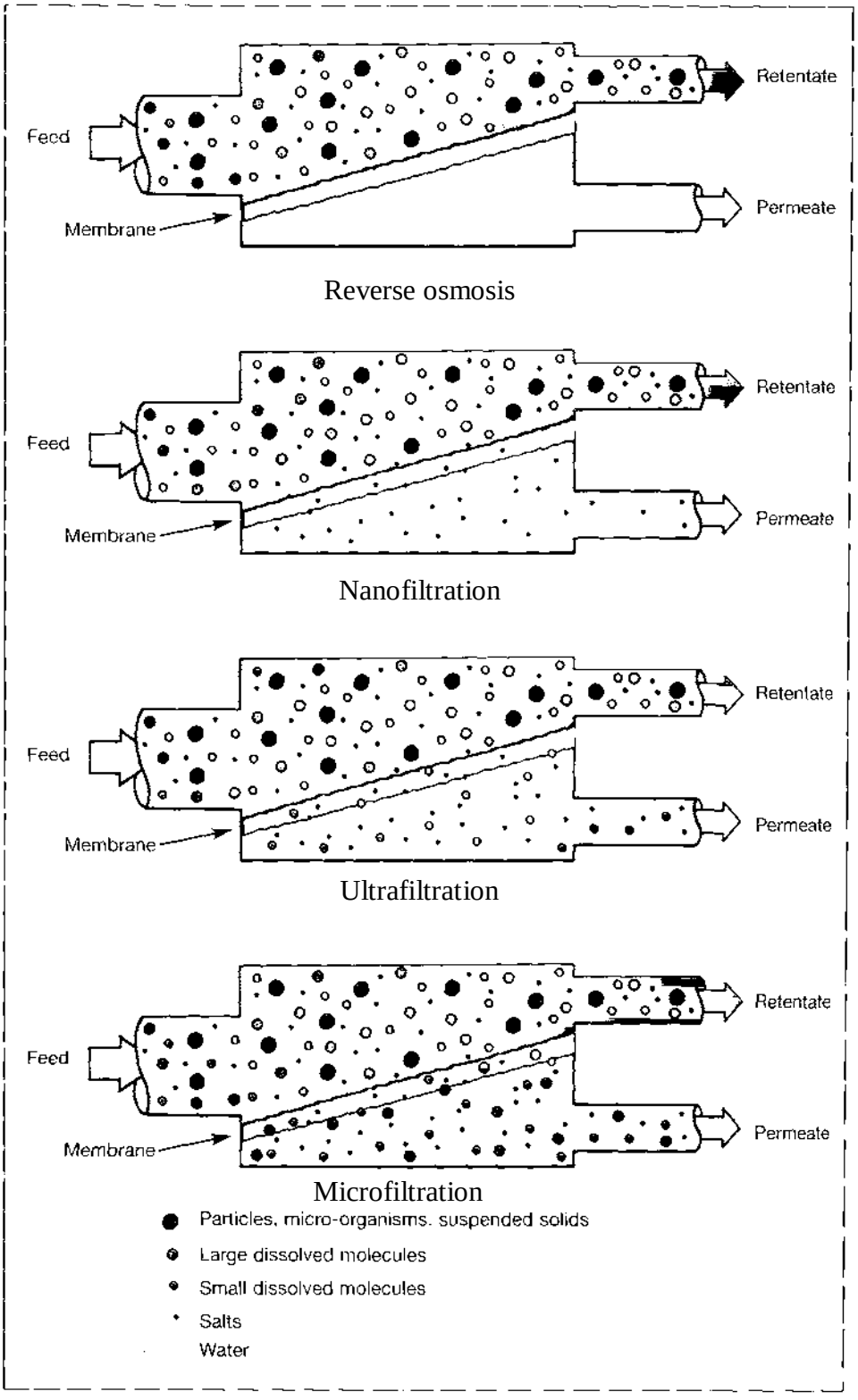

Figure 1. Four different types of pressure driven membrane filtration processes (Nielsen, 2000).

2.2.3 Types of feed flow

The feed flow through the membrane plays a very important role as it effects the permeate flux which determines the performance and efficiency of the process. The feed flow can be either dead end flow as shown in Figure 2 or cross flow as shown in Figure 3.

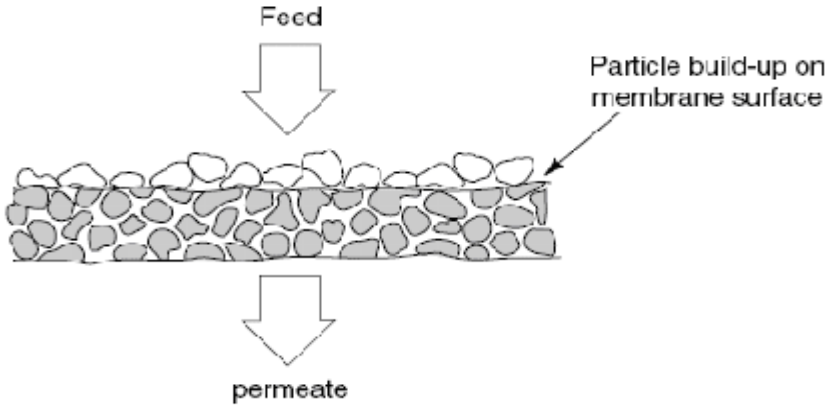

Figure 2. Schematic of dead end flow through the membrane, showing the build up of large particles on the membrane surface (Baker, 2005).

In dead end filtration the feed flow enters perpendicular to the membrane surface as shown in Figure 2. Dead end flow is often not desired in ultrafiltration as this causes build up of particles larger than the pore size on the membrane surface. The large particle build up over time forms a layer through which the smaller particles that are being separated must diffuse. Hence, decreasing the smaller particle concentration in permeate and increasing the cost of separation due to the higher operational costs such as pumping costs (Baker, 2005).

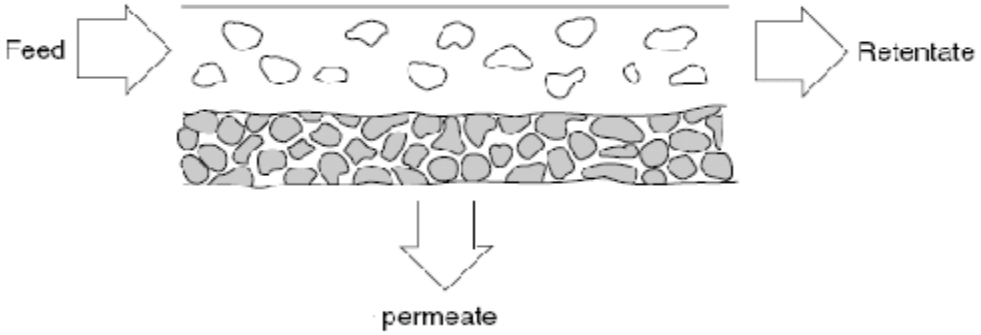

Figure 3. Schematic of cross flow or tangential flow through the membrane, showing the sweeping action of the feed flow (Baker, 2005).

The feed flow parallel to the membrane surface or perpendicular to permeate flow is known as the cross or tangential flow as shown Figure 3 (Baker, 2005; Song and Elimelech, 1995). Cross flow is more appealing in ultrafiltration as it avoids deposition of large particles on the membrane surface by the sweeping effect caused due to the feed flow being parallel to the membrane surface. Cross flow filtration results in less fouling in comparison to the dead end flow. However, all cross flow filtration processes over time demonstrate the concentration polarisation effect.

Concentration polarisation is where the particle concentration is greater at the membrane surface than in the bulk (Song and Elimelech, 1995). The cross flow velocity plays a significant part in membrane filtration as it directly influences concentration polarisation. An increase in cross flow velocity results in a decrease in concentration polarisation. It is vital that concentration polarisation is reduced as much as possible as it has a very strong impact on permeate flux, operating costs and the overall performance of the process.

2.3 Ultrafiltration

2.3.1 Applications

Ultrafiltration is used in latex concentration, pigment recovery and concentration of proteins for biopharmaceuticals. It is used in sewage treatment and production of sterile water for antibiotics (Kuriyel, 2000).

Ultrafiltration is used in the food industry for production of concentrates and isolates from soya beans, sunflower and cotton seeds. Ultrafiltration is utilised to remove glucose from egg white and partial concentration before drying. Other useful applications in the food industry include refining sugar solutions, fractionation and concentration of gelatin (Pal, 2003).

In the dairy industry ultrafiltration is used in a number of applications including cheese making. Ultrafiltration is successfully implemented into the cheese making process for cheeses such as feta, quarg, camembert, goat's milk cheese and mozzarella (Cheryan, 1998). Other applications of ultrafiltration in the dairy industry are recovery of protein from cheese and casein whey that are produced from traditional cheese making processes (Kuriyel, 2000). Ultrafiltration is also

used for protein standardisation to eliminate seasonal differences in protein content of milk (Pal, 2003). High value whey protein concentrates are produced from ultrafiltration of whey to remove lactose and salt. Ultrafiltration can also be used to produce milk protein concentrates (Nielsen, 2000).

Ultrafiltration is also used to produce superior taste and textured milk based fluids and fermented products (Cheryan, 1998). Research has been carried out to investigate using ultrafiltration to recover lactose from the dairy wastewater (Chollangi and Hossain, 2007). It decreases the biological oxygen demand and allows direct discharge of wastewater into the sewage (Atra et al., 2004).

Other applications of ultrafiltration include electrocoat paint recovery, textile size recovery and recovery of lubricating oil in the chemical industry. For medical applications it is used in kidney dialysis operation and in the biotechnology industry it is used to recover protein from animal blood, gelatin and glue (Yeh, 2002).

2.3.2 Ultrafiltration Membranes and Flux Models

Ultrafiltration membranes come in plate, spiral wound or tubular forms (Winston and Sirkar, 1992). The tubular membranes are either self supported like the capillary hollow fibre or supported by other means (Cheryan, 1998; Kuriyel, 2000). The plate membranes are not commonly used in the dairy industry, because the surface area to volume ratio is much lower for plate membranes in comparison to other types of membranes (Kuriyel, 2000).

The four fundamental parameters that affect the permeate flux in ultrafiltration are the transmembrane pressure, turbulence in the feed channel, concentration of the feed and the temperature (Cheryan, 1998).

The permeate flux of ultrafiltration can be analysed using the gel polarisation model, the osmotic pressure model or the resistance in series model (Yeh, 2002). The gel polarisation model is used when the transmembrane pressure of ultrafiltration is large enough to form a gel layer on the membrane surface. The gel layer limits the membrane permeation rate irrespective of a further increase in transmembrane pressure. In the osmotic pressure model, permeate flux declines

due to a drop in transmembrane pressure as the osmotic pressure of the retentate increases. The resistance in series model is utilised when permeate flux decreases due to the resistance from fouling or solute adsorption and concentration polarization (Yeh, 2002).

2.3.3 Spiral wound membranes

Cheryan (1998) describes the spiral wound membrane as one of the most compact and inexpensive ultrafiltration membranes. Two flat sheet membranes are placed together with their active sides facing away from each other. Each flat sheet membrane has one active side through which the smaller molecules permeate through. A feed spacer, which is a mesh like material, is placed between the two flat sheet membranes. The two flat sheet membranes with the feed spacer separating them are rolled around a perforated tube (Cheryan, 1998; Nielsen 2000). Figure 4 shows the schematic of a spiral wound membrane. The advantages and disadvantages of spiral wound membranes are listed below.

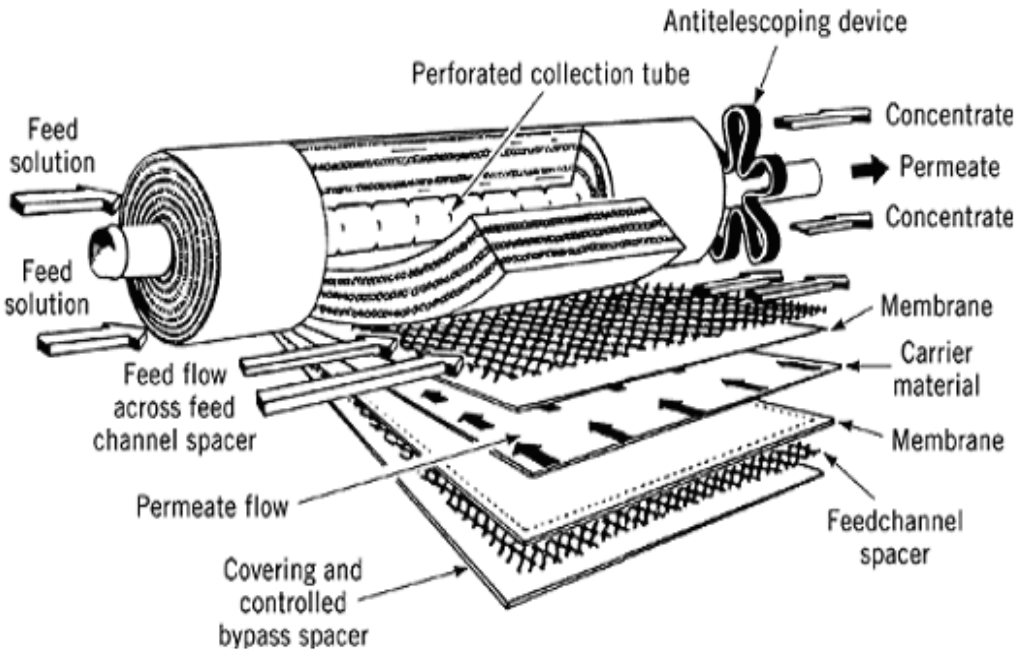

Figure 4. Schematic of a Spiral wound module showing the direction of the feed flow and the set up of the spiral wound membrane (Cheryan, 1998).

Advantages of spiral wound membranes:

- Narrow channel heights allow larger membrane areas for a given cartridge size (Cheryan, 1998; Winston and Sirkar, 1992).

- Turbulent flow is induced due to the presence of a feed spacer, which contributes to a decrease in concentration polarization (Cheryan, 1998; Winston and Sirkar, 1992).
- High surface area to volume ratio (Strathmann, 1987; Wagner, 2001).
- Lowest in energy consumption provided that the flow rates and pressure drop are kept low (Cheryan, 1998).
- Capital costs are lowest amongst all the available module designs (Strathmann, 1987; Wagner, 2001).
- Low membrane replacement costs (Nielsen, 2000).
- Requires lower feed velocity in comparison to other types of modules (Winston and Sirkar, 1992; Wagner, 2001).

Disadvantages of spiral wound membranes:

- High pressure drop due to the presence of the feed spacer (Cheryan, 1998).
- An anti-telescoping device (ATD) is required to overcome the telescoping effect (Cheryan, 1998).
- Identical modules from different suppliers have different permeate collection tube sizes, therefore difficult to substitute one manufacturer's spirals with another (Cheryan, 1998).
- No back flushing facility available (Nielsen, 2000).

The high pressure drop associated with the presence of feed spacer coupled with high flow rates causes the telescoping effect. The telescoping effect is when the spiral pushes itself out in the direction of the flow, changing its position. The telescoping effect can cause damage to the membrane, therefore anti-telescoping devices are required on the downstream end of the module (Cheryan, 1998).

Clarke and Heath (1997) found that as the transmembrane pressure and cross-flow velocity increase, the permeate flux increases in spiral wound modules. This outcome is in agreement with literature that an increase in driving force increases the permeate flux generated (Cheryan, 1998). As stated in literature, an increase in cross flow velocity reduces the effect of concentration polarization therefore an increase in permeate flux (Cheryan, 1998). The research was performed to develop a model that predicts the permeate flux of a spiral wound module based

on the experiments conducted on a flat (Plate) membrane (Clark and Heath, 1997). It was found that the permeate flux of the spiral wound module shows a strong dependence on cross flow velocity, it was suggested that further research should be conducted to determine the cause of this strong dependence (Clark and Heath, 1997).

2.3.4 Hollow fibre membranes

Figure 5 shows the cross section of a hollow fibre membrane. Bundles of the individual fibres are sealed into a hydraulically symmetrical housing to make one module (Kuriyel, 2000).

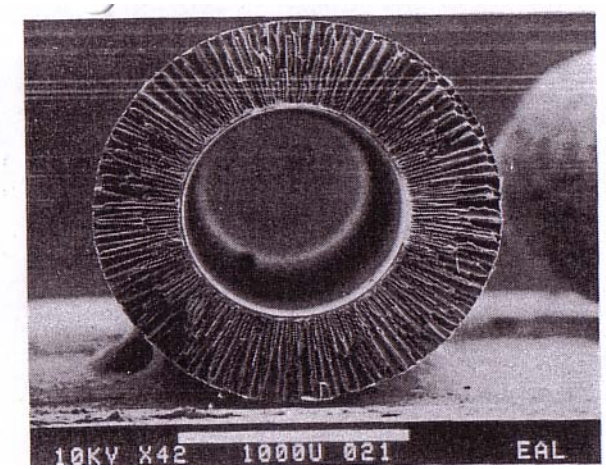

Figure 5. Cross sectional view of an individual hollow fibre, the pores are clearly visible on the fibre (Cheryan, 1998).

The bundles are arranged in a shell and tube arrangement as shown in Figure 6 (Yeh et al., 2003). The feed enters and exits the shell as shown in Figure 6, where as the permeate that has passed through each individual hollow fibre is collected on the tube side as shown below.

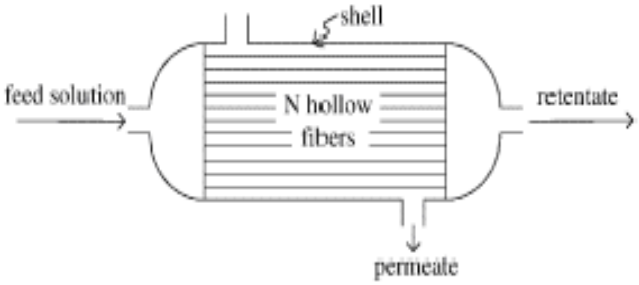

Figure 6. Schematic of the shell and tube arrangement and the flow directions of the hollow fibre module (Yeh et al., 2003).

The advantages and disadvantages of utilizing a hollow fibre module are listed below.

Advantages of hollow fibre membranes:

- One of the more economical membranes with respect to energy consumption, as long as the unit is operated under a combination of low-pressure drop and recommended flow rates (Cheryan, 1998; Nielsen, 2000).
- Very high shear rate due to thin channels (Cheryan, 1998).
- Highest surface area to volume ratio amongst all modules along with low hold up volume (Mulder, 1991; Winston and Sirkar, 1992; Wagner, 2001).
- Biggest advantage is back flushing capability, therefore improved cleanability (Cheryan, 1998; Nielsen, 2000).
- Low energy costs (Nielsen, 2000).
- Very low hold up volume (Winston and Sirkar, 1992; Nielsen, 2000)

Disadvantages of hollow fibre membranes:

- Most hollow fibre modules operate in the laminar flow region hence sensitive to fouling (Cheryan, 1998; Nielsen, 2000).
- Low pressure operating conditions to avoid fibre failure (Cheryan, 1998).
- Cross flow rate is proportional to pressure drop therefore restrictions on the inlet pressure limit the inlet flow rate (Cheryan, 1998).
- Out of a bundle consisting of 50-3000 fibres, one fibre bursts and the module has to be replaced (Cheryan, 1998).
- Replacement membrane costs are high (Nielsen, 2000; Cheryan, 1998).
- High velocity required to avoid plugging due to concentration polarisation at the inlet (Wagner, 2001; Cheryan, 1998).
- High plant investment costs (Wagner, 2001; Strathmann, 1987).

A study on the fibre failing frequency of hollow fibre membranes utilized in water treatment plants states that four principal factors contribute to the failure of fibres (Gijsbertsen-Abrahamsea et al., 2006). The four factors are: strength of membrane material and chemical degradation, construction of the module, design

and operating conditions of the module, and the incidents that occur while the module is in use (Gijsbertsen-Abrahamsea et al., 2006).

Most commonly fibre failure is caused by specific incidents such as failure of a pre-treatment step. Another important factor contributing to the fibre failure was identified to be the transmembrane pressure. Although transmembrane pressure causes fibre failure, it is difficult to determine what transmembrane pressure or backwash pressure causes fibre failure (Gijsbertsen-Abrahamsea et al., 2006). It was stated that operating at higher transmembrane pressures than the manufacturer's recommended operating transmembrane pressures was found as a source of fibre failure.

A study on the effect of the design and operating conditions on permeate flux of a hollow fibre ultrafiltration module shows, that as the transmembrane pressure decreases the permeate flux declines (Yeh et al., 2003). Other parameters that affect the permeate flux are identified to be the feed concentration and the feed cross flow velocity. It was found that as the feed concentration increases the permeate flux decreases, this is due to the concentration polarization phenomenon (Yeh et al., 2003).

The increase in feed cross flow velocity shows an increase in permeate flux at the entrance of the tube, but as the feed flows through the length of the tube the permeate flux decreases. The initial increase in permeate flux is considered to be due to the decrease in concentration polarization by the increase in feed velocity (Yeh et al., 2003). The initial decrease in concentration polarisation coupled with better retention of proteins at the entrance of the tube increases the viscosity of the feed solution as it progresses along the tube (Cheryan, 1998). The increase in protein concentration along the tube, increases concentration polarization along the tube, resulting in a decrease in permeate flux down the tube (Yeh, 2002).

It was suggested that the other factor contributing to the decrease in permeate flux towards the end of the tube is the higher feed velocity, which causes a decrease in transmembrane pressure (Yeh et al., 2003). High velocities result in greater pressure drop along the tube due to friction. Therefore the pressure in the tube

towards the end would be lower than that at the entrance of the tube, this results in lower transmembrane pressure at the end of the tube.

The study also shows that an increase in the length of the hollow fibre tube decreases the permeate flux generated. As the length of the tube increases the pressure drop along the tube increases, which causes a decrease in transmembrane pressure along the tube. The decrease in the driving force results in a decrease in permeate flux. Overall the study identifies that the decrease in transmembrane pressure and the increase in concentration polarization are the more significant factors that cause a decrease in permeate flux (Yeh et al., 2003).

Research was conducted on decline of permeate flux along the membrane in a tubular module by Yeh (2002). It was found that the permeate flux along the tube decreased significantly at lower transmembrane pressures as the effective driving force decreased in the downstream part of the tube. The permeate flux along the tubes was calculated from an equation derived based on the resistance-in-series model. The research also shows that a rapid decline in permeate flux can be observed with increased feed concentration. It is observed that this is due to the substantial increase in concentration polarisation layer along the tube (Yeh, 2002).

Secchi et al (1999) showed with comparison of data from experiments and equations that the effects of concentration polarization and adsorption on the membrane surface are of significance in hollow fibre ultrafiltration modules. The limitations of the study were that the experimental data were obtained from literature and that both sets of results are obtained using slightly different methods (Secchi et al., 1999).

The increase in operating temperature results in a decrease in the viscosity of the fluid, hence increasing the permeate flux. Liu and Wu (1998) found that an increase of one degree Celsius results in a 2.5% decrease in viscosity of water and an increase of 3% in permeate flux. Although higher temperature increases permeate flux, the operating temperature should be within the limits that the membrane and the feed solution could withstand without degradation (Liu and Wu, 1998). It should also be noted that higher temperatures result in higher energy consumption.

2.3.5 Comparison of spiral wound and hollow fibre membranes

Cheryan compares the flow characteristics of spiral wound and hollow fibre membranes. The state of the flow in terms of turbulence or laminar can be determined by the relationship between pressure drop (ΔP) and flow rate (Q) in the feed channel.

Figure 7 illustrates that the spiral wound module follows a turbulent flow characteristic as the relationship between pressure drop and flow rate is not linear. The hollow fibre data show a laminar flow characteristic as the relationship between pressure drop and flow rate is linear (Cheryan 1998). Although turbulent flow is preferred as it decreases concentration polarisation, it cannot be concluded that the spiral wound module is the better option. It can be observed from the scale of the y-axis in Figure 7 that spiral wound modules operate at a significantly lower flow rate in comparison to the hollow fibre modules.

The pressure drop in Figure 7 is based on the inlet and outlet pressures on the feed side and the flow rate measured is also on the feed side. The dimensions of the spiral wound and hollow fibres modules used in Figure 7 are 0.0762 m and 0.0508 m in diameter respectively and 0.635 m and 0.889 m in length respectively.

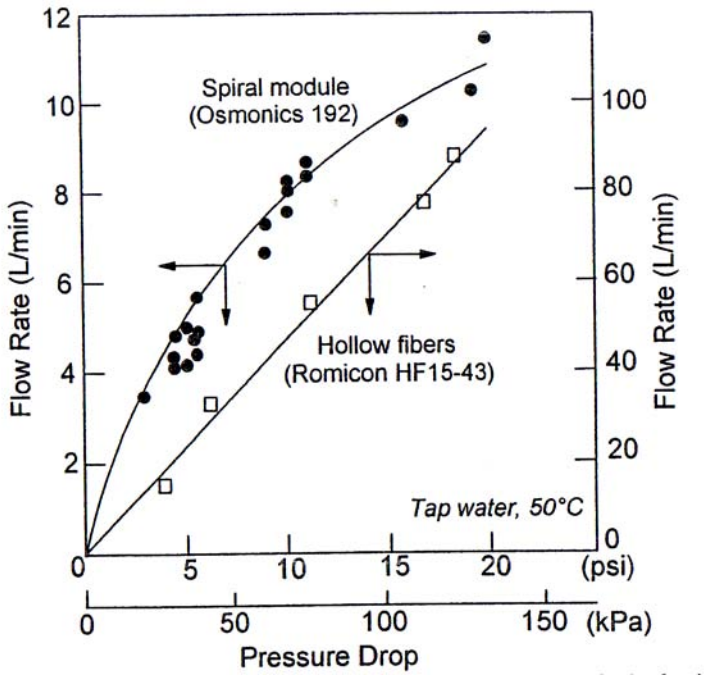

Figure 7. Plot of flow rate vs. pressure drop of hollow fibre and spiral wound modules. It can be observed that the spiral wound shows turbulent flow characteristics whereas hollow fibre shows laminar flow characteristics (Cheryan 1998).

A study by Akoum et al (2005) measures the performance of a vibrating module and compares the results with a tubular module and spiral wound module performance. Experiments on the vibrating module and tubular module were conducted using reconstituted skim milk, whereas spiral wound module experiments were based on diluted skim milk (Akoum et al., 2005). The performance was measured based on the permeate flux obtained for a given transmembrane pressure. However the performances of the tubular and spiral wound modules are not directly compared against each other. It was reported that vibrating modules consume less energy to generate higher permeate flux and higher shear rates than tubular modules.

Grandison et al (2000) observed that a significant difference exists between the flux patterns generated by hollow fibre and spiral wound membrane for skim milk. They suggest that this is due to the different hydrodynamic operating conditions of the membranes. It was stated that deposition on the membrane surface at higher transmembrane pressure is closely packed, hence difficult to remove (Grandison et al, 2000).

The study on the use of hollow fibre and spiral wound modules for whey ultrafiltration shows that the concentrates achieved are identical from both modules, operating under the same conditions (Castro and Gerla, 2005). Castro and Gerla (2005) found that the significant resistance in the hollow fibre membranes was due to the fouling in the membrane pores. The study also showed that the principal resistance in the spiral wound module was due to concentration polarisation. Cheryan (1998) states that concentration polarisation is a phenomenon that can be decreased by altering the feed velocity and feed concentration. However the study was conducted with frozen cheese whey which was thawed at room temperature before ultrafiltration, therefore results may vary for different feeds. The data obtained from this study on the fouling resistance and membrane intrinsic resistance were in agreement with literature (Cheryan, 1998).

The study on the physicochemical, microbiological and physical stability of frozen ultrafiltered milk concentrates by Voutsinas et al (1995) found good protein stability in the frozen concentrated sheep's milk. It was also reported that in frozen concentrated cow's milk protein precipitation was one of the important sources of instability (Voutsinas et al., 1995). Freezing results in rearrangement of the casein structure due to expansion caused by solidification, hence altering the pH of milk. The pH significantly affects the solubility of protein in milk, therefore resulting in protein precipitation (Molins, 1991).

2.4 Cheese

2.4.1 Manufacturing process of Feta cheese

Cheryan (1998) stated that incorporation of ultrafiltration into feta cheese production has remarkable advantages. Figure 8 represents the traditional feta cheese making process. Figure 9 summarises the modified manufacturing process of feta cheese making. The modified manufacturing process of feta cheese that utilises ultrafiltration differs slightly from the traditional feta cheese making process as observed in Figures 8 and 9.

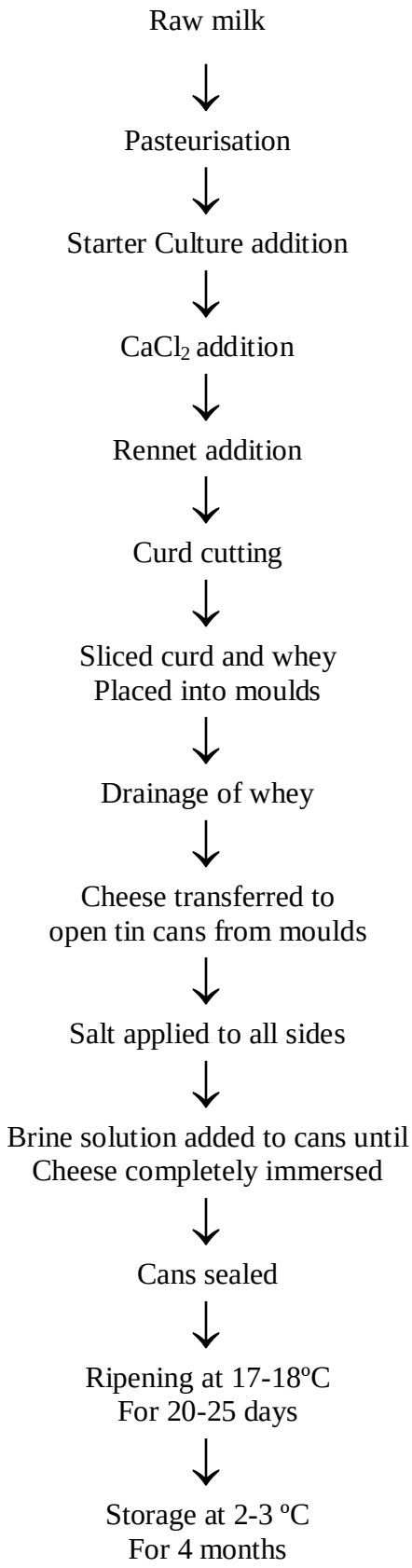

Figure 8. Summarised flow chart of the traditional cheese making process (Mallatou et al., 1994).

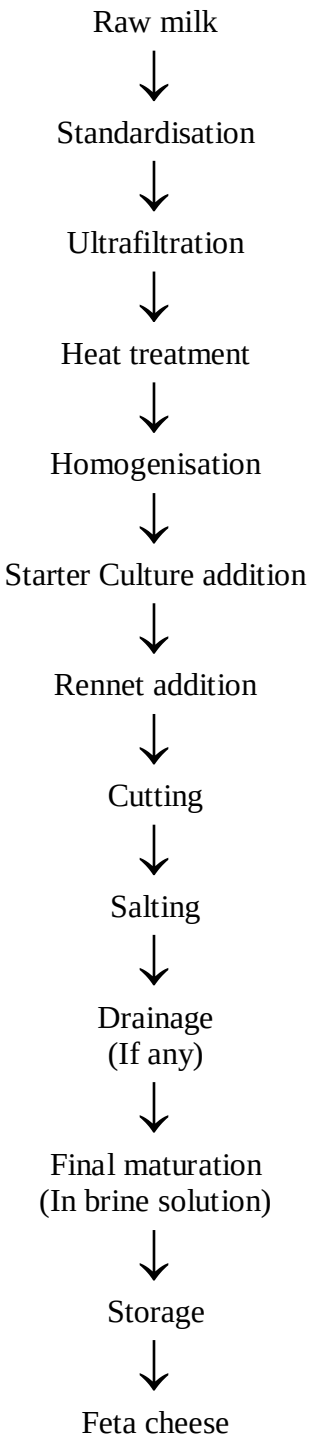

Figure 9. Summarised flow chart of the manufacturing process of feta cheese utilising ultrafiltration (Scott, 1998).

Raw milk is standardised to ensure that it is of standard composition at all times of the year. The right composition is critical in cheese making, legal standards for various cheeses exist which determine the required fat-to-protein ratio (Gunasekaran and Mehmet, 2003).

Ultrafiltration (UF) is utilised to increase the concentration of larger molecules such as protein and fat in the milk. There are two commercial methods used to make feta cheese using ultrafiltration: they are known as the UF-Feta 2.5 process and the UF-Feta 5 process (Zeman and Zydney, 1996; Cheryan, 1998).

The UF-Feta 2.5 process involves concentrating milk by a factor of 2.5 before addition of rennet and other culturing agents. The resulting curd still needs to be cut to release the extra water and soluble components (Cheryan, 1998). The yield of whey that is released is considerably smaller than the traditional process. An increase of around 15% in the overall yield of feta cheese can be obtained through this method (Zeman and Zydney, 1996).

UF-Feta 5 process utilises a concentration factor of 5, the resulting solution is to be directly cultured and moulded without any production of whey (Cheryan, 1998). This process results in elimination of the curd cutting and whey draining stage and an increase of 30% on the overall cheese yield in comparison to the traditional method as shown in Figure 8 (Zeman and Zydney, 1996).

Heat treatment also known as pasteurisation is required to destroy harmful microorganisms and enzymes in raw milk to ensure a product that is good in quality and safe to consume (Gunasekaran and Mehmet, 2003).

Fat globules present in milk over time rise to the surface because of their lower density and form a cream line (Varnam and Sutherland, 1994; Hall, 2007). Traditionally milk is homogenised to prevent formation of the cream line. In homogenisation fat globules present in milk are broken up into smaller and uniform particles (Deeth and Fitz-Gerald, 1976; Hall, 2007). This results in a stable solution and prevents fat globules coming together and prevents creaming (Varnam and Sutherland, 1994). The homogenised milk is pasteurised or heat treated to eliminate microbial contamination (Varnam and Sutherland, 1994) and to deactivate the lipase enzymes.

Homogenisation breaks down the fat globule membrane to form smaller and uniform fat globules, with this milk fat loses its natural protective membrane and is coated with a new membrane consisting of small casein particles (Deeth and

Fitz-Gerald, 1976; McMahon et al., 1992). The new membrane is less structured and more permeable making the new smaller fat globules more vulnerable to lipolysis (Deeth and Fitz-Gerald, 1976). Raw milk contains lipase enzymes and the new permeable membrane brings these enzymes into close proximity of the fat hence starting lipolysis (Scott, 1998). Heat treatment of raw milk or homogenised milk immediately after homogenisation would deactivate the lipase enzymes and prevent lipolysis (Deeth and Fitz-Gerald, 1976; Varnam and Sutherland, 1994).

Lactic acid bacteria are added to the milk in order to break down the milk sugar called lactose. Lactic acid is produced as a result of the break down, which lowers the pH. The type of lactic acid bacteria added depends on the type of cheese to be produced. Achieving the correct pH is vital in cheese making as different enzymes have different optimum pH at which they are most active, different enzymes contribute to different characteristics of a cheese. High acidic or basic conditions cause denaturation of enzymes (Gunasekaran and Mehmet, 2003).

For cheeses with whey drainage the pH at drain is of critical importance in determining the characteristics of the cheese. As the pH decreases the colloidal calcium phosphate dissolves and is lost in the whey (Fox, 2004). The calcium concentration of the cheese affects the casein matrix, hence a decrease in calcium results in weaker bonding between the casein micelles which produces a weaker gel (Sharma et al, 1993).

As the required pH is obtained rennet (enzyme) is added. Enzymatic coagulation occurs through the addition of rennet. Coagulation is formation of a milk gel by milk proteins coming together. This occurs by the action of a proteolytic enzyme present in the added rennet and the lowering of pH below the isoelectric point of the milk proteins to around pH 4.6 (Gunasekaran and Mehmet, 2003).

After addition of the rennet and allowing a setting time, the formed curd (milk protein gel) is cut into the required size and shape. The shaped cheese blocks are salted with dry salt then drained for any whey, if present (Gunasekaran and Mehmet, 2003). After draining, the cheese blocks are dipped in a brine solution for final maturation. The cheese blocks are then packed and cold stored.

2.4.2 Composition of feta cheese

Tamime (1991) specified that feta cheese made from cow's milk which was ultrafiltered to a concentration factor of 5.5 will have total solids of 42% and 18.5% fat. All measurements were on a % by weight basis. The protein content was not reported. Food Standards Australia New Zealand (2006) defines that feta cheese made either from cow, goat or sheep milk should have 47.1% total solids, 22.8% fat and 17.4% protein. All measurements are on a % by weight basis.

2.4.3 Benefits of Ultrafiltration in cheese making

Approximately 20% of the total protein and some of the fats in milk are lost in the whey in the traditional cheese making process (Atra et al., 2004). Ultrafiltration is utilised to concentrate milk to the final cheese composition, therefore, the amount of whey removed can be reduced or eliminated. Retention of whey proteins in the concentrated milk, from ultrafiltration, results in an increase in the overall cheese yield obtained in comparison to the traditional method (Pal, 2003).

In the traditional cheese making process 70% the enzyme added for coagulation is lost in the whey (Bech, 1993), with the application of an ultrafiltration process to cheese making whey removed can be minimised or eliminated, therefore the amount of enzyme used is reduced with the inclusion of ultrafiltration (Bech, 1993). The curd-cutting step can be eliminated when ultrafiltered milk is used as no whey is expelled from the curd.

A decrease in whey production reduces the amount of waste generated from the cheese making process. The permeate stream which is enriched with lactose can be further processed to extract the lactose which can be utilised in the sweets or pharmaceutical fermentation industries (Atra et al., 2004). The lactose rich permeate stream could also be used for other useful applications such as feed for pigs. Elimination of the presence of lactose and whey proteins in the waste stream decreases the biological oxygen demand of the stream; it can therefore be discharged into the sewage wastewater (Atra et al., 2004). All the above-mentioned outcomes make the cheese making process with ultrafiltration more efficient.

Other advantages of having whey proteins in cheese is that whey proteins have high nutritional value, therefore, the presence of whey proteins in cheese increases the nutritional value of the cheese (Bech, 1993). Vitamin B12 and folic acid are bound to protein in milk hence concentration of proteins through ultrafiltration results in an increase in the concentration of these vitamins. Vitamins D, E and K are bound by fat, hence increases in the concentration of these vitamins also occurs in the ultrafiltered milk. The increase in concentration of various minerals and the presence of whey proteins increases the nutritional value of the cheese, therefore, improves the quality of the cheese (Pal, 2003).

2.4.4 Limitations of ultrafiltration in cheese-making

The most significant limiting factor in ultrafiltration of dairy products is flux decline over time caused by fouling and concentration polarisation (Rao, 2002). Barros et al (2006) looked at the impact of a low concentration factor during ultrafiltration on composition and yield of reduced fat prato cheese. They used a hollow fibre membrane with 10kDa molecular cut-off and concentration factors of 1.0, 1.5 and 2.0. It was found that the different concentration factors had no significant effect on the chemical composition of the cheeses. It was also found that as the concentration factor increased higher fat and protein recoveries were obtained, hence increasing the yield with increasing concentration factor. The effect of the concentration factor on permeate flux was not considered.

Research on flux patterns in ultrafiltration of whole milk, skim milk, butter milk and sweet or acid whey by Rao (2002) shows that whole milk had lower initial flux in comparison to the flux generated from whey filtration. Rao (2002) found that feed with high protein concentration such as whole milk formed a concentration polarisation layer within seconds after the start hence lower initial flux. It was also found that the permeate flux drop over the first hour of operation was minimal although an increase in fouling coefficient was observed. The limitations of this work are that the experiments were conducted on total recycle mode hence the effect of change in viscosity over time was not taken into account. The flux patterns beyond the first operating hour were not evaluated.

2.4.5 Factors affecting the quality of cheese

Calcium plays a vital role in the coagulation of milk proteins: it is a standard practice to add $CaCl_2$ to milk to increase the concentration of soluble, colloidal and ionized calcium (Fox, 2004). Calcium hardens the protein deposit on the membrane surface and considerably increases the resistance of the deposited layer, causing a decrease in the permeate flux (Rao, 2002).

Results from a study on the influence of pH and sodium chloride (NaCl) on the rheological properties showed that a decrease in pH leads to an increase in solubility of calcium phosphate and ionic strength (Rao, 2002; Karlsso et al., 2007). Ionic strength was adjusted by addition of NaCl. The increase in solubility of calcium phosphate and increasing ionic strength resulted in an increase in the level of bonds and interactions between the casein micelles (Fox, 2004), therefore, showing an increase in the stress fracture of casein gels. However the increased level of calcium phosphate could have secondary effects with respect to the microstructure of cheese, which were not investigated as part of the study (Karlsso et al., 2007).

Salt (NaCl) is used in cheese production as a preservative as it selectively inhibits microflora and has a significant effect on many enzyme activities. As salt directly contributes to flavour and affects the microflora and enzymes, it is vital that a uniform concentration of salt is achieved throughout the cheese in order to obtain good quality (Fox, 2004).

The coagulant is mostly responsible for the texture of the cheese during the early stages of the ripening process. The most commonly used coagulant is rennet, an increased amount or an inaccurate amount of rennet leads to bitter tasting cheese. As rennet has a significant effect on both texture and functionality of cheese it is important that rennet activity is monitored. The retention of rennet in cheese is affected by various parameters such as pH, moisture content in cheese and temperature (Fox, 2004).

Ripening temperature is also known to have a strong influence on the quality of the cheese. Ripening temperature varies depending on the type of cheese to be produced. The ripening temperature defines the reactions that will take place,

hence increasing the ripening temperature increases the rate of the desirable reactions. The downside to increasing the ripening temperature is that it also increases the undesirable reactions, therefore, an unbalance in flavour or off-flavour maybe noticed in the ripened cheese (Fox, 2004).

The other important characteristic that affects the quality of cheese is the type of starter culture used. Some strains of starter culture produce bitter and unsatisfactory cheese, therefore it is vital that the correct type of starter culture is used for the specific cheese that is to be made (Fox, 2004).

2.4.6 Effect of temperature on cheese-making

High heat treatment of milk results in low bacterial load in milk increasing the shelf life of the final product. Above a temperature of 65 ºC whey proteins present in milk begin to denature by unfolding of their polypeptides. The unfolded whey protein can either interact with casein micelles or aggregate with itself (Singh and Waungana, 2001). The interaction of denatured whey protein forms a complex with kappa-casein (k-casein) on the surface of the casein micelles. The whey protein/k-casein complex looks like a thread with appendages protruding from the casein micelles (McMahon et al., 1993; Singh and Waungana, 2001).

Figure 10 shows the casein micelle and casein submicelle structure. Rennet coagulation of milk depends on cleavage of the phe104-met106 bond of k-casein located on the casein micelle surface (McMahon et al., 1993). The formation of the whey protein/k-casein micelle complex makes the phe104-met106 bond less accessible for cleavage, hence interferes with the aggregation of casein micelles. Thus the presence of whey protein increases the coagulation time and decreases the strength of the gel formed (Singh and Waungana, 2001). Whey proteins have a higher water holding capacity, therefore, retention of whey proteins results in higher moisture content and finer curd (Erdem, 2004).

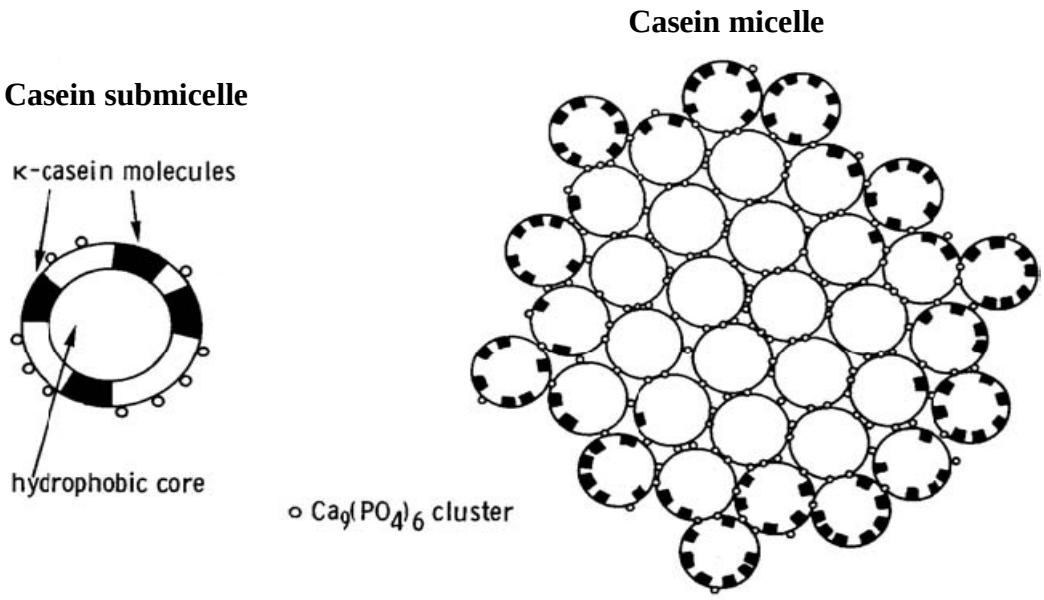

Figure 10. The structure of casein micelle and casein submicelle (Farrell et al., 2006).

Heating causes changes to the calcium phosphate equilibrium by transferring soluble calcium and phosphate into the colloidal phase. Calcium is required to attach the casein protein micelles together and it also influences the extent of denaturation of whey proteins (Singh and Waungana, 2001).

2.5 Chemical analysis

2.5.1 pH

pH needs to be monitored constantly throughout the cheese making process as at certain pHs specific microbial organisms and enzymes are active (Gunasekaran and Mehmet, 2003). If the pH of the cheese does not decrease at the required rate or to the desired extent during production, it is likely due to contamination through bacteria in the milk or phage attack (Fox, 2004). The rate of pH drop is critical in determining cheese characteristics. Therefore, pH needs to be checked to ensure that the required enzymes or microorganisms are active for the kind of cheese that is to be produced. pH is measured using a pH-meter with a glass electrode.

Robinson and Tamime (1991) state that to achieve good quality feta cheese the starting pH of milk should be greater than 6.55. To achieve a moisture content of less than 56%, the pH value after brining the feta cheese and storing for 2 weeks

should be between 4.4-4.6 (Robinson and Tamime, 1991). According to Lillevang (2004) the minimum pH can be achieved within 24 hours of addition of starter culture, which for feta cheese is around 4.6.

2.5.2 Milk Composition

It is important to determine the composition of milk as it has a significant effect on the quality of cheese made (Fox, 2004). Cow's milk is made up of approximately 4% fat, 3.3% proteins, 4.6% lactose, 0.75% salts, 0.05% phospholipids and 87% water (Scott, 1998; Pal, 2003; Brans et al., 2004). Infrared spectrometry can be used to measure fat, protein, lactose and total solids in a single test (Hall, 2007) or different tests for each individual component present in milk can be used to determine the milk composition. Changes in milk composition especially protein content of milk affect the quality and composition of cheese made (Pal, 2003).

2.5.3 Fat in cheese

It is important to check the fat content in the cheese made, as fat significantly contributes to the flavour of the cheese. Fat content could affect the texture of the cheese made. Fat percent can be determined by any of the following tests, Babcock test, modified Schmid-Bondzynski-Ratzlaff method or Gerber test. The Gerber method utilises sulphuric acid to dissolve the non-fat solids and the mixture is centrifuged, the fat percentage is read off the graduated scale present on the butyrometers. In the modified Schmid-Bondzynski-Ratzlaff method fat is extracted with diethyl ether and petroleum ether, the fat content is determined by evaporating the solvent and weighing the fat extracted (Ceirwyn, 1995).

2.5.4 Protein

Protein present in cheese is determined to check for the quality of cheese and to ensure the cheese contains the level of protein specified in the nutritional label present on the packaging. Protein concentration is most commonly determined by the Kjeldahl method. The other methods include titration, gasometric, colorimetric, spectroscopic and dye binding tests (Ceirwyn, 1995; Scott, 1998; Fox, 2004).

The Kjeldahl method is known to produce accurate results. The Kjeldahl method estimates the nitrogen content of cheese, which is converted to the protein content based on the conversion factor for cheese of 6.38. The Kjeldahl method converts nitrogen in food to ammonium sulphate which is then converted into ammonia gas through heating with sodium hydroxide in the presence of steam. The ammonia gas is collected into excess boric acid solution to form ammonium borate which is titrated with standard hydrochloric acid to determine the nitrogen content (Ceirwyn, 1995).

2.5.5 Acidity

Acidity specifies the buffering capacity, therefore, it is monitored to ensure good quality cheese is produced. It is a standard practice in cheese manufacturing process to measure the acidity of milk, although pH is now more commonly used. Acidity in milk is determined by a titration method known as the Dornic method (Scott, 1998) or the method specified by the AOAC standard methods (AOAC International, 2005).

2.5.6 Salt

Salt contributes significantly to the taste of cheese. It also decreases moisture present in cheese. It is vital the moisture in cheese is controlled as different levels of moisture define the hardness of the cheese (Fox, 2004). Salt acts as a major inhibitor towards microbial activity and has a significant effect on the activity of enzymes in cheese. Through the effect on microbial activity and enzyme activity salt contributes considerably towards the ripening, flavour and quality of cheese (Fox, 2004). Salt in cheese is determined using the modified Volhard test. In the Volhard method salt in the cheese reacts with excess standard silver nitrate solution and the unreacted silver nitrate is titrated with potassium thiocyanate to determine the salt content in cheese (Ceirwyn, 1995).

2.5.7 Total Solids (TS)

Total solids consist of fat and non-fat solids (non fat solids consists of protein, lactose and minerals). TS content is determined by drying a cheese sample at 105ºC until constant weight is achieved. Total solids percent is used to determine the moisture content of cheese. It is important to observe the moisture content in cheese as it contributes to the softness of the cheese. Deviation of moisture

content could affect the appearance and taste of the cheese (AOAC International, 2005).

2.5.8 Lipase activity

Lipolysis is the process of fat hydrolysis. Fat contains lipophilic flavour compounds, which develop or are released by microbial or enzymatic action through oxidation, decarboxylation and reduction of decarboxyl compounds (Gunasekaran and Mehmet, 2003).

Lipolysis is an important biochemical process which occurs during cheese ripening (Georgala et al., 2005; Svensson et al., 2005). This process produces fatty acids which are essential for flavour development in cheese (Fox, 2004). The amount of free fatty acids (FFA) produced in cheese during ripening is considered as an overall measure of lipolysis (Svensson et al., 2005). The accumulation of FFA during cheese ripening is variable depending on the type of cheese, ripening time and manufacturing technology (Georgala, 2005; Svensson et al., 2005). Long chain FFAs (greater than 12 carbon atoms) play a minor role in cheese flavour (Gunasekaran and Mehmet, 2003), short chain FFAs can release both desirable and rancid flavours to the cheese (Chavarri et al., 1997). As free fatty acids (FFA) make significant contribution to the sensory characteristics of the cheese, an accurate determination of FFA is important (Chavarri et al., 1997).

Determination of FFA in cheese is complicated as the FFAs are present in very small quantities, they represent <0.5% of total fat and the short chain FFA are extremely volatile (Chavarri et al., 1997).

Due to the above mentioned difficulties, the analysis of FFA usually involves the separation of FFA from the bulk extracted fat by ion-exchange chromatography (Chavari et al., 1997; Fox, 2004; Svensson et al., 2005; Deegan, 2007). FFA content can also be determined by using a solvent to extract the fat fraction from cheese, then preparation of methyl esters of FFA. The methyl esters are separated and quantified using gas chromatography (Chavarri et al., 1997; Fox, 2004). Quantification of the total level of FFA can also be achieved by extraction and titration of FFA by alcoholic potassium hydroxide (Deeth and Fitz-Gerald, 1976; Fox, 2004). The results from the extraction and titration method are expressed as

number of milliequivalents of alkali required to neutralise the FFAs in 100 g of fat also known as the acid degree value (ADV) (Fox, 2004).

2.6 Rheology and texture Analysis

Rheology is the study of deformation and flow of materials under stress or strain, where as texture refers to the hardness, firmness, springiness, crumbliness or adhesiveness of a material. The deformation of materials is associated with its elasticity, viscosity and viscoelasticity, which also defines the texture in terms of firmness and hardness. Therefore rheological analyses can be used as a means to measure the texture of cheese (Fox et al., 2000).

There are various tests specified in literature such as penetration tests, compression tests, creep/stress relaxation tests and large-scale deformation tests (Fox, 2004). All methods induce stress in different ways and measure the effect of stress/strain. The most common process of rheological analyses was found to be the texture profile analysis.

This method involves placing a sample between two plates where the top plate is set to move down at a pre-set rate until the sample is compressed to the required height (Fox et al., 2000; Fox, 2004). Measurements are taken to generate a compression chart from which the fracturability, firmness, springiness, cohesiveness, gumminess, chewiness and adhesiveness are calculated based on the height of peaks and area under the curves (Fox, 2004).

2.7 Microbiology

The contamination of cheese by bacteria has led to a number of outbreaks of food poisoning all over the world (Varnam and Sutherland, 1994). It is vital that microbiological analysis is performed on cheese to detect any hazards to public health, off-flavours and spoilage. Microbiology is also used to detect the level of desirable bacteria present in cheese and milk (Fox, 2004).

Soft unripened cheese is required to be checked for spoilage micro-organisms, spoilage occurs through growth of yeasts and moulds. Therefore, to detect the likely spoilage of cheese it is tested for the presence of yeasts and moulds. Soft cheeses should be analysed for presence of diarrhoeagenic strains of *E.coli,*

Staphylococcus aureus and *Listeria monocytogenes* (Varnam and Sutherland, 1994). Coliforms can be used to identify any post process contamination of cheese. Coliforms are also used as an indicator of sanitation conditions during the processing of milk and as a check for re-contamination after pasteurisation (Tatini and Kauppi, 2002).

2.8 Sensory Analysis

Sensory properties are associated with flavour, texture, aroma and appearance. These are the aspects that define consumer acceptability and bring customers that are loyal to a certain cheese. The sensory properties are complex and defined by interaction of many structural and compositional properties. Slight changes in the sensory properties are detected by the sophisticated human sensory system. The changes in sensory properties are contributed by compounds that are present in very low concentrations. The low concentration of compounds makes detection hard even with state of the art analytical equipment. Hence various sensory evaluation techniques have become a common practice in the dairy industry (Delahunty, 2002).

The difference between each sensory evaluation technique is the objective to be achieved. The five different techniques are listed below (Fox, 2004).

1. Quality scoring.
2. Discrimination testing.
3. Descriptive testing.
4. Time-intensity testing.
5. Consumer acceptability testing.

2.8.1 Quality Scoring

Quality scoring also known as the grading practice is the formal sensory evaluation in the dairy industry for over 50 years (Delahunty, 2002). The objective of this sensory evaluation technique is to determine defects in the sample cheese and define how much they deviate in quality from the traditional cheese. This method consists of grading certain aspects that are of significant importance in a particular cheese. The aspects to be graded are associated with aroma, flavour, texture, taste and appearance of the cheese (Delahunty, 2002).

The scale that is to be used for grading varies depending on the importance of that particular aspect for a specific cheese, generally the scale is either 1 to 5 or 1 to 10 (Mallatou et al., 1994; Pappas et al., 1994). The grading practice works on the basis that for each defect observed for a certain aspect of cheese one point is to be deducted, so when the total grade for all the aspects is added it can easily be determined how much the quality of the sample cheese deviates from that of the traditional cheese. It is to be noted that although the defects of the cheese are acknowledged, the severity of the defect is not quantitatively measured (Delahunty, 2002).

A trained panel performs the grading. Previous knowledge of the traditional cheese is of significant importance in order to obtain accurate results and train the panel. Once the panel is selected the descriptive sensory terms that are to be used to grade the cheese are established. The panel is generally made up of five to 12 members (Bakker, 1997).

2.8.2 Discrimination testing

Discrimination testing is used to determine the differences if any that exist between samples for a specific sensory characteristic. The commonly used discrimination tests are paired comparison test, duo-trio test, triangular test and the ranking test (Delahunty, 2002).

In a paired comparison test two samples are tested for each sensory attribute. The assessors are asked to determine which of the samples shows the greater tendency for the sensory characteristic in question. In the duo-trio test, two samples and a reference sample are tasted. Assessors are asked to identify which of the samples is more like the reference sample. The triangular test utilizes three samples of cheese, assessors are to choose which of the two samples are alike. The ranking test involves a number of samples compared against each other for a specific sensory characteristic, assessors are asked to rank the samples from the highest intensity of the attribute to the lowest (Delahunty, 2002).

Although this method of sensory evaluation does not require the panel to be trained, it requires the panel members to have a strong sensibility in order to differentiate the differences accurately. The panel is not strictly limited to a

certain number of people. This sensory evaluation technique is better suited for research conditions as it eliminates the subjectivity associated with the expert evaluators (Delahunty, 2002).

2.8.3 Description testing

Descriptive testing is similar to discrimination testing. The differences in the discrimination testing are identified whereas in descriptive testing the differences are identified and quantified (Delahunty, 2002).

Implementation of descriptive testing consists of three stages.

- Selecting a panel of assessors.
- Determining the sensory terminology or a vocabulary to describe the characteristics that define the cheese.
- Defining the process to quantify the differences between the samples for a specific characteristic.

There are various different methods of descriptive testing. The method to be utilised is chosen based on the product to be tested, potential time available for testing and the financial investment on product testing. The rapid method of description testing is known as the quantitative description analysis (QDA). This method does not require a specific definition or reference of the sensory characteristics (Delahunty, 2002). The most time consuming method of description testing is the spectrum method, this method requires previous knowledge of the sensory characteristics of the product to develop a standardised method of analysis. Therefore the spectrum method requires several meetings held with in the assessors, which makes this method a time consuming process (Delahunty, 2002).

2.8.4 Time-intensity measurements

The Time intensity measurement technique consists of continuous scaling or recording of the intensity of an attribute until the intensity is zero or the recording period has stopped. This method requires a computer software to collect the data and the assessors are to be trained with the method used to record the level of intensity. Time-intensity measurement method is very time consuming hence sometimes it is used in conjunction with qualitative descriptive analysis to narrow

down the number of sensory characteristics that require further analysis (Bakker, 1997).

2.8.5 Consumer acceptability testing

Consumer acceptability testing is used when making developments or changes to an existing product or designing a new innovative product. This method of testing is similar to discrimination testing, in consumer acceptability testing the preference between samples is measured. As the panel is untrained consumers, the results obtained are not reproducible (Delahunty, 2002).

2.9 Conclusions

- The four pressure driven membrane filtration processes are reverse osmosis, nanofiltration, ultrafiltration and microfiltration.
- The pressure driven membrane filtration processes are classified based on the size of the components passing through the membrane.
- Ultrafiltration membranes come in plate, spiral wound or tubular (either self supported like hollow fibre or supported by other means) forms.
- Important parameters affecting the performance of ultrafiltration are transmembrane pressure, temperature, turbulence and concentration of the feed.
- The main advantages of spiral wound membranes are the ability to attain turbulent flow, low membrane replacement costs, low capital costs and high surface area to volume ratio.
- The disadvantages of spiral wound membranes are high pressure drop and no back flushing facility.
- The main advantages of hollow fibre are low hold up volume, back flushing capacity and low energy costs.
- The disadvantages are operation in the laminar flow region hence sensitivity to fouling, low operating pressures, membrane replacement and plant investment costs are high.
- Increasing transmembrane pressure and cross flow velocity results in an increase in permeate flux generated.
- Increasing feed concentration causes a decrease in permeate flux due to greater concentration polarisation effect.

- Increase in temperature results in a decrease in viscosity, hence an increase in permeate flux.
- Cheese-making incorporating ultrafiltration requires no whey draining, therefore increase in the overall yield from inclusion of whey proteins.
- Raw milk is pasteurised to destroy harmful micro organisms and deactivate enzymes such as lipase, then homogenised for fat stabilisation.
- Lactic acid bacteria added to homogenised milk to break down lactose and lower the pH of milk, rennet is added for coagulation of casein micelles.
- Activity of enzyme is dependant on pH, pH also affects the solubility of colloidal calcium in milk, which affects the structure of the casein matrix.
- Feta cheese composition made from either cow, goat or sheep's milk should be 47.1% total solids, 22.8% fat and 17.4% protein on weight basis.
- The rate of pH drop in cheese-making is critical for the characteristics of cheese, pH should be measured using glass electrode pH meter.
- The composition of milk especially protein and fat content affect the quality of cheese produced, hence need to be checked.
- Protein can be measured using the kjeldahl method, which is the most accurate method of testing for protein.
- Fat and salt contribute to flavour of the cheese, they can be checked using the modified Schmid-Bondzynski-Ratzlaff method and modified volhard method respectively.
- Total solids is used to determine the moisture content in cheese, moisture needs to be monitored as it affects the softness of cheese. The total solids can be determined using the air oven drying method.
- Lipolysis is a measure of free fatty acids which are essential for flavour development, the lipase activity can be measured using the acid degree value method.
- The microbiology of cheese needs to be checked to ensure the cheese is safe to consume.
- The texture of cheese is determined using the texture profile analysis.
- Various sensory methods exist, depending on the objective to be achieved the appropriate sensory evaluation technique can be chosen.

3.0 Cheese-making at Puhoi Valley Cheeses

The feta cheese production at Puhoi Valley Cheeses (PVC) from raw milk to cheese packing stage is summarised in the process flow diagram as shown in Figure 11 below.

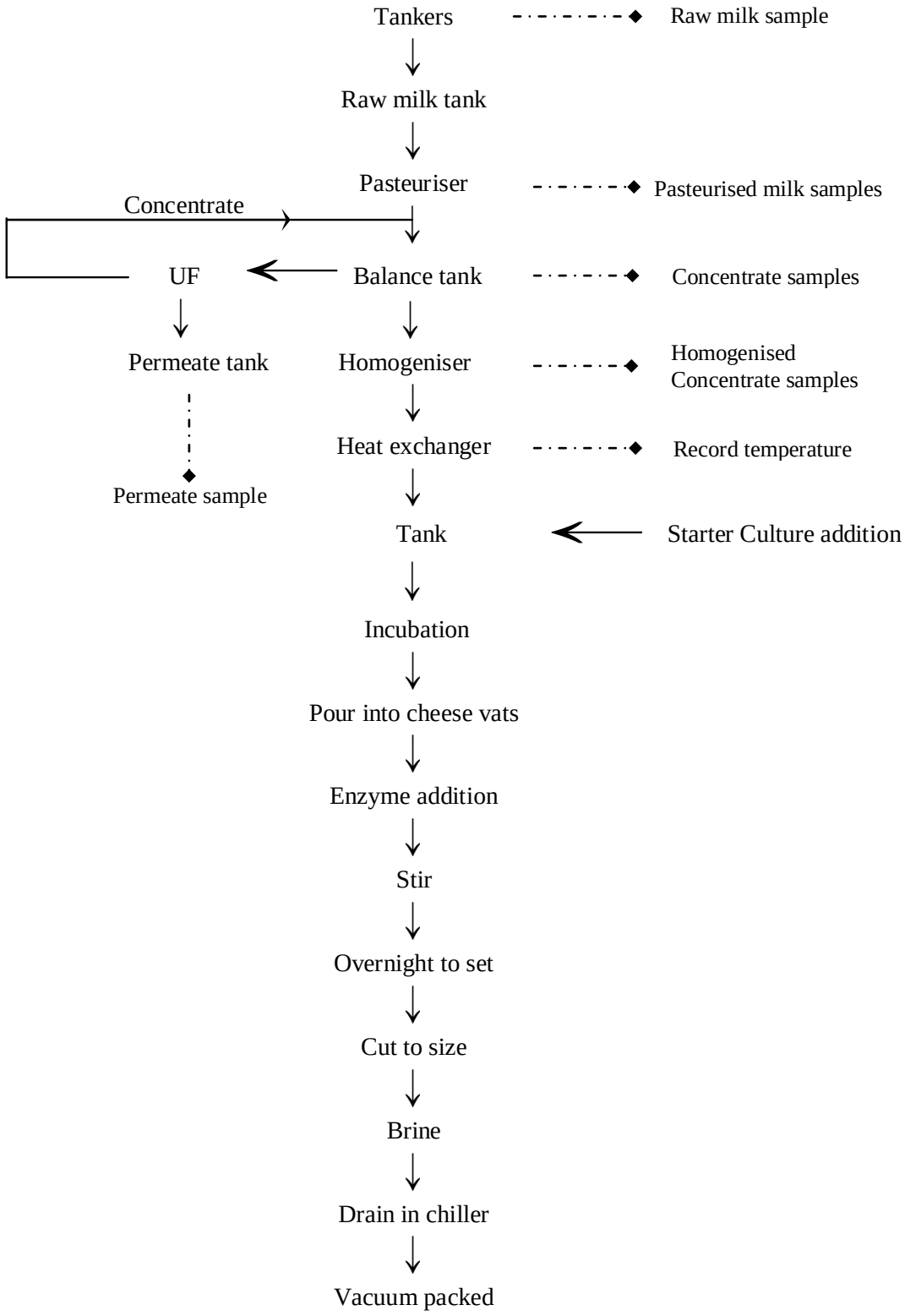

Figure 11. Flow chart of cheese production from raw milk delivery to packaging as at Puhoi valley cheeses..

3.1 Raw milk

Raw milk (supplied by Fonterra Co-operative Group) from the tankers is tested for pH, total solids (TS) and titratable acidity (TA) by the operators. The pH of raw milk should be greater than pH 6.60, TS should be approximately 13% $^{w}/_{w}$ and TA measured as % lactic acid should be less than 0.17% $^{w}/_{v}$. Raw milk is also tested for coliforms, aerobic plate count (APC), fat content, protein content and %TS again by the microbiology testing staff.

TS can be found using a Foss Milko-scan^{TM} S50 (for low viscosity milk) or CEM Microwave Lab-wave 9000 (for higher viscosity or higher TS milk). The Milko-scan is used to determine the composition of milk in terms of fat, protein, lactose and total solids. The lab-wave is a microwave oven used to rapidly determine the total solids of concentrated or viscous milk. The lab-wave microwave oven drying method requires the sample milk to be placed on the paper pads (to eliminate spattering of sample during drying) and selecting the setting for the specific application (milk drying) as defined by the manufacturer. The lab-wave 9000 calculates the TS in the sample as a percentage of the weight of the specific sample.

TA is found by titration with 0.1 M sodium hydroxide (NaOH) based on the AOAC method with the following modifications. The milk sample is not diluted with water at PVC and the results are expressed as $^{w}/_{v}$ where as AOAC method expresses results in terms of $^{w}/_{w}$ basis (AOAC International, 2005). The titratable acidity was determined by using 20mL of milk which is titrated with 0.1M NaOH solution to reach the end point of a persistent pink colour, using Equation (1), if the volume of 0.1M NaOH titre is 3.2mL then the TA is calculated to be 0.144% $^{w}/_{v}$ for a batch of raw milk.

$$\text{TA} = \frac{\text{mL of NaOH} \times 0.009\ \text{g of lactic acid}}{20\ \text{mL of milk}} \times 100 \qquad (1)$$

Maximum TA is 0.17% lactic acid ($^{w}/_{v}$).

Raw milk from the tankers is released to the site once the pH is confirmed (pH > 6.60) and the TA is checked by the operators.

3.2 Pasteurisation

Raw milk is pasteurised for 15s at 76°C in a plate heat exchanger, the pasteurised milk is then collected into a balance tank as the rate of pasteurisation is faster than the rate of ultrafiltration. Most commonly milk is pasteurised at 72°C for 15s, however, Gunasekaran and Mehmet (2003) state that pasteurisation above 75°C results in a slight reduction in whey expulsion. Samples of pasteurised milk are collected and tested for phosphatase at the start, middle and end of the pasteurisation stage for each batch of milk to ensure proper pasteurisation of milk.

The test for pasteurisation also known as phosphatase test consists of adding a buffer substrate to the sample and incubating for 2 hours. The colour change after incubation is compared against a control (raw milk sample) and the lovibond scale. The lovibond scale consists of different intensities of the colour where the intensity of the colour is proportional to the amount of phosphatase present.

Pasteurisation kills all the harmful bacteria, therefore there is a safety measure in place to avoid unpasteurised milk being used in the cheese production process. If it is detected that the temperature of the milk has not reached the required temperature the unpasteurised milk is diverted to be collected back into the pre-pasteurisation tank.

3.3 Ultrafiltration

The pasteurised milk at a temperature of 45 ± 5 °C is passed through the UF unit. The UF unit at PVC consists of two banks of 12 hollow fibre cartridges, Figure 12 shows one bank of cartridges and the direction of the flow, each cartridge is 0.127 m in diameter, 1.092 m in length and have 6.1 m^2 of membrane area (Total membrane area of the PVC UF plant is 146.4 m^2). The hollow fibre membranes are supplied by Koch with a molecular weight cut off (MWCO) of 50 kDa and 60 mil (one thousandth of an inch) gap/spacing. The membranes are designed to be operated at a maximum inlet pressure of 2.8 bar, maximum transmembrane pressure of 2.4 bar at 25°C and maximum temperature of 60°C at a pH of 6.

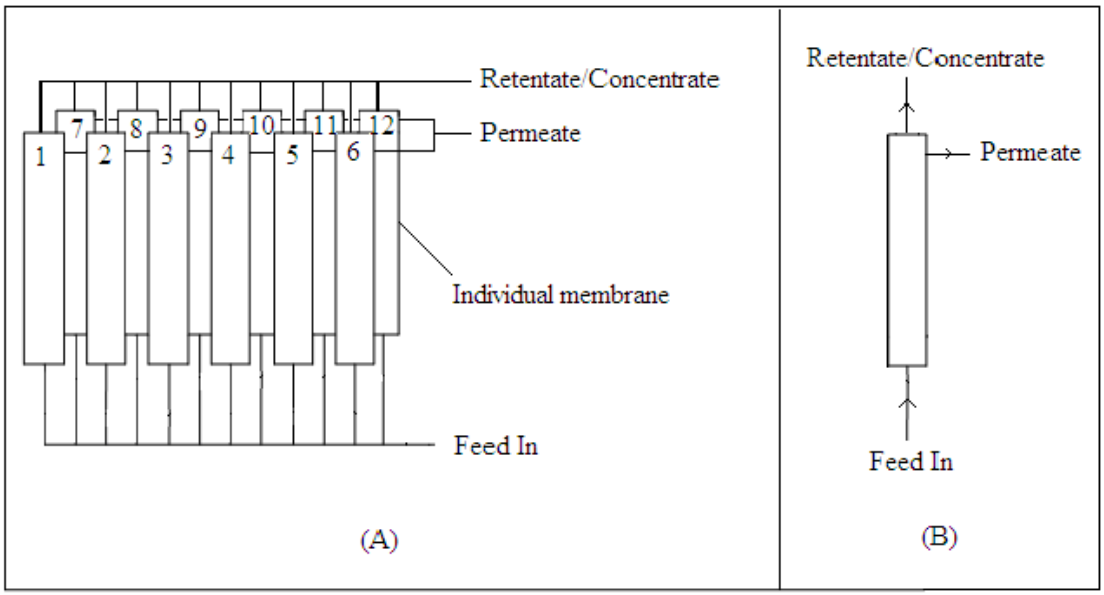

Figure 12. (A) The layout of one bank of 12 hollow fibre cartridge of the ultrafiltration system as at PVC. (B) The direction of the flow in, permeate and retentate/concentrate.

The UF is operated under recycle mode as a batch process, where part of the concentrate stream is recycled back to the feed tank. Hence over time the pasteurised milk increases in viscosity and TS. The milk concentrate is recycled through the UF unit until a TS of 37.5 ± 0.5 % is achieved. The TS% is checked by the lab-wave oven when the estimated concentrate volume is approximately reached. Samples of concentrate and permeate are collected at the start, middle and end of ultrafiltration for laboratory analysis. Permeate is collected in a tank which is then sold as feed for pigs.

The inlet pressure (P_{in}), outlet pressure (P_{out}), feed temperature, total solids percentage, recycle rate flow and the permeate flow rate were measured every 20 minutes for the duration of the ultrafiltration run. The pressure difference along the membrane (ΔP) and the transmembrane pressure (TMP) are calculated using Equations (2) and (3). The TMP is calculated as shown in Equation (3) by determining the difference between the pressure on the retentate side and the permeate side, the pressure on the retentate side is the average pressure across the membrane. The pressure on the permeate side is atmospheric. The inlet and outlet pressure are maintained at 2.1 and 1.1 bar respectively. The ΔP and the TMP represented for each run are the average values.

$$\Delta P = P_{in} - P_{out} \quad (2)$$

$$TMP = \frac{P_{in} + P_{out}}{2} - P_P \quad (3)$$

Where P_{in} is the inlet pressure on feed/retentate side

P_{out} is the outlet pressure on feed/retentate side

P_P is the pressure on the permeate side

3.4 Homogeniser and Heat exchanger

The concentrate at TS of 37.5 ± 0.5 % is homogenised at 400 psi. Samples of homogenised concentrate are collected at the start, middle and end. The homogenised concentrate is heated to a temperature between 78-80°C and cooled to 30-35°C using a plate heat exchanger. The heating and cooling temperatures of the heat exchanger are recorded every 10 minutes. The cooled homogenised concentrate from the heat exchanger is collected into a tank.

3.5 Starter Culture and Incubation

The amount of starter culture required for the batch is calculated using Equation (4) as derived by PVC. The equations were derived as easy to use instructions for operators, Equation (4) is based on the amount of starter required for a theoretical yield (concentrate) in grams. The variables in Equation (4) are calculated using Equations (5) and (6).

$$\text{Starter} = \frac{\text{Yield} \times \left(1\,\text{bag weight} - 10\ {}^{g}\!/_{\text{bag}}\right) \times 0.7}{500} \quad (4)$$

$$\text{Yield} = \text{Milk volume} \times \text{Concentration factor} \quad (5)$$

$$\text{Concentration factor} = TS \times 1.686 \quad (6)$$

Where:

- Yield (concentrate) in litres, bag weight (total weight of one starter culture bag with the packing) is in grams per bag, 0.7 is the units of starter culture

required per litre of concentrate, 500 is the number of units of starter culture per bag and 10 grams per bag is the weight of the packing of one starter culture bag.

- Milk volume in litres is the raw milk volume used for the batch.
- Bag weight is the weight of one starter culture bag in grams.
- TS is total solids in the raw milk, the constant 1.686 defined by PVC is used to determine the theoretical concentrate volume which would give the required total solids of 37.5% based on the initial total solids of the milk.

Two litres of Meadow Fresh family homogenised milk is heated to between 30-35°C, the calculated starter amount is then added to the heated milk and incubated for 20 minutes. After the 20 minutes incubation the milk with starter culture is added to the tank containing the homogenised milk concentrate and further incubated for 30 minutes in the tank at 30°C. The incubated concentrate from the tank is poured into the cheese vats to a height of 8.9 cm. The pH of the concentrate is checked to ensure it is between pH of 6.5 – 6.6 before addition of enzyme.

3.6 Enzyme

Traditionally rennet from the stomach of the calf is used as the enzyme for cheese production. At PVC Fromase® XL supplied by DSM Food specialities, dairy ingredients in the Netherlands is used as the enzyme, Fromase® XL is a microbial enzyme derived from a soil fungus hence is 100% vegetarian (DSM Food Specialities, 2007). The relative activity of Fromase® XL is 75% at between 30 - 35°C, the relative activity of Fromase® XL is the same as calf rennet in usual coagulation temperatures (up to 38°C). Above 38°C the relative activity of Fromase® XL and calf rennet differ. Both show an increase in relative activity above 38°C, however, the maximum relative activity of Fromase® XL is much lower and is achieved at a much lower temperature than that of calf rennet (DSM Food Specialities, 2007).

A Fromase® XL solution of 4% $^{v}/_{v}$ concentration is made by making up 40mL of concentrated Fromase® XL to 1L with autoclaved water. One millilitre of the 4% $^{v}/_{v}$ Fromase® XL solution is added per 1L of concentrate into the cheese vats. It is vital that the enzyme is distributed equally throughout the vat in order to avoid uneven texture. To obtain even distribution of the enzyme, the concentrate immediately

after enzyme addition is stirred well for four minutes. After stirring, the cheese vats with concentrate are left overnight without any movement in the setting room. The setting temperature affects the texture of the cheese produced, hence it is maintained between 20-24°C.

3.7 Cutting, brining and packing

The cheese, set overnight in the vats, is cut into 200g blocks using a wire cutter. The pH of the set cheese is checked to be between 4.6 – 4.9 before cutting to size. The maximum sodium chloride (NaCl) dissolvable in water is 26% $^{w}/_{w}$, also known as the saturated solution. The brine concentration used for salting cheese was between 80 – 90% (20.8 – 23.4 % $^{w}/_{w}$ NaCl brine solution) of the maximum dissolvable NaCl solution, it shall further be referred to as 80 – 90% saturated solution. The blocks of cheese are brined in approximately 90% $^{w}/_{w}$ saturated brine solution (NaCl) at 17ºC ± 2ºC for 90 minutes. After 90 minutes the cheese blocks are removed from the brine solution, covered with plastic bags and stored in a chiller at 5°C overnight to drain. The plastic bags eliminate the formation of dry crusty surfaces. The next day the cheese blocks are individually vacuum packed.

For each batch of feta cheese produced at Puhoi, the operators are required to fill out a data sheet to keep a paper track. The data sheet for feta cheese is attached in Appendix-A Data sheets.

4.0 Cheese making with pilot plant

The process to make cheese using the two pilot plant UF units was as shown below in Figure 13 is slightly different to the cheese-making process of PVC (Figure 11).

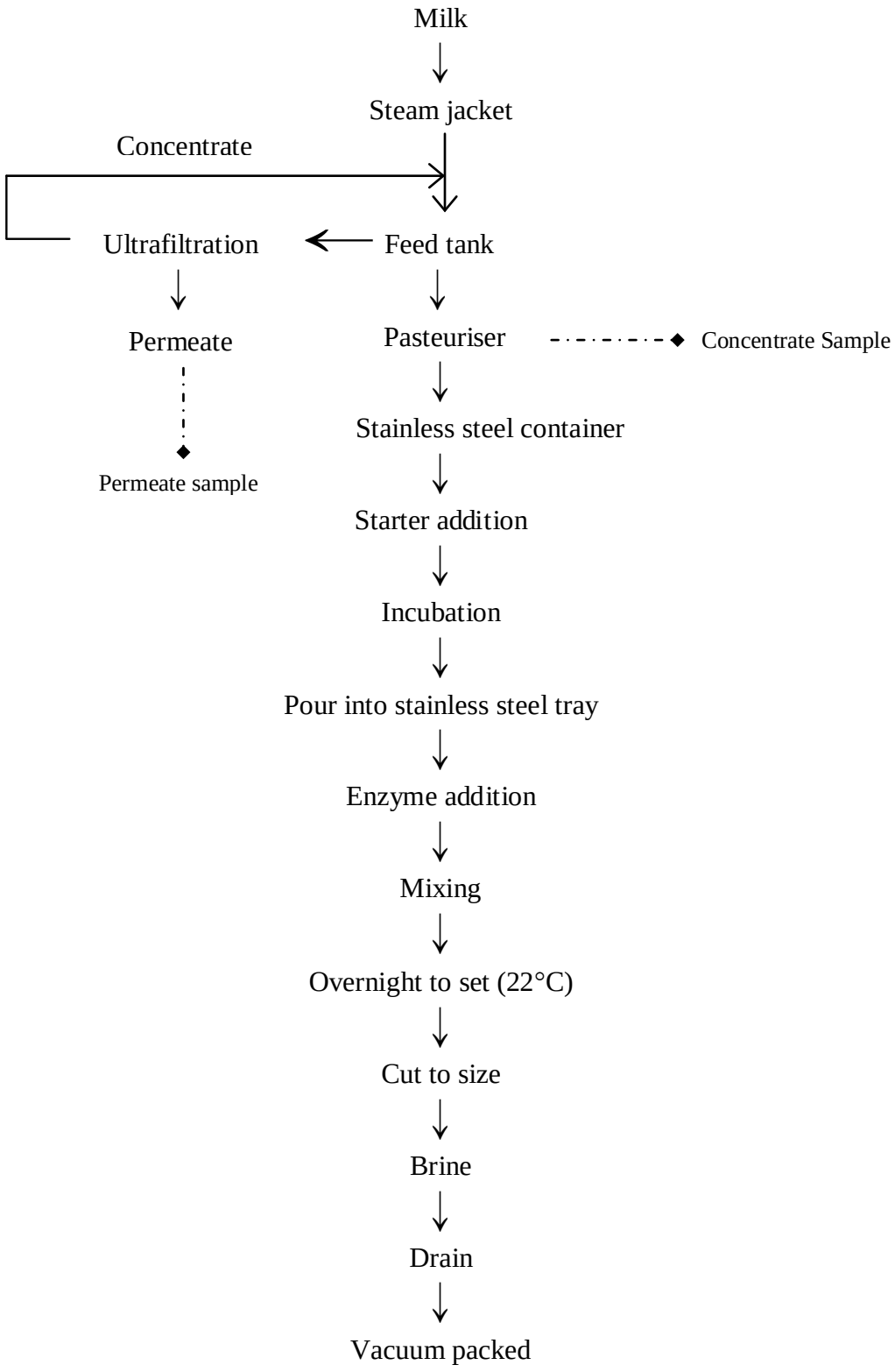

Figure 13. Flow chart of the pilot plant cheese making process utilised while using either hollow fibre or spiral wound ultrafiltration pilot plant units.

The difference in the process flow diagram of hollow fibre and spiral wound pilot plants and PVC is the milk concentrated on pilot plant units was homogenised therefore the homogenisation step was eliminated on pilot plant runs.

The milk for cheese production with the pilot plant units was supplied by the Goodman Fielder plant at Longburn. The milk supplied was standardised, pasteurised and homogenised blue top milk delivered in 10L bags. The milk (100 L for hollow fibre and 140 L for spiral wound unit) was heated to 45-50°C in a steam jacketed vessel and transferred to the feed tank on the UF pilot plant units. The temperature of the milk was maintained between 45-50°C during the filtration process by the heating jacket on the hollow fibre unit and by recirculating through the heat exchanger on the spiral wound unit. The experimental setup of the hollow fibre and spiral wound pilot plants is as shown in Figure 14 and 15, respectively.

The feed and concentrate volume, feed/retentate inlet and outlet pressures, feed and permeate flow rate (where applicable), operating temperature, run time, recycle rate (the feed fed back to the feed tank), pH before enzyme Fromase® XL addition, before cutting set cheese to blocks and pH of the final cheese blocks are shown in Table 1, 2 and 3 provided in the results and discussion section (Section 6.1) for both hollow fibre and spiral wound pilot plants. As comparison the above mentioned data for the PVC ultrafiltration systems is also provided in Table 1, 2 and 3.

The hollow fibre membrane used was made from polysulfone material by Koch membrane systems (supplied by APV – New Zealand, Te Rapa), it had a membrane surface area of 1.3 m^2, length of 0.635m, outer diameter of 0.0762m, 50KDa (MWCo), 60 mil gap. The model number was 3"HF14-60-PM50. The hollow fibre pilot plant was a UFS-1 Ultrafiltration pilot plant built by Alfa-Laval filtration systems. The spiral wound membrane was made from polyvinyldene fluoride (PVDF) and supplied by Synder Filtration (Larsram Limited, the NZ branch located in Cambridge). The spiral wound membrane had a membrane surface area of 3.6 m^2, length of 0.965m, outer diameter of 0.0965m, 50KDa (MWCo), 80 mil gap. The model number was BN-5B-3838. The spiral wound membrane was specifically chosen to be with a larger gap size to cope with the high viscosity due to higher total solids concentration. The spiral wound pilot plant was built by Massey University.

The pilot plants were operated on recycle mode where part of the concentrate stream was bled off to the feed tank and the rest of the stream recirculated and mixed with the fresh feed entering the unit. The TS% was checked every 15 minutes until the required 37.5 ± 0.5 % of TS was achieved. The TS% during the runs was determined using a refractometer. The detailed operating procedure of the ultrafiltration pilot plants is provided in Appendix-B. After the required TS% was achieved the concentrate was drained from the unit and pasteurised at 73°C for 15 seconds and cooled to 30-35°C. The pasteuriser used was custom built by Alfa-Laval for Massey University, the heating for the pasteuriser was achieved by a tubular heat exchanger using steam and the cooling was achieved by using cold water.

The amount of starter culture required was calculated using Equations (4), (5) and (6), as at PVC. The calculated amount of starter culture was added to 80mL of homogenised blue top milk heated to 30-35°C and incubated for 20 minutes. The incubated milk was then added to the concentrate and further incubated for 30 minutes in an incubator at 30 ± 1 °C. After 30 minutes the concentrate was poured into a stainless steel container to a height of 8.9 cm. The concentrated enzyme Fromase® XL obtained from PVC was diluted to 4% $^{v}/_{v}$ Fromase solution and added to the concentrate and stirred for four minutes, as at PVC. The stainless steel containers were then left in the incubator at 22 ± 1 °C overnight to set. The next day the pH of the set unsalted cheese was checked and cut to size to obtain 200g blocks of cheese. The blocks were brined in approximately 90% $^{w}/_{w}$ saturated brine solution (NaCl) at 17ºC ± 2ºC for 90 minutes. The brined cheese blocks were drained in the chiller (5 ºC) overnight as at PVC and vacuum packed the next day.

4.1 Experimental design

The most significant factor affecting the performance of the ultrafiltration membranes is the transmembrane pressure. Hence the effect of transmembrane pressure on the operation of the spiral wound pilot plant unit and the quality of cheese was investigated.

The hollow fibre unit was operated at a constant transmembrane pressure of 1.5 bar for all cheese made on this pilot plant, as the hollow fibre ultrafiltration system at PVC is operated at a transmembrane pressure of 1.6 bar. The hollow fibre pilot plant operation was to be an imitation of the PVC operating procedure. The cheese

made using concentrate from the spiral wound unit was produced at a transmembrane pressure of 1.0 or 1.3 bar.

The maximum transmembrane pressure to be operated on the spiral wound membrane was specified to be 1.3 bar by the supplier. Hence, the highest transmembrane pressure used on the spiral wound membrane was lower than that used at PVC. The effect of transmembrane pressure on the quality of cheese produced was to be investigated, therefore milk concentrated in the spiral wound unit was ultrafiltered at two different transmembrane pressures.

The experiments were planned to be repeated four times. Each replication consisted of three lots of cheese, two produced on the spiral wound unit at the two different transmembrane pressures mentioned above (Spiral DP1 @ 1 bar and Spiral DP2 @ 1.3 bar) and one from the hollow fibre unit. To ensure that the data gathered from the experimental design would produce accurate results in terms of statistics, a statistician was consulted. The statistician advised to randomise the experiments as this would enable us to identify any external factors that would affect the cheese production.

Two of the replicates were conducted a week apart in December 2007 and the cheese from the other two replicates was produced in January 2008 a week apart.

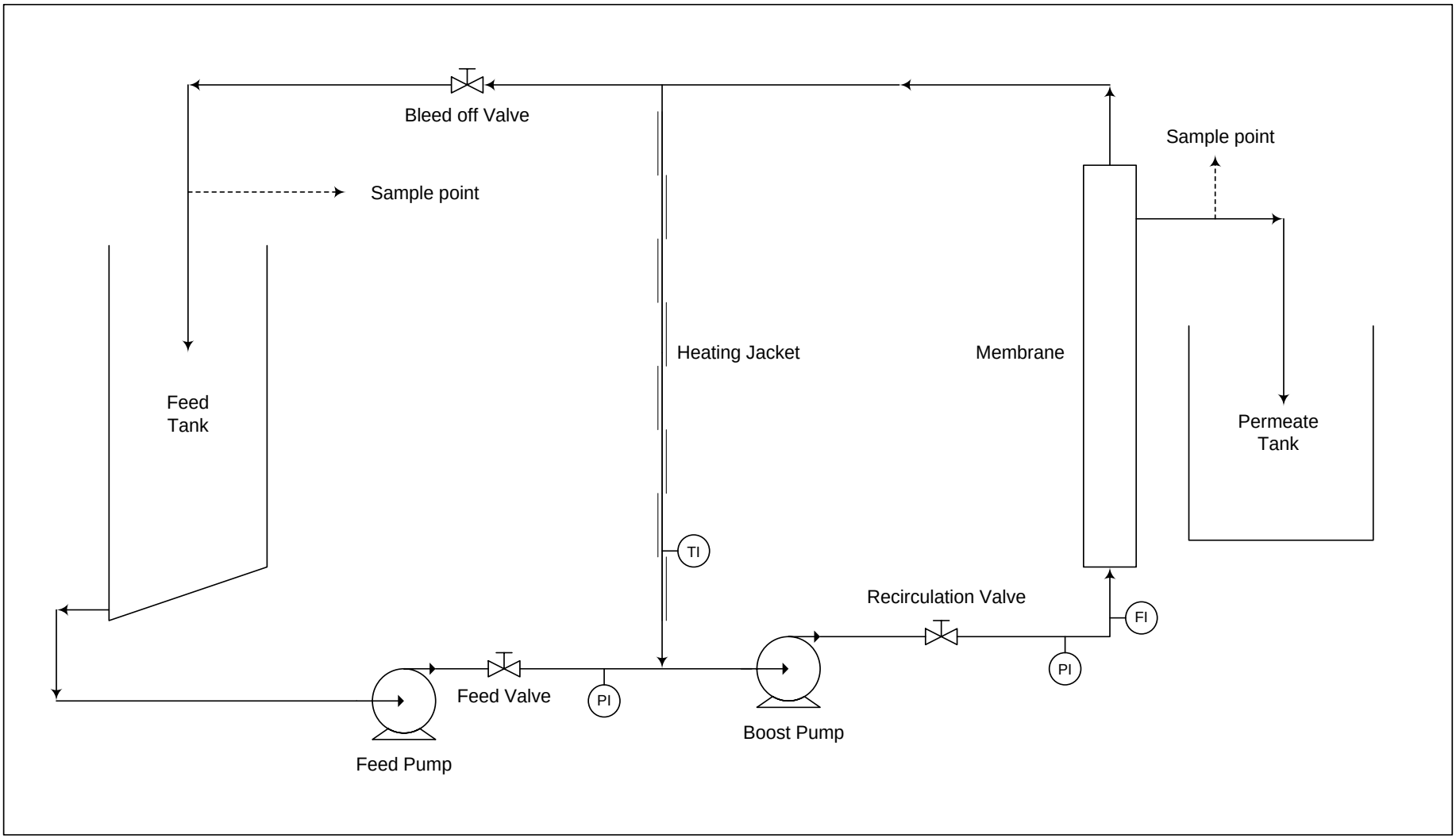

Figure 14. Schematic of hollow fibre membrane ultrafiltration pilot plant.

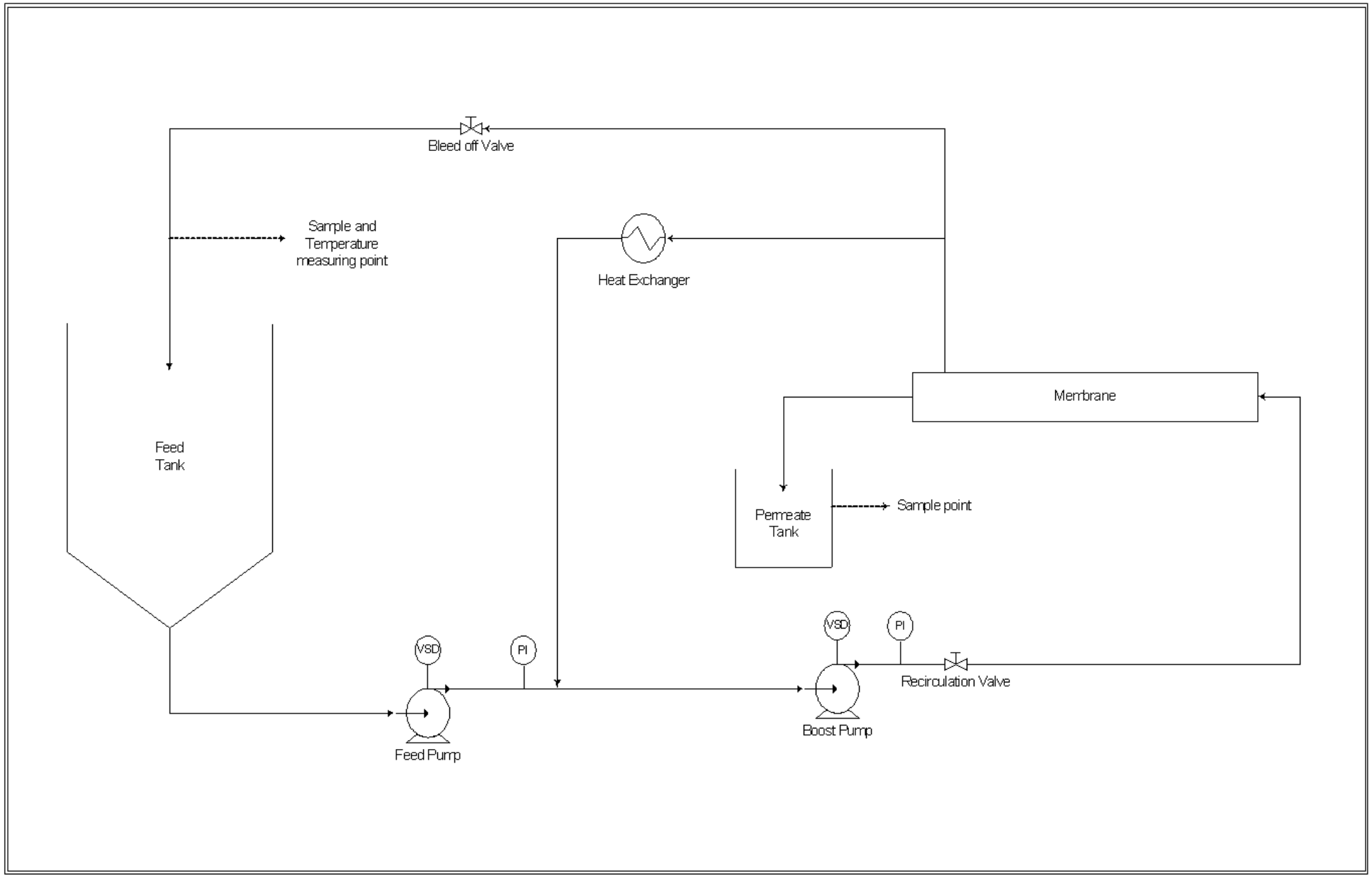

Figure 15. Schematic of the spiral wound membrane ultrafiltration pilot plant.

5.0 Methods of Analysis

5.1 Chemical Analysis

5.1.1 Composition of milk

The protein and fat contents and total acidity of the milk were obtained from Goodman Fielder, Longburn factory, the supplier of milk. The composition of milk was determined using the Foss Milko-scan^{TM} S50.

5.1.2 Protein in cheese

The protein content in cheese was determined using the Kjeldahl method as it is the most accurate method available for protein. The detailed experimental method for protein in cheese is attached in Appendix-C. The experimental method was based on the AOAC official method 2001.14 (33.7.12A) (AOAC International, 2005).

5.1.3 Fat in cheese

Fat percentage in cheese was evaluated using the modified Schmid-Bondzynski-Ratzlaff method. The experimental methods sheet is attached in the Appendix-C, which was based on the ISO 1735:198 E (Ceirwyn, 1995).

5.1.4 Total solids and moisture content in cheese

The total solids percentage in cheese was determined using the air drying oven as specified in the AOAC standards official method 926.08 (AOAC International, 2005). The description of the experimental method used is attached in Appendix-C. The moisture content was determined using the total solids percentage as shown in the total solids experimental method in Appendix-C. The moisture content is inversely proportional to the total solids content.

The total solids content can also be measured using an abbe refractometer, the refractometer was initially calibrated to zero on the scale using water. The sample was placed in the refractometer, the divider line was adjusted such that the darker side is separated from the lighter side and the value for that specific position read off the scale to obtain the total solids content as a percentage. This method of total solids determination is more rapid than that of the air drying method, however it is also less

accurate. The accuracy of the total solids percentage is affected by the ambient temperature and includes the human error associated with the reading taken off the scale.

5.1.5 Salt in cheese

The salt content in the cheese was determined using the Volhard method. The experimental method that is used is provided in Appendix-C, which is based on the AOAC official method 935.43 (33.7.10) (AOAC International, 2005) and the procedure provided by Ceirwyn (1995).

5.1.6 Lipase activity

The lipase activity in the cheese was determined by the acid degree value method as specified by Deeth and Fitz-Gerald (1976). The experimental procedure is provided in Appendix-C.

5.2 Microbiology

The cheese produced on the pilot plants was checked for *Staphylococcus aureus*, *Escherichia coli*, coliforms and yeast and mould. All microbiological testing was done at Goodman Fielder, Longburn factory by trained laboratory technicians. The tests at Longburn are performed as defined by the New Zealand testing methods (NZTM) (NZTM 2.48.7 for *E.Coli*, NZTM 2.48.1 for *coliforms* and NZTM 2.61.1 for yeast and moulds) and approved by the New Zealand Food Safety Authority (2008).

5.3 Sensory

Initially all judges on the panel were screened for four tastes salty, sweet, sour, and bitter before the first session of the feta cheese sensory. The form used for screening is attached in Appendix-C.

The panel consisted of nine judges, who tasted the same batch of cheese three times in different sessions every month (to obtain a large enough sample size for statistical analysis). Each batch of cheese was tasted by the judges every month for a storage period of four months. Therefore for each batch of cheese a sample size of 27 was

obtained per month. For each session the judges were asked to taste the pilot plant cheese and reference cheese from PVC and rate the difference. This method of sensory evaluation is known as the difference from control method (Morten et al. 2007). The difference was rated on a scale of 1 -7, where 1 – 2 was interpreted as sample being the same as the reference, a rating of 3 – 4 represented a moderate difference between reference and sample and 5 – 7 rating showed that the two samples tasted were extremely different. If a difference greater than two was perceived, the judges were asked to identify the difference in terms of a few given attributes.

All data from sensory were analysed using Minitab 5.0 using a general linear model. The difference from control form used for sensory is attached in Appendix-C.

5.4 Texture

The texture analysis was performed using a Stable Micro systems texture analyzer (model: TA.XT.Plus). The texture measurements were evaluated using a cheese sample of 20mm cubes, 50kg load and 50 mm diameter plunger which generates a compression curve. The hardness and fracturability of the cheese were determined from the first and second peak of the compression curve. The detailed procedure is attached in Appendix-C.

6.0 Results and Discussion

6.1 Ultrafiltration runs

Whole milk was concentrated to a total solids concentration of 37.5 ± 0.5 % using hollow fibre and spiral wound ultrafiltration membranes as described in the cheese-making section (Section 4.0). All experiments were conducted with flow across the membrane (cross flow filtration) and the operating temperature maintained between 45 - 50°C. The hollow fibre plant was operated with a pressure difference along the membrane (ΔP) of 0.3 bar (on feed/retentate side of membrane), whereas the spiral wound unit was run at one of two pressure differences; with a ΔP of 0.3 (Spiral DP1) and 1.0 bar (Spiral DP2).

The inlet pressure (P_{in}), outlet pressure (P_{out}), pressure difference along the membrane (ΔP), transmembrane pressure (TMP), pressure drop per unit length (ΔP/L) and the area to volume ratio for the corresponding run are listed in Table 1. The transmembrane pressure is the pressure exerted across the membrane, which is also known as the driving force for the ultrafiltration systems. The membrane area to milk volume ratio shows the membrane area in m^2 available for each litre of milk that needs to be concentrated.

Table 1. The inlet pressure (P_{in}), outlet pressure (P_{out}), pressure difference along membrane (ΔP), transmembrane pressure (TMP), pressure drop per unit length (ΔP/L) and the milk volume to membrane area ratio for the runs on hollow fibre, spiral wound pilot plants and PVC ultrafiltration system.

	P_{in} (bar)	P_{out} (bar)	ΔP (bar)	TMP (bar)	ΔP/L (bar/m)	Area:Vol
Hollow fibre	1.6	1.3	0.3	1.5	0.5	0.013
Spiral DP1	1.0	0.7	0.3	1.0	0.3	0.026
Spiral DP2	1.7	0.7	1.0	1.3	1.0	0.026
PVC*	2.1	1.1	1.0	1.6	0.9	0.015

*PVC represents Puhoi Valley Cheese hollow fibre ultrafiltration system data.

The operating temperature, total run time, volume of milk used and the volume of concentrate produced for the runs on the hollow fibre, spiral wound pilot plants and the PVC ultrafiltration system are as shown in Table 2.

Table 2. The milk volume, concentrate collected, operating temperature and the run time for the hollow fibre, spiral wound pilot plant and PVC runs. The pilot plant data is an average of four runs, where as the PVC data is an average of ten runs.

Sample	Milk Volume (L)	Concentrate collected (L)	Operating temperature (ºC)	Run time (minutes)
Hollow fibre	100	21.0 ± 0.6	49.6 ± 0.4	180
Spiral DP1	140	25.5 ± 0.3	46.6 ± 0.4	185
Spiral DP2	140	25.3 ± 0.5	47.6 ± 0.2	120
PVC	10000	1875 ± 60	46.7 ± 0.4	180

Due to the presence of the heat exchanger on the spiral wound pilot plant, the hold up volume is 10L, where as the hold up volume on the hollow fibre unit is approximately 2L. Therefore, a larger volume of milk than the hollow fibre runs was concentrated on the spiral wound unit to obtain a sufficient amount of concentrate. When the volume of milk used on the spiral wound unit was the same as the volume used in the hollow fibre unit, tiny air bubbles were present in the concentrate. Due to the high viscosity of the concentrate, the air bubbles did not rise to the surface therefore the cheese made from this concentrate had small air bubbles in the structure.

As the flow meter on the spiral wound unit was out of order, the feed flow rate into the membrane could not be measured. However, the ΔP and TMP on the hollow fibre and spiral wound units were maintained as close as possible to that of PVC, given the limitations of the pilot plant equipment. The maximum ΔP that could be achieved on the hollow fibre pilot plant was 0.3 bar hence, one of the runs on the spiral wound pilot plant unit was designed to be operated at a ΔP of 0.3 bar.

Table 3 shows the pH of the milk concentrate (before enzyme addition), after the concentrate was set into curds or before brining (before cutting to size) and the final cheese (after brining the cheese).

Table 3. The pH of concentrate before enzyme addition, before cutting the set cheese and after brining the cheese for the pilot plant runs and the PVC runs. The pilot plant data are mean ± SE (n=4), the PVC data (n=10)

	pH		
Sample	**before enzyme addition**	**before cutting to size**	**after brining the cheese**
Hollow fibre	6.54 ± 0.01	4.80 ± 0.03	4.66 ± 0.03
Spiral DP1	6.59 ± 0.03	4.82 ± 0.03	4.72 ± 0.08
Spiral DP2	6.54 ± 0.03	4.83 ± 0.01	4.58 ± 0.08
PVC	6.63 ± 0.03	4.75 ± 0.03	4.73 ± 0.01

The recycle rate for the PVC runs was maintained at 25% of the feed throughout the run. The hollow fibre recycle rate was initially set at 12% (0.143 L/sec) of the feed and not altered during the run, as the total solids content in the concentrate increases the feed flow rate is required to be decreased to maintain the constant ΔP. The decrease in feed flow rate and the constant recycle rate of 0.143 L/sec causes the recycle rate to increase from 12% to 25% of the feed. The recycle rate on the spiral wound pilot plant was set as close as possible to that of the hollow fibre plant in terms of L/sec as the feed flow rate on the spiral wound unit could not be measured. The feed and permeate flow rates and the recycle rate for the runs on the hollow fibre and spiral wound pilot plants and the PVC ultrafiltration system are shown in Table 4.

Table 4. The initial and final feed, permeate flow rate and the recycle rate for the hollow fibre, spiral wound and PVC runs. The data is the mean ± SE (n=4) for the pilot plant and for PVC (n=10).

	Feed Flow rate (L/sec)		Permeate Flow rate (L/sec)		
Sample	**Initial**	**Final**	**Initial**	**Final**	**Recycle rate (L/sec)**
Hollow fibre	1.159 ± 0.004	0.56 ± 0.04	0.0116 ± 0.0004	0.0025 ± 0.0002	0.143 ± 0.002
Spiral DP1	-	-	0.018 ± 0.002	0.0023 ± 0.0002	0.197 ± 0.003
Spiral DP2	-	-	0.0249 ± 0.0006	0.0027 ± 0.0003	0.200 ± 0.001
PVC	0.483 ± 0.005	0.374 ± 0.001	0.041 ± 0.001	0.0111 ± 0.0007	25% of feed

6.1.1 Fluxes

Figure 16 represents the permeate flux drop with time data from hollow fibre and spiral wound runs. All pilot plant data presented are a mean of four runs for each system.

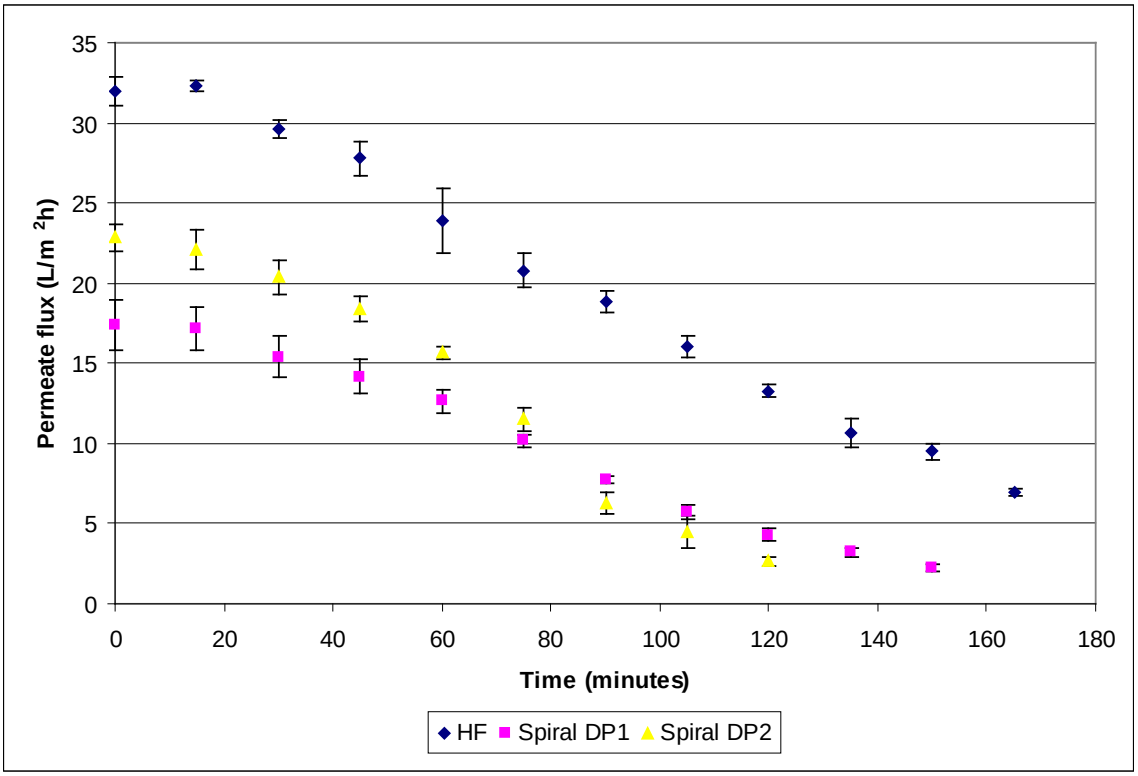

Figure 16. Plot of permeate flux (LMH) versus time (minutes) for runs on hollow fibre at ΔP of 0.3 bar, spiral wound unit at ΔP of 0.3 and 1.0 bar. Data are mean ± SE (n = 4).

The data from both units at all differential pressures followed a similar trend of decrease in permeate flux over time which corresponded to the increase in the total solids concentration with time. As stated in the literature review an increase in the solids concentration in the feed causes a decline in permeate flux due to concentration polarisation in both hollow fibre and spiral wound membranes (Yeh et al., 2003, Castro and Gerla, 2005).

Higher permeate flux was observed from the hollow fibre runs than spiral wound runs. From Table 1 it can be seen that the average driving force (TMP) for spiral DP1 run was lower than the HF and spiral DP2 runs. The inlet pressure (P_{in}) and driving force (TMP) of both hollow fibre and spiral DP2 run were similar, the outlet pressure (P_{out}) however for hollow fibre was higher. The pressure in both units was controlled by adjusting the flow, hence the hollow fibre unit had a higher cross flow velocity. Yeh et al (2003) showed that the presence of a larger driving force (TMP) and high cross flow velocity generated a higher permeate flux. In accordance with Yeh et al (2003) the hollow fibre produces the highest permeate flux, which is due to high transmembrane pressure and high cross flow velocity than the runs on the spiral wound unit.

The permeate flux generated by PVC ultrafiltration system and the hollow fibre pilot plant are compared in Figure 17. All PVC data is a representative of ten runs where as hollow fibre pilot plant data is from four runs.

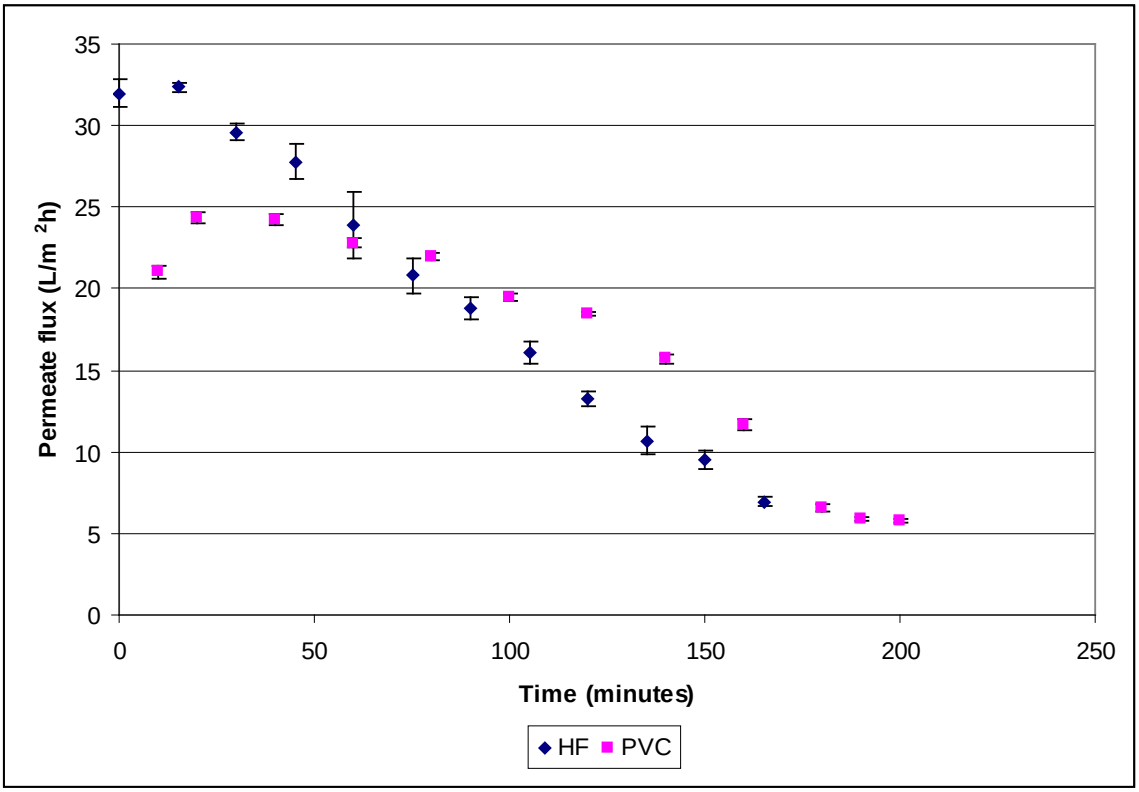

Figure 17. Plot of permeate flux (LMH or Litres per Meter squared Hour) versus time (minutes) data is mean ± SE (PVC n = 10, HF n = 4) PVC runs operated with ΔP of 1.0 bar and hollow fibre pilot plant operated with ΔP of 0.3 bar, both operated between 45-50ºC.

From Figure 17 it can be observed that the permeate flux decreased with time for both PVC and hollow fibre pilot plant runs. The PVC data showed a different rate of decrease in permeate flux with time to that of the pilot plant data. The pasteurised milk at PVC that was to be ultrafiltered was collected into two tanks where one of the tanks was connected to the ultrafiltration unit, as the level of the milk decreases in this tank the milk from the second tank was added to the first tank connected to the ultrafiltration plant. The addition of pasteurised milk to partly concentrated milk decreases the total solids level, hence the membranes at PVC are exposed to lower total solids level for longer period than the hollow fibre pilot plant. Therefore, the rate of decrease in permeate flux for PVC was slower than that observed in the hollow fibre pilot plant.

The pressure drop per unit length of the ultrafiltration system in PVC was 0.9bar/m whereas the for the hollow fibre pilot plant it was 0.5bar/m. Cheryan (1998) shows that an increase in pressure drop also increases turbulence, which further decreases concentration polarisation and improves permeate flux. It can be observed from Figure 17 that the rate of permeate flux drop for the PVC data was lower than that of hollow fibre pilot plant data due to higher turbulence in the feed at PVC.

6.1.2 Increase in total solids

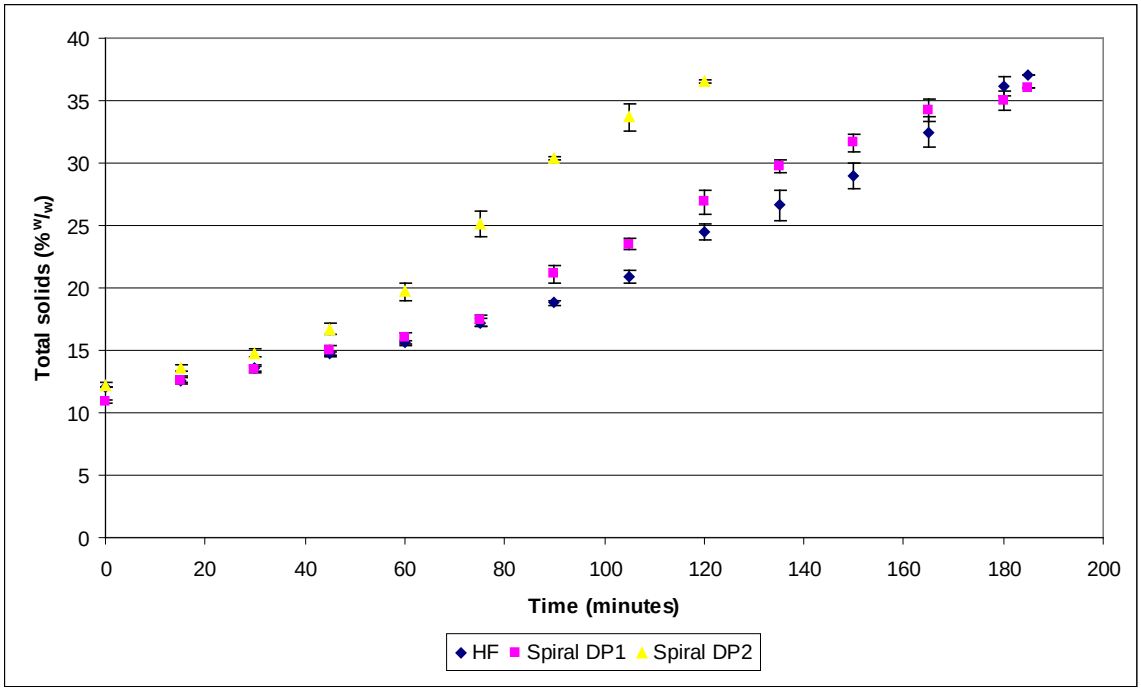

Figure 18. Plot of total solids % $^{w}/_{w}$ versus time (minutes) for hollow fibre at ΔP of 0.3 bar, spiral wound at ΔP of 0.3 bar and 1.0 bar. Each set of data is the mean ± SE (n = 4).

An increase in total solids over time was observed at all transmembrane pressures for both membranes as shown in Figure 18. The spiral DP2 run required less time to achieve the final total solids of 37.5 ± 0.5%, data from Figure 18 shows that spiral DP2 reached the required total solids in 120 minutes which was 70 minutes less than the pilot plant hollow fibre and spiral DP1 runs. The run time required to achieve the final total solids for hollow fibre and spiral DP1 runs was 190 minutes.

The permeate flux for spiral DP2 run showed a large drop and generated lower permeate flux than the spiral DP1 run after 60 minutes (Figure 16). From Figure 18 it can be observed that both hollow fibre and spiral DP1 runs increased in total solids in two linear phases over time. The first phase was observed to be between 0 – 60 minutes, whereas the second phase was from 60 minutes to the end of the run. The rate of increase in total solids in the second phase was higher than in the first phase. The spiral DP2 run also showed the similar two-phase linear relationship between total solids and time, however the first phase was between 0 – 40 minutes. The increase in the total solids resulted in the increase in feed concentration and probably concentration polarisation and protein fouling on membrane surface contributed to the large decline in permeate flux (Cheryan, 1998).

From Table 1 it can be observed that both hollow fibre and spiral DP1 runs have a similar ΔP but the hollow fibre run had a higher TMP (driving force) than the spiral DP1 run. Although the driving force was similar for hollow fibre and spiral DP2 runs, the spiral DP2 run achieved the required total solids in a shorter time due to the higher pressure drop in the spiral DP2 runs. Increase in pressure drop can be related to an increase in turbulence (Cheryan, 1998). The increased state of turbulence further enhances the effect of cross flow velocity in removal of protein deposits on the membrane surface, hence better permeate flux and less time required to achieve the final total solids concentration.

The membrane surface area is 1.3 m^2 and 3.6 m^2 for hollow fibre and spiral wound respectively. The spiral wound membrane had greater surface area, although the milk volume processed by the spiral wound unit (140 L) was greater than the hollow fibre unit (100L) the membrane area to milk volume ratio was larger for spiral wound membrane than that of hollow fibre membrane. The larger area to volume ratio means for each litre of milk that needed to be processed the spiral wound membrane could provide more surface area. Therefore, the spiral DP2 run was able to achieve the desired total solids increase in a faster time than the hollow fibre unit.

The Spiral DP1 run shows similar increase in total solids in comparison to the hollow fibre run although the spiral wound had larger membrane surface area available per litre of milk. As listed in Table 1 the driving force for the spiral DP1 run is 1.0 bar which was lower than that of the hollow fibre at 1.5 bar and spiral DP2 run at 1.3 bar. Due to the lower driving force the spiral DP1 run took a longer time than spiral DP2 to achieve the final total solids concentration.

The total solids concentration data from the hollow fibre pilot plant and the data obtained from PVC hollow fibre ultrafiltration system are presented in Figure 19 for comparison. The hollow fibre pilot plant data is a mean of four runs whereas PVC data is the mean of ten runs.

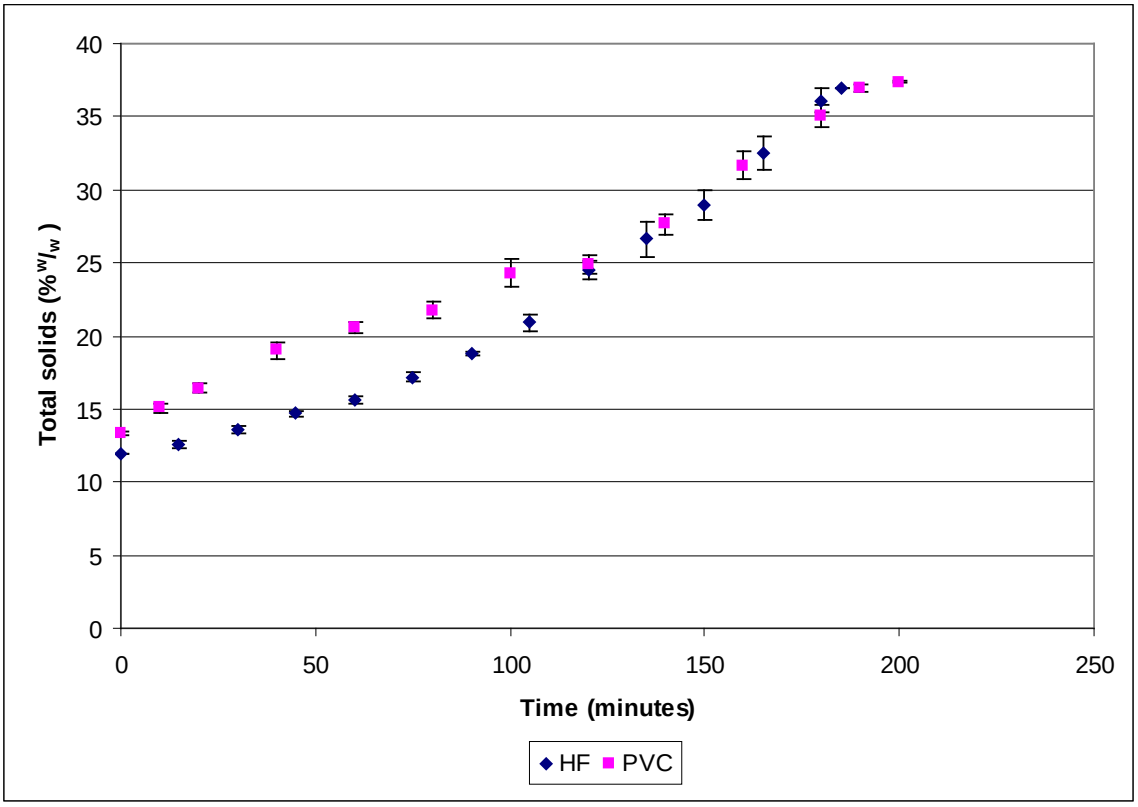

Figure 19. Graph of total solids % $^{w}/_{w}$ versus time (minutes), data is mean ± SE (PVC n = 10, HF n = 4). PVC runs operated with ΔP of 1.0 bar and hollow fibre pilot plant operated with ΔP of 0.3 bar, both operated between 45-50ºC.

The PVC data in Figure 19 shows that the rate of increase in total solids % increased constantly initially, however the rate changed between 45 – 125 minutes

and increased constantly during the period of 125 – 200 minutes. In comparison the hollow fibre pilot plant data showed two linear phases of increase in total solids with time, where first phase was between 0 – 60 minutes and the second phase was from 60 minutes to the end of the run. All data from PVC are obtained from 10,000L production runs. As stated earlier at PVC the pasteurised milk is collected in two tanks where the first tank is connected to the ultrafiltration unit. This first tank is recycled through the ultrafiltration unit, as the milk is concentrated the level in the tank drops with time and the pasteurised milk from the second tank is added to the concentrated milk. This would explain the variation in the rate of increase in total solids between 45 –125 minutes in the PVC runs, as the second tank of fresh milk is pumped into the system, effectively diluting the concentrate. It can also be observed that both PVC and hollow fibre pilot plant systems produced the same total solids percentage at around 200 minutes.

6.1.3. Total solids vs. permeate flux

The relationship between the permeate flux and total solids of the runs on the hollow fibre and spiral wound pilot plant units is shown in Figure 20. It can be observed from Figure17 that the permeate flux decreases with increasing total solids for all runs. This is in agreement with Cheryan (1998) who states that increase in feed concentration results in a decrease in permeate flux due to concentration polarisation.

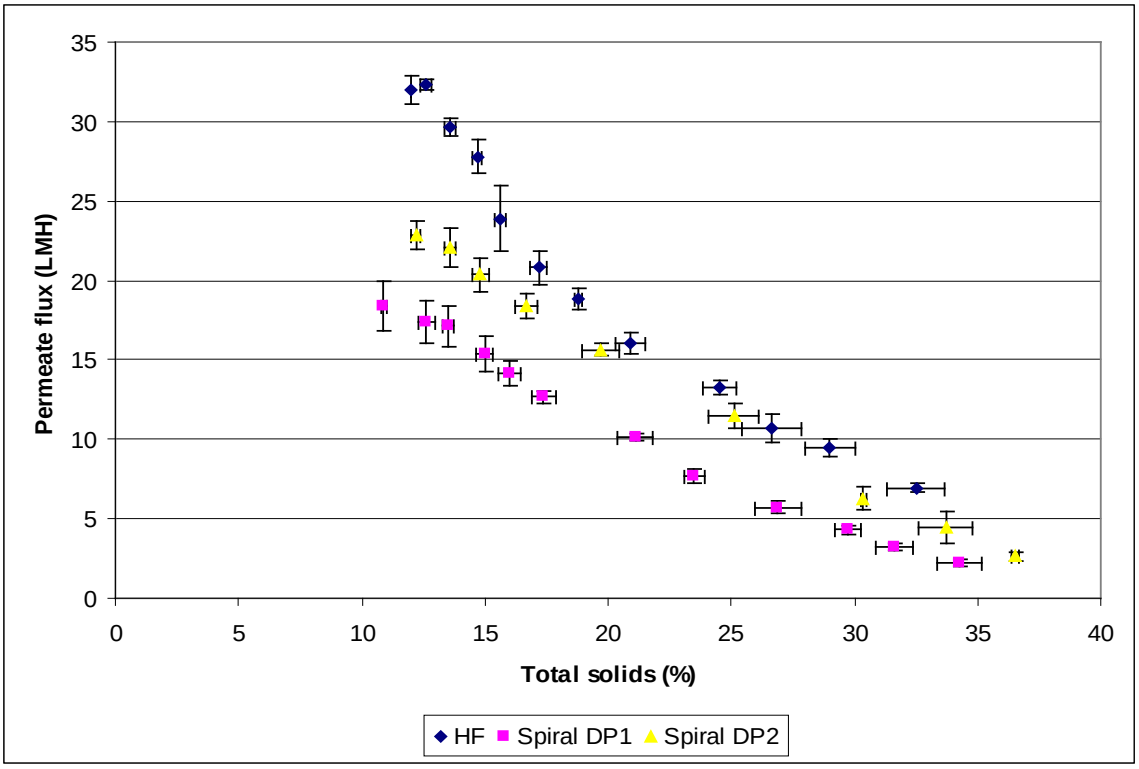

Figure 20. Plot of permeate flux (LMH) versus total solids (%) for runs on hollow fibre at ΔP of 0.3bar, spiral wound unit at ΔP of 0.3 bar and 1.0 bar. Data are a representation of mean ± SE (n=4).

Initially at lower total solids concentration the highest permeate flux was generated by the hollow fibre pilot plant runs. As mentioned earlier in the fluxes section (6.1.1 section), this was due to a high driving force and higher cross flow velocity than the runs (Spiral DP1 and DP2) on the spiral wound unit. The rate of decrease in permeate flux with an increase in total solids was also the greatest in the runs on the hollow fibre unit. Secchi et al (1999) and Cheryan (1998) stated that the effects of concentration polarisation and adsorption on the membrane surface are of greater significance in hollow fibre membranes due to the laminar flow operating conditions.

The spiral DP1 runs generate lower permeate flux than all the other runs due to the lower TMP as mentioned in the fluxes section. All pilot plant data in Figure 20 are a mean of four runs.

6.1.4 The effect of recycle rate

The effect of recycle rate on the permeate flux was examined by operating the spiral wound unit at two different recycle rates of 7.5 and 12 L/min. However, due to time constraints the experiments with 7.5 L/min recycle rate could not be repeated for accuracy. Hence all data for the 7.5L/min were obtained from one ultrafiltration run. Figure 21 shows the effect of recycle rate on the permeate flux at ΔP of 0.3 bar on the spiral wound unit.

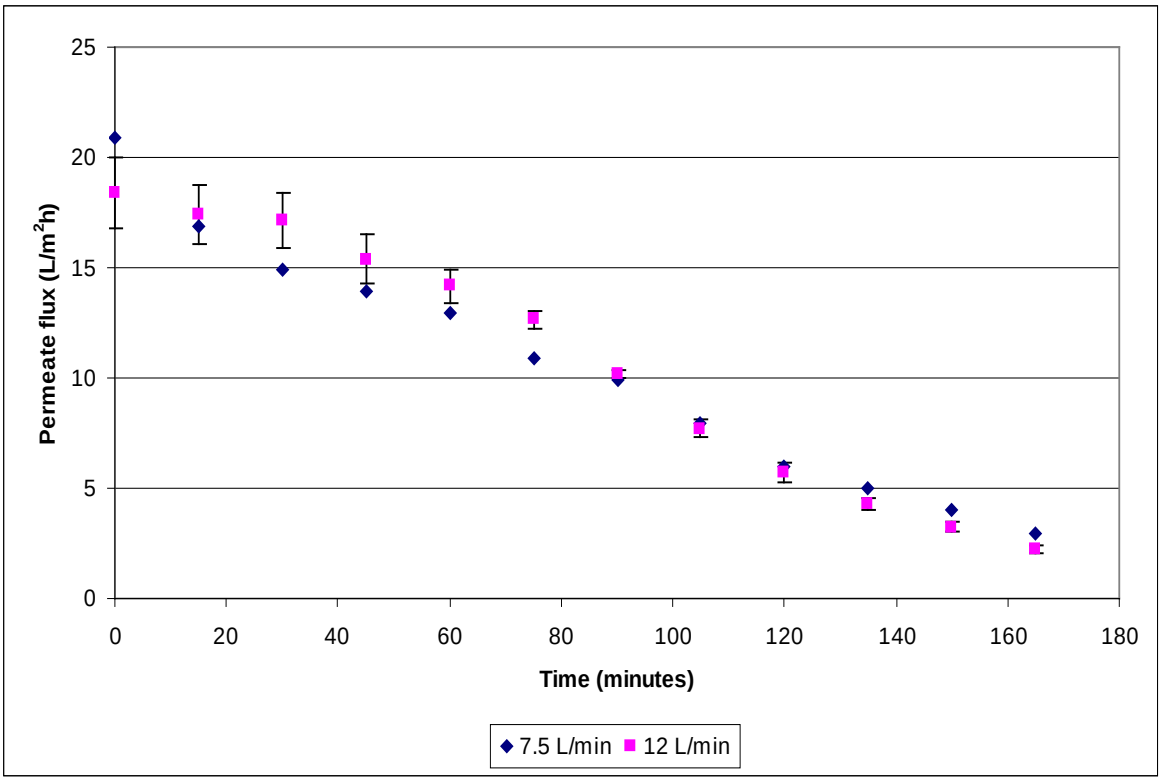

Figure 21. Graph of permeate flux (LMH) versus time (minutes) for runs at different concentrate recycle rates of 7.5 and 12 litres per minute on spiral wound unit at ΔP of 0.3 bar. The 12L/min data is a mean ± SE (n = 4) s and 7.5L/min data (n = 1).

The recycle rate is the amount of retentate from the ultrafiltration unit that is bled back to the feed tank. Decreasing the recycle rate results in less of the retentate from the ultrafiltration system is bled back to the feed tank. Hence, with lower recycle rate the milk in the UF unit is exposed to the membrane for a longer time resulting in greater increase in total solids and greater concentration polarisation.

The increase in the concentration polarisation causes a greater decrease in permeate flux. Therefore, at lower recycle rates the permeate flux decline should be higher than that observed at higher recycle rates.

The permeate flux decreased with time for both recycle rates as shown in Figure 21. It can be determined from Figure 21 that the 12 L/min recycle rate run data showed a slightly higher flux between 20 to 80 minutes than the 7.5 L/min run data. The increase in recycle rate from 7.5 to 12 L/min did not significantly improve the permeate flux performance in the spiral wound membrane unit. However, greater increases in recycle rate could affect the permeate flux performance. Higher recycle rate results in higher proportion of the feed exposed to the membrane being the lower total solids concentrate in the tank. The lower total solids content of the concentrate decreases concentration polarisation, hence the permeate flux would be improved.

The increase in total solids concentration over time for concentrate recycle rates of 7.5 and 12 L/min using the spiral wound unit at ΔP of 0.3 bar is as shown in Figure22.

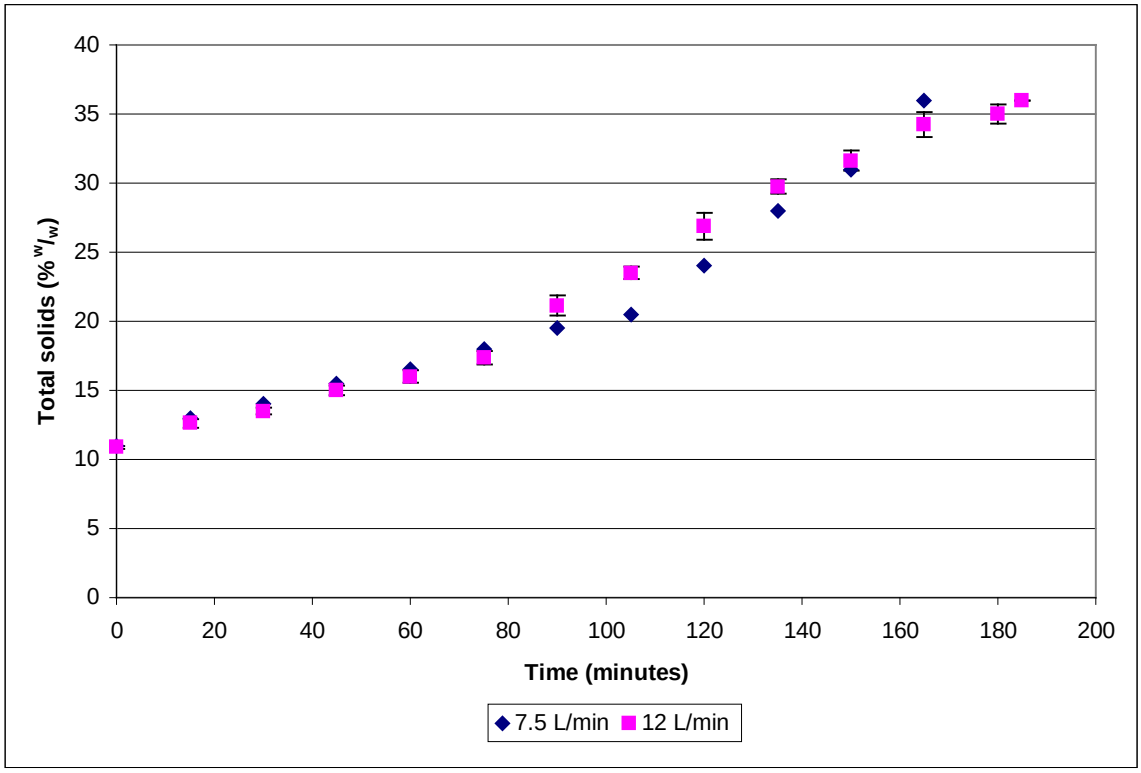

Figure 22. Graph of total solids (% w/w) versus time (minutes) for different recycle rates of 7.5 and 12 litres per minute for runs on spiral wound unit at ΔP of 0.3 bar. The 12L/min data is a mean ± SE (n = 4) s and 7.5L/min data (n = 1).

Figure 22 shows that no significant difference existed between the total solids data from concentrate recycle rates of 7.5 and 12 L/min at ΔP of 0.3 bar.

The permeate flux performance over time for concentrate recycle rates of 7.5 and 12L/min at ΔP of 1.0 bar on the spiral unit is shown in Figure 23.

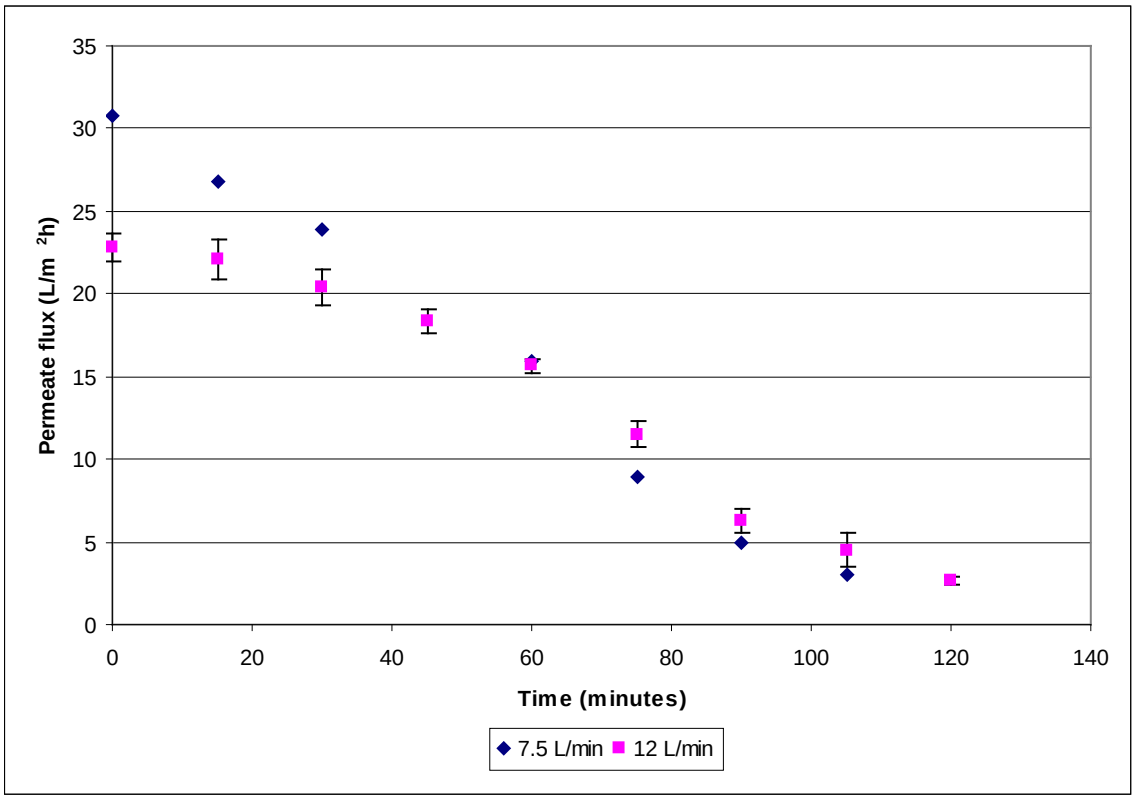

Figure 23. Plot of permeate flux (LMH) versus time (minutes) for runs on spiral wound unit with ΔP of 1.0 bar at concentrate recycle rate of 7.5 and 12 litres per minute. The 12L/min data is a mean ± SE (n = 4) and 7.5L/min data (n = 1).

Data in Figure 23 shows that at both recycle rates, the permeate flux decreased over time and levelled off towards the end of the run. The data from 7.5 L/min recycle rate shows slightly higher initial values of permeate flux in the first 30 minutes of the run than the 12 L/min run. The data for 7.5 L/min run is from one run, the initial temperature of the milk for this run at ΔP of 1.0 bar was 50ºC. The average initial milk temperature of the 12 L/min runs was 44.5 ºC.

High temperature aids in diffusion (Cheryan, 1998), hence the 7.5 L/min run had higher permeate flux initially. In the first 30 minutes of the 7.5 L/min run the temperature decreased to 44 ºC, as 50 ºC was the maximum operating temperature of membrane. This decrease in the temperature coupled with the effect of concentration polarisation decreased the permeate flux as seen in Figure 23 in the initial 30 minutes. After the initial 30 minutes there seems very little difference in

the permeate flux drop between the 7.5 and 12 L/min recycle rates. This shows that there was no significant effect of concentrate recycle rate on the permeate flux.

Figure 24 shows the total solids data obtained from runs on spiral wound unit for concentrate recycle rates of 7.5 and 12 L/min at a ΔP of 1.0 bar. The 12 L/min data are average of four runs, whereas the 7.5 L/min is data from one run.

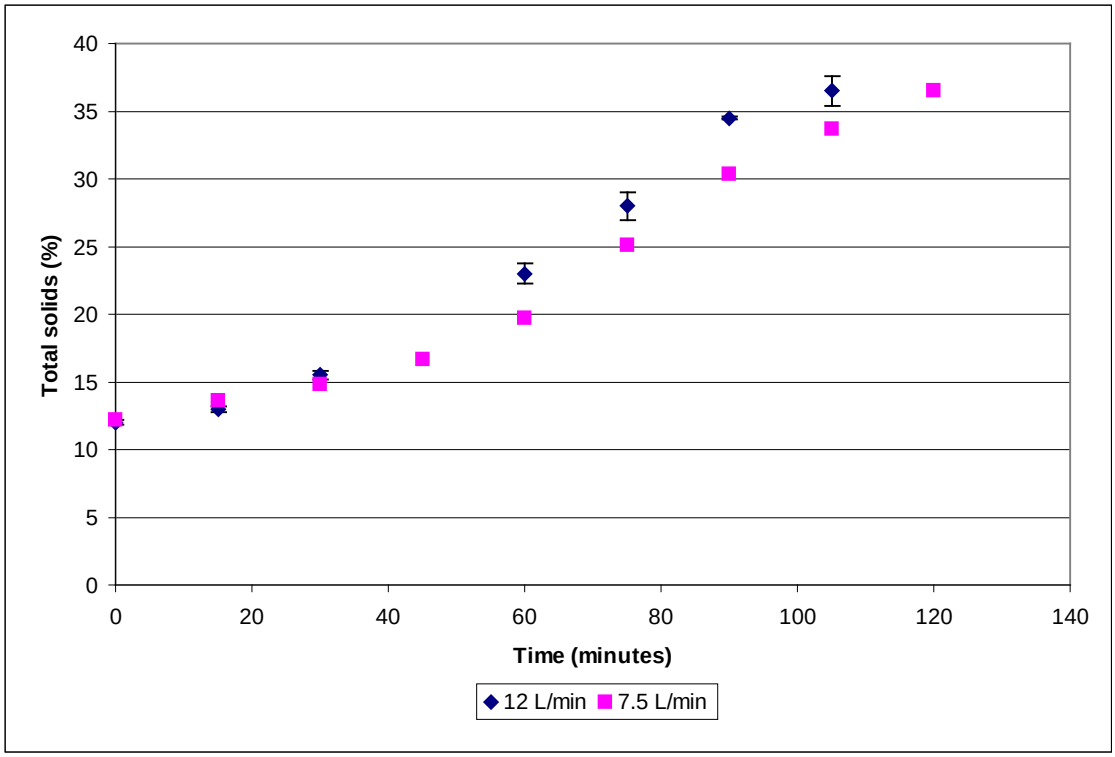

Figure 24. Graph of total solids (%) versus time (minutes) for runs on spiral wound unit at ΔP of 1.0 bar at concentrate recycle rates of 7.5 and 12 litres per minute. The 12L/min data is a mean ± SE (n = 4) and 7.5L/min data (n =1).

A similar trend in the increase of total solids with time was observed with both concentrate recycle rates of 7.5 and 12 L/min runs in Figure 24. The rate of increase in total solids for the 12 L/min run was slightly higher than the 7.5 L/min run. However, this difference in the rate of increase in total solids was not considerably high. This shows that the concentrate recycle rate used did not have a considerable affect on the total solids increase with time.

In summary, hollow fibre, spiral wound and PVC units show that the permeate flux decreased and total solids concentration increased with time at anyΔP. Spiral DP2 run required less time to achieve the final total solids percentage due to a combination of higher membrane surface area, turbulent flow characteristics and higher TMP. The hollow fibre pilot plant generated higher permeate flux than spiral wound unit at any differential pressure due to higher cross flow velocity. The runs conducted at different concentrate recycle rate showed that concentrate recycle rate did not have any significant effect on either the rate of increase in total solids or rate of decrease in permeate flux over time.

The transmembrane pressure and the membrane surface area available per litre of milk that was to be concentrated for the hollow fibre pilot plant and PVC ultrafiltration is similar. Hence the time required to achieve the final total solids concentration was 180 minutes for both hollow fibre pilot plant and PVC ultrafiltration runs. However, the pressure drop per unit length along the hollow fibre pilot plant membrane was smaller than that of PVC membrane. The lower pressure drop signified less turbulence, therefore the permeate flux data from the pilot plant slightly differs to PVC data. The flow characteristics of the pilot plant hollow fibre membrane were checked to be laminar and since the hollow fibre membrane generally shows laminar flow characteristics (Cheryan, 1998), the level of turbulence difference between the hollow fibre pilot plant and PVC is not expected to be very significant. The hollow fibre pilot plant was able to mimic the milk concentration as at PVC, however the permeate flux could not be exactly imitated as at PVC but the hollow fibre pilot plant managed to achieve the required total solids in the same time as at PVC.

The spiral wound membrane pilot plant due to the presence of the feed spacer was operated under turbulent flow characteristics. The spiral membrane was chosen with a larger a gap to cope with the increasing viscosity with increase in total solids. The larger gap membrane, along with turbulent flow successfully enabled the desired concentration of total solids to be achieved reproducibly. Although the permeate flux generated by the spiral wound membrane unit were lower than that of the hollow fibre pilot plant unit, the desired concentration of total solids was achieved.

6.2 Chemical Analyses of Feta Cheese

6.2.1 Protein, fat, moisture and salt content

The cheese made from the concentrated milk from the pilot plants was analysed for protein, fat, total solids, salt and lipase activity. The protein, fat, total solids and salt content in cheese made using the concentrated milk from the hollow fibre unit and spiral wound unit operated at ΔP of 0.3 and 1.0 bar expressed as percentage $^{w}/_{w}$ of cheese are shown in Figure 25 along with results for the reference (cheese from PVC). Each set of data expressed in Figure 25 is a mean value of four replicate samples (duplicate analyses of each replicate was performed to obtain an average for each replicate). The four replicates were produced over two periods, where the first and second replicates were produced in December 2007 a week apart and the third and fourth replicates were manufactured in January 2008 a week apart. The reference sample was obtained from PVC with a manufacturing date as close as possible to the respective replicate.

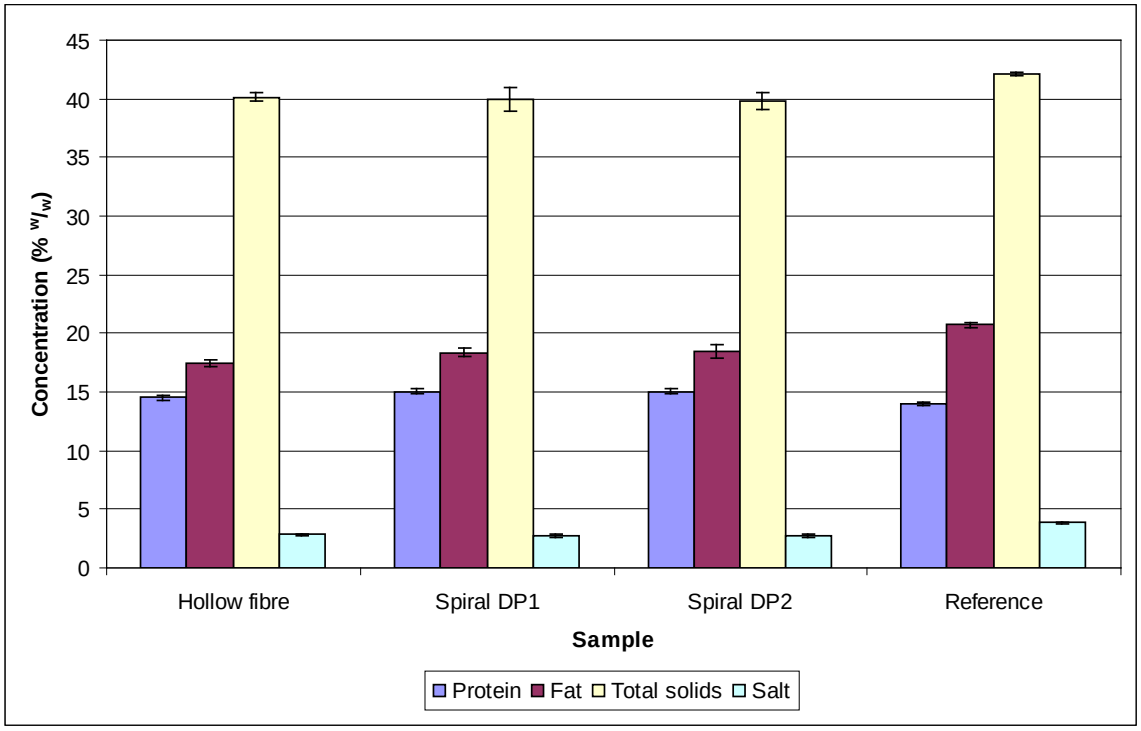

Figure 25. Graph of protein, fat, total solids and salt content in feta cheese made using hollow fibre and spiral wound at ΔP of 0.3 bar and 1.0 bar and the reference sample from PVC.

There was no significant difference in the concentration of protein, fat and total solids in the various pilot plant produced feta cheeses as shown in Figure 23. However in comparison to the reference feta sample, the protein content in the pilot plant cheeses was slightly higher. In terms of fat content, as can be determined from Figure 25 the fat percentage in the reference sample was significantly higher than that of the pilot plant cheeses. The reference sample showed higher total solids percentage than the cheese made from milk concentrated in the two UF pilot plants due to the presence of higher fat content in the milk used at PVC than the milk concentrated on the pilot plants. There was no significant difference between the pilot plant cheeses in terms of salt content but the reference sample showed a higher salt content in comparison.

The difference in the fat and protein content is due to the difference in protein to fat ratio in the milk used to make cheese using PVC UF system and the pilot plant units. The protein content in the milk concentrated on the pilot plant trials was 3.1 ± 0.01 % $^{w}/_{w}$, whereas the PVC milk was 3.5 ± 0.04 % $^{w}/_{w}$. The fat content was 3.3 ± 0.01 % $^{w}/_{w}$ and 4.3 ± 0.1 % $^{w}/_{w}$ in milk prior to be being concentrated using the UF pilot plants and PVC UF system, respectively. The milk concentrated in the pilot plants was standardised at the Longburn factory, therefore there was a very small variation associated with the protein and fat content. The milk used at PVC for concentration in the UF system was raw unstandardised milk, hence the variation in the protein and fat content of this milk.

The fat to protein ratio of the milk used for cheese production at PVC was 1:1.23 whereas the milk used to make cheese on the pilot plants had a fat to protein ratio of 1:1.06. The milk is acceptable for feta cheese making at PVC if the fat to protein ratio is less than 1:1.4, a higher ratio results in a cheese that is creamier and very similar to cream cheese.

In the PVC cheese, the presence of higher concentrations of fat globules in the raw milk resulted in more fat being entrapped in the casein matrix during the coagulation stage of cheese making. Hence, leading to an increased fat

concentration in the PVC cheese and decreased protein content. The fat and moisture give the casein matrix flexibility, which in terms of texture gives the cheese its softness (Madadlou et al., 2007). The decrease in the fat content contributes to a decrease in the softness of the cheese, hence making the cheese firmer.

The brine concentration affects the diffusion of the salt into the cheese blocks, the volume of brine was similar to that of the cheese volume that was to be brined. During brining the concentration of brine decreases due to moisture diffusing out of the cheese (Walstra et al., 1999). To account for this occurrence, the volume of brine should be five times larger than the volume of cheese (Tamime, 2006). The volume of brine to cheese used for the pilot plant cheeses was one to one ratio.

Figure 25 shows that the total solids content in pilot plant cheeses was lower than that of PVC, this in turn implies that the moisture content in the pilot plant cheeses was greater than that of the PVC cheese. The slightly higher moisture content in pilot plant cheeses can also explain the lower salt content in these cheeses. As salt diffuses in during the brining process it displaces water (moisture) which diffuses out (Madadlou et al., 2007). In the pilot plant cheeses, as there was less salt diffusion into the cheese blocks more moisture would have been retained in the feta cheeses, resulting in a lower total solids and lower salt content compared to the PVC cheese.

The variation in total solids content could also be due to the variation in the total solids content in the concentrated milk. At PVC the total solids of the milk was checked using a microwave air drying oven, hence the total solids content in the concentrated milk can be known accurately within a few minutes. For the pilot plant trials, the total solids content was estimated with the use of a refractometer, as it was not digital the total solids estimated has an associated human error. The final total solids of the milk concentrated using the pilot plants was also checked by the air drying oven for accuracy. At PVC the acceptable total solids concentration is $37.5 \pm 0.5\%$ where as the total solids concentration achieved using the pilot plant

units was 37.5 ± 2%. This variation in the total solids content of the concentrated milk could have contributed to the difference in the total solids content of the pilot plant and PVC cheeses. However, the total solids content of all cheeses made from milk concentrated in the pilot plant units was within the range of 36 – 44% $^{w}/_{w}$ which is defined by PVC as the specification to be met in terms of total solids content of cheese.

6.2.2 Lipase activity

The lipase activity of the cheese was determined using the acid degree value (ADV) as specified by Deeth and Fitzgerald (1976). The ADV unit is defined as mL of alkali required to titrate the free fatty acids in 100 g of fat. The lipase activity was determined through extraction of fat using a detergent and solvents. It is important to measure the lipase activity as it is a measure of lipolysis (breakdown of fat) which affects the taste and texture of cheese. Hence, the lipase activity of all the cheeses was to be checked every month. Figure 26 shows the data on the lipase activity of cheese stored for two and four months.

The data from the second month of storage was a mean value of four replicates where as the data for the fourth month of storage was the mean of two replicates. The first two replicates were produced a month before the third and fourth replicate, the third and fourth replicates did not reach the fourth month of shelf life storage by the end of the project. Therefore, the fourth month data is an average of two replicates.

For the fourth month data, the first replicate of the spiral DP1 and the second replicate of the spiral DP2 runs showed extremely high ADV values hence to obtain a more reasonable representation of the data, they are not included in the data shown in Figure 26. Therefore, the fourth month data for the spiral DP1 and spiral DP2 is from the one replicate each, hence no error bars. The reference cheese samples had the same or very similar acid degree values for all replicates, hence very small or no error bars.

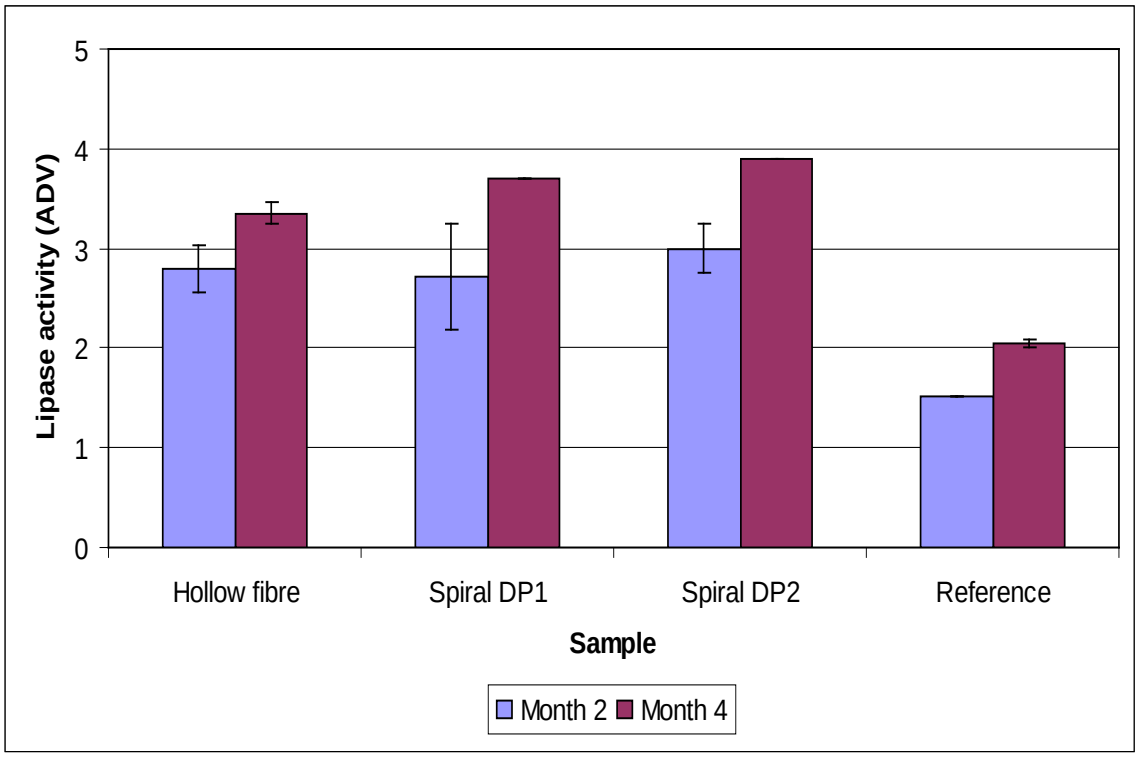

Figure 26. Graph of lipase activity as measured in acid degree value (ADV) after two and four months of storage time of cheese made by concentrating milk on hollow fibre, spiral wound unit operated at differential pressures of 0.3 and 1.0 bar and the reference cheese from PVC. Storage temperature of 5ºC.

It can be observed from Figure 26 that the lipase activity in the pilot plant cheeses was greater than that observed in PVC cheese after two and four months of storage. It can also be observed that there was an increase in lipase activity from the second to the fourth month for all cheeses. One replicate from each of spiral DP1 and spiral DP2 runs showed an unusually high lipase activity.

When investigated it was observed that there were experiments with meat being conducted in the laboratory where the pilot plants were located. The high lipase activity could have been the result of brine contamination while salting cheese by meat, which is a source of lipase. As a precaution the brining was further carried out in an enclosed space sterilized by ultra violet light. The lipase activity data for this replicate was considered to be an outlier and not included in the mean data represented in Figure 26 as mentioned earlier. The concentrate and raw milk samples of the batches with extremely high lipase activity were analysed in terms of

microbiology, however the results did not show any presence of *staphylococcus aureus*, coliforms, *E.Coli* and yeast and moulds.

Other factors affecting the lipase activity in cheese was the incomplete inactivation of the lipase enzyme in milk during the pasteurisation stage. The pasteurised milk used to make cheese on the pilot plants was pasteurised at 72.5ºC for 15 seconds prior to receipt at Massey, after ultrafiltration the concentrate was re-pasteurised at 72ºC for 15 seconds. At PVC raw milk is initially pasteurised at 76ºC for 15 seconds, after the pasteurised milk is concentrated through ultrafiltration it is homogenized and further heated to between 78 - 80ºC then cooled to 30ºC to completely inactivate the lipase.

Fox (2004) states that for complete inactivation of the enzymes, milk should be heated to 78ºC for 10 seconds, if not pasteurised cheese milk is susceptible to lipolysis. Whereas, Saxby (1996) states that slightly above pasteurisation temperature is sufficient to avoid flavour problems associated with lipase but complete inactivation requires heating milk to approximately 98°C for 1s. He also suggests that heat resistant lipases such as Pseudomonas species of bacteria may give milk a rancid flavour and may cause severe flavour problems in cheese. The higher lipase activity in all the pilot plant cheeses could be due to incomplete inactivation of the enzyme or the presence of the heat stable lipases. The aliquot required to titrate the extracted fat was very small, ranging from 0.3 to 0.6 mL, the experimental error associated with the titration was also great.

In summary, the feta cheese made on the pilot plants was slightly different to the feta cheese made at PVC. The pilot plant cheeses were slightly lower in fat and total solids content than the PVC cheeses. However, the cheese made from the pilot plant concentrated milk was very similar to the PVC cheese in terms of protein. These differences were considered to be due to the differences in the fat and protein content in the milk used to make the cheese at PVC and with the pilot plants. The salt content differed significantly in pilot plant cheeses when compared to PVC cheese. In terms of lipase activity, a large difference between the pilot plant cheeses and PVC

cheese was found. This difference was considered to be due to contamination of concentrate from other lipase containing sources present in the laboratory, incomplete inactivation of lipase enzyme present in milk, presence of thermostable lipase or due to experimental error.

6.3 Sensory

6.3.1 Degree of difference

The difference from control method was used to determine the quality of feta cheeses in terms of sensory evaluation. The sensory evaluation was performed by a panel of nine judges, who were initially screened for the four tastes, sweet, salty, sour and bitter. Each judge was presented with four samples, which consisted of hollow fibre, spiral wound at two differential pressures and a reference (control) sample from PVC. The judges identified the degree of difference between the pilot plant cheeses and the reference cheese using the scale provided.

The scale consisted of 1-7 degrees of difference, where rating of 1-2 defined the pilot plant cheese to be the same as the reference, 3-4 represented the existence of moderate difference and 5-7 was interpreted as the samples being extremely different from the control. If the samples were given a rating greater than 2, the judges were asked to pick how the cheese differs from reference in terms of the specific attributes that were provided. The judges also determined if the attributes were more or less than the reference. Sensory was performed on all four replicates for all four samples every month; the data in Figure 27 is the mean sensory data obtained for the four replicates, four cheeses after storage at 5 °C.

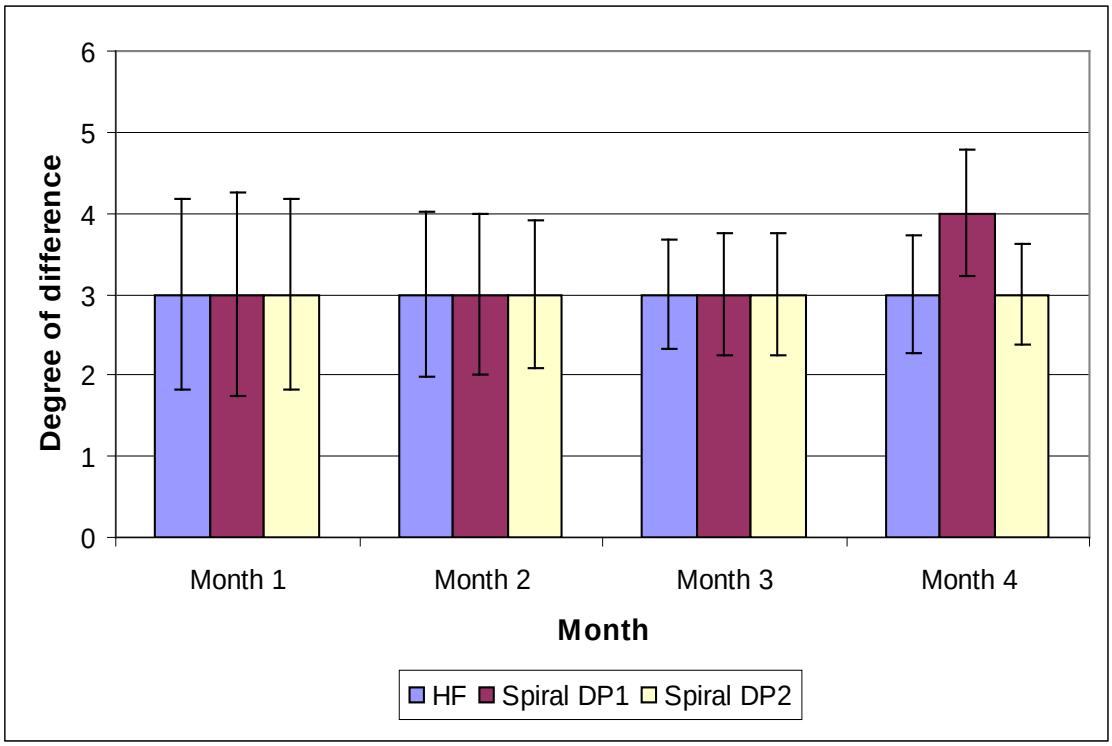

Figure 27. Graph of degree of difference detected by the judges for all the cheese made on hollow fibre unit and spiral wound at two differential pressures compared to the reference feta cheese from PVC. (n = 27 ± SE)

The judges identified that there was a difference between all the pilot plant feta cheeses and the reference feta cheese from PVC. They perceived the difference to be moderate as shown in Figure 27. The degree of difference between the pilot plant and reference feta cheeses over the shelf life period of four months did not change, the judges perceived it to be a moderate difference over the four months.

During ripening the process of proteolysis takes place, which is breakdown of proteins into lower molecular weight compounds and amino acids. The process of proteolysis contributes to the body and texture of the cheese (Gunasekaran and Mehmet, 2003). The constant degree of difference over the four months shows that the ripening taking place in the reference cheese from PVC is similar to that of the pilot plant cheeses.

The degree of difference for the hollow fibre cheese and the spiral wound cheeses with respect to the reference was the same, which suggests that the hollow fibre and spiral wound cheeses were very similar in terms of sensory evaluation. The statistical analysis of the sensory data showed that there was a significant difference ($P < 0.05$) between the pilot plant cheeses and the reference cheese from PVC. Hence, the differences were defined by the judges in terms of a few specific attributes.

6.3.2 Difference in terms of attributes

If a degree of difference greater than 1-2 was perceived by the judges they were asked to define the difference in terms of the given attributes. The attributes that were distinguished by the majority of the judges are shown in Figure 28. The most commonly identified attributes as shown in Figure 28 were firmness, creaminess and saltiness. The firmness and creaminess of both hollow fibre and spiral wound pilot plant cheeses were determined to be greater than the reference, whereas the saltiness was perceived to be less than the reference.

Other attributes identified by the judges were the sourness or acidity, colour, bitterness, brittleness and the presence of the aftertaste.

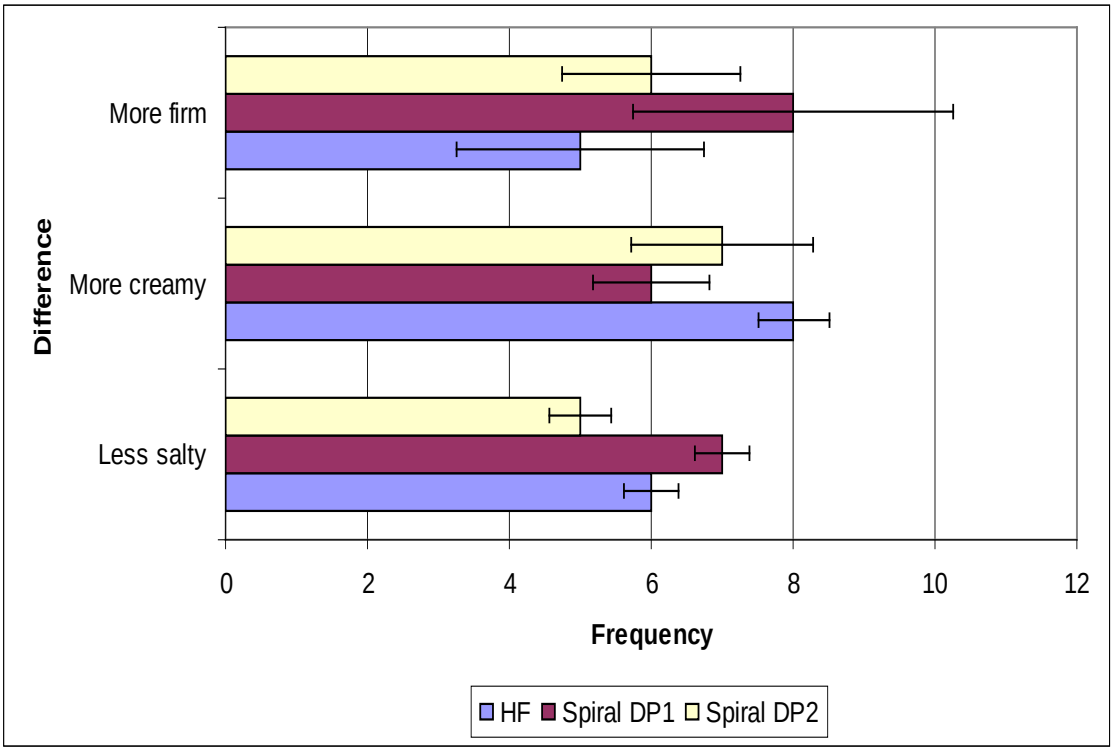

Figure 28. Plot of difference in terms of attributes as defined by the judges during sensory evaluation over a period of three months for cheese made on hollow fibre and spiral wound at transmembrane pressure of 1.0 and 1.3 bar compared to the reference feta cheese from PVC (n= 27 ± SE). The frequency represents the number of judges that perceived that specific attribute.

Figure 28 represents the attributes that were perceived to be different between the pilot plant feta cheeses and the PVC feta cheese from the sensory evaluation after three months of storage at 5 °C. As explained earlier the four replicates of cheese on both the pilot plant units were made such that the last two replicates of cheese were made a month after the first two replicates. Hence, the sensory data obtained during the project duration for the first two replicates was for four months, whereas the sensory data for the last two replicates was attained for three months. Due to incomplete set of data for the fourth month of sensory evaluation, Figure 28 represents an average of three months rather than four months data.

The same degree of difference perceived by the judges when comparing the pilot plant cheeses to the PVC cheese showed that the hollow fibre and the spiral wound cheese were very similar in terms of sensory.

In summary, the sensory evaluation shows that a moderate difference exists between the pilot plant cheeses and the PVC cheese. The difference was consistently moderate over three months of storage. It was determined that similar ripening process to that of PVC cheese takes place in the pilot plant cheeses. The same difference between hollow fibre cheese when compared to PVC cheese and spiral wound cheese when compared to PVC cheese shows that there is a similarity between the pilot plant cheeses. The differences between the pilot plant and PVC cheeses were perceived by the judges to be mostly in terms of saltiness, creaminess and firmness.

6.4 Microbiology

The microbiological quality of the cheese made from milk concentrated in the pilot plant ultrafiltration units was checked by the laboratory staff at Goodman Fielder, Longburn factory. The pasteurised, standardised and homogenised milk supplied was tested for microbiology before packing, hence no microbiology was done on milk before concentration. However, the concentrate was checked for microbiological contamination for batches that resulted in very high lipase activity.

The cheese was checked for *Staphylococcus aureus*, yeast and moulds, coliforms and *E.Coli*. Table 5 represents the microbiology results of cheese after four months of storage time. The *Staphylococcus aureus*, yeast and mould, coliforms and *E.Coli* are within the acceptable limit as specified by PVC.

Table 5. Microbiological results for all batches of cheese made from milk concentrated in ultrafiltration pilot plants after four months of storage time. All data is representative of four replicates.

Treatment	***Staphylococcus aureus***	Yeast and Moulds	Coliforms	***E.Coli***
Hollow fibre	<10	>250cfu/ml	<1	<1
Spiral DP1	<10	>250cfu/ml	<1	<1
Spiral DP2	<10	>250cfu/ml	<1	<1
Trigger Limits	>10	>500cfu/ml	>10	>10

6.5 Texture

The texture of the cheese made from the milk concentrated in the ultrafiltration pilot plants was analysed by determining the hardness and the fracturability of the cheese. The fracturability or brittleness is the force required to fracture the cheese sample which is obtained from the first peak of the compression curve. The hardness is the force required to get 70% compression of the sample, which is represented by the second peak of the compression curve (Mallatou et al., 1994).

The compression curve is generated for the one bite method, where the plunger goes directly down on to the sample and compresses to the specified compression and immediately back up to imitate a single bite (Mallatou et al., 1994). The fracturability and hardness of the cheese made from pilot plant concentrates and the reference are represented in Figure 29. The reference represents the feta cheese samples obtained from PVC with the same or closest production date to that when the cheese was made using concentrated milk from the pilot plants.

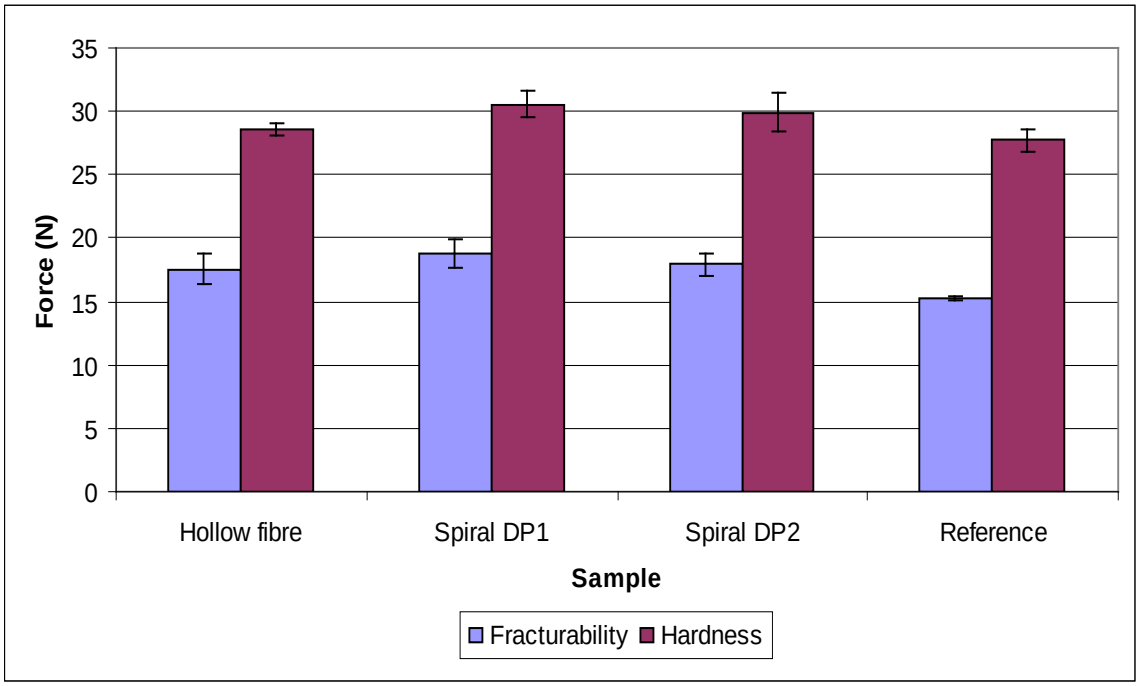

Figure 29. Graph of fracturability and hardness of cheese made using the concentrated milk from the hollow fibre unit at 0.3 bar differential pressure, spiral wound unit at differential pressure of 0.3 and 1.0 bar and the reference from PVC for all batches of cheese. All data are mean ± SE (n = 4 runs)

From Figure 29 it can be observed that the fracturability of the cheese made on hollow fibre and spiral wound units at both differential pressures was significantly higher than that of the reference from PVC. The fracturability or the force required to fracture the sample for cheese made using the pilot plant milk concentrates was considered to be greater due to the difference in the composition of raw milk used.

The fracturability could also be used as a measure of brittleness. The reference sample showed that it was more brittle than the pilot plant cheese, as it fractured at a lower force. Other factors contributing to the difference in fracturability of pilot plant cheese and PVC cheese could be the experimental errors associated with cutting the samples. The cheese sample with uneven surface fractures at a lower force, the uneven surface is the result of using the wire cutter to cut the cheese to size. It is vital to get the cheese samples to be precise 20mm cubes, as a larger surface area can withstand larger forces before it fractures.

The hardness of the cheeses made using the milk concentrates from the pilot plants were similar when compared against each other. The difference between the cheese made using concentrate from spiral wound plant and reference from PVC was higher than that of cheese made using the hollow fibre concentrate and the reference (PVC). Hardness of the cheese made from the concentrate from hollow fibre was very similar to that of the reference cheese from PVC when error bars are taken into account. The hardness is the force required to compress the cheese sample to 70% of its original height. The hardness is affected by the firmness, as the harder the sample the greater the force required to compress it.

7.0 Discussion

7.1 Composition

The composition of milk in terms of protein, fat and moisture content affected the properties of the final cheese. Increase in protein in milk results in more cross linking between the casein micelles, hence the gel produced from coagulation would be very firm (Fox, 2004). During coagulation the fat is entrapped within the casein matrix, acting as an inert filler in the gel produced. Decrease in fat content promotes more cross linking of the casein micelles therefore resulting in a more firm gel (Gunasekaran and Mehmet, 2003).

The salt uptake of the cheese is influenced by a number of factors such as the brine concentration, shape and size of the cheese blocks, type, structure and composition of cheese and the volume of brine to cheese ratio (Walstra et al, 1999; Tamime, 2006). The increase in brine concentration results in accelerated diffusion of salt. The shape and size define the surface area available for salt diffusion, hence smaller or flat surfaces have higher salt intake than large or cylindrical shapes. The salt diffusing into the cheese has to travel from the outer surface to the centre of the block through the casein network, encountering obstructions such as the protein strands and fat globules as they cannot be penetrated (Tamime, 2006). Hence, the composition and structure of the cheese contribute to the salt intake. During salt uptake, the moisture diffuses out and salt diffuses in. Therefore, the brine solution would decrease in salt concentration over time. The brine volume should be greater than five times the volume of cheese to overcome this phenomenon (Tamime, 2006). Alternatively, to maintain the brine concentration salt can be added and the surplus brine (due to the increase in volume from adding salt) is discharged.

The moisture lost into the brine is twice as much as the salt that has diffused into the cheese. The Na^+ and Cl^- ions are twice in size than that of H^+ and OH^- ions, therefore the brined cheeses are generally smaller and lighter than the unsalted cheeses (Tamime, 2006).

The composition of milk used at PVC was 3.5 ± 0.04 % $^{w}/_{w}$ protein, 4.3 ± 0.1 % $^{w}/_{w}$ fat and 13.4 ± 0.2 % $^{w}/_{w}$, total solids. The initial milk composition for the pilot plant cheeses was 3.1 ± 0.01 % $^{w}/_{w}$ protein, 3.3 ± 0.01 % $^{w}/_{w}$ fat and 12.0 ± 0.1 % $^{w}/_{w}$ total solids. The protein, fat and total solids content of milk concentrated on the pilot plants was slightly lower than the milk concentrated at PVC. The total solids in the concentrated milk and permeate flux data for the pilot plant runs are as shown in Appendix - D.

7.1.1 Comparison of cheese made using Hollow fibre unit against PVC cheese

The composition of the feta cheese made from milk concentrated using the hollow fibre pilot plant unit was 14.5 ± 0.2 % protein, 17.5 ± 0.3% fat, 2.8 ± 0.1% salt and 40.2 ± 0.3 % total solids, all values are expressed as %$^{w}/_{w}$. The composition of the feta cheese from PVC was 14.0 ± 0.1% protein, 20.8 ± 0.2% fat, 3.8 ± 0.1% salt and 42.2 ± 0.1% total solids, all measurements are on $^{w}/_{w}$ basis.

The protein content in the cheese made using concentrated milk from the hollow fibre pilot plant was very similar to that of the cheese from PVC. The fat content in cheese made from hollow fibre concentrated milk was significantly less than the cheese from PVC. The lower fat content in the hollow fibre cheeses was due to the lower fat percentage in the milk used for concentration on the pilot plants than that of the milk used at PVC.

The lower fat content decreases the number of fat globules entrapped in the casein matrix, which results in less obstruction for the salt molecules diffusing in. Therefore the salt content in the hollow fibre cheeses should have been greater than the PVC cheese. However, the results contradicted this theory, the hollow fibre cheeses were found to have lower salt content than the PVC cheeses. When further investigated it was found that the volume of brine was equal to the volume of cheese during brining. As stated earlier the brine concentration decreases during brining and lower concentrations of brine resulted in lower salt intake. Hence, the lower salt percentage in the cheese made from hollow fibre concentrated milk. Other factors affecting the

salt content are the initial concentration of brine used for salting the cheese, the range of brine concentrations that is acceptable is between 80 – 90% saturated solution.

Higher moisture content results from a lower salt content in the cheese, as the salt concentration in hollow fibre cheese was less than the PVC cheese, the moisture content in hollow fibre cheese was higher than the PVC cheese. The total solids content is inversely proportional to the moisture content present in the milk, concentrated milk or cheese. Therefore, the moisture content differences in the hollow fibre cheese and PVC cheese could also be due to the differences in the total solids of the concentrate used to make the respective cheeses. At PVC the total solids in the concentrated milk are at 37.5 ± 0.5% where as, the total solids in the milk concentrated using the pilot plants was at 37.5 ± 2%. The method used to measure the total solids on the pilot plants was not as accurate as the method used at PVC.

The total solids in the concentrate at PVC were checked using the microwave oven which is a rapid method for checking for total solids and the results were obtained within two minutes. The determination of total solids in the milk concentrate from hollow fibre involve using a dry air oven, which requires at least four hours of drying in the oven. Hence, a refractometer was used to monitor the total solids during the run as it requires less time. The refractometer was used to monitor the total solids level in the milk during the run and the final total solids for each run was checked using the air drying oven. The total solids content determined by the refractometer were with in ± 2% of the measurements obtained from the dry air oven.

The cheese made from the concentrated milk from the hollow fibre unit was very similar to the cheese from PVC. The differences in terms of fat and salt can be eliminated by using the same milk as at PVC to concentrate on the hollow fibre unit and by using a greater volume of brine to salt the cheeses. Hence if the same milk were used in both PVC and hollow fibre pilot plant a cheese of the same composition and physical properties could be produced.

7.1.2 Comparison of cheese made using Spiral wound unit against PVC cheese

The protein content in feta cheese based on the nutritional panel on the package of feta cheese manufactured at PVC should be at 15% by weight. The protein percentage of cheese made on the spiral wound ultrafiltration unit at transmembrane pressure of 1.0 bar and 1.3 bar was slightly higher than the reference cheese sample from PVC. The protein percentage in the cheese made on the spiral wound pilot plant unit at transmembrane pressure of 1.0 bar was 15.0 ± 0.2 % $^{w}/_{w}$ and the cheese made at transmembrane pressure of 1.3 bar was 15.0 ± 0.2 % $^{w}/_{w}$ where as the reference cheese was 14.0 ± 0.1 % $^{w}/_{w}$. The cheeses made using the concentrate from spiral wound unit have the same protein content, where as the protein content of reference sample is 1% higher. This slight variation of the protein content between the pilot plant cheeses and the reference cheese is considered to be due to the presence of higher protein content in the milk used to make the PVC cheese than the milk used to make pilot plant cheeses as mentioned earlier.

The fat content was determined to be 18.4 ± 0.3 %, 18.4 ± 0.6 % and 20.8 ± 0.2 % $^{w}/_{w}$ in the cheeses made from the concentrated milk from the spiral wound ultrafiltration unit at transmembrane pressures of 1.0 and 1.3 bar and the reference cheese from PVC, respectively. As mentioned earlier the fat content in the milk concentrated on spiral wound was lower in fat content than that of the milk concentrated on the PVC ultrafiltration system. The protein content of the pilot plant cheeses was very similar to that of the PVC cheese, therefore if milk with higher fat to protein ratio was used for concentration on the pilot plants the fat content would be closer to the PVC cheese.

The salt content expressed as a percentage of cheese (by weight) in the cheese made from milk concentrated in the spiral wound unit at transmembrane pressure of 1.0 bar and 1.3 bar was 2.8 ± 0.1 % and 2.7 ± 0.1 %, respectively. The salt content in the reference cheese sample from PVC was 3.8 ± 0.1 %. It was observed by the staff at PVC that there is variation in cheese manufactured in terms of salt content. This variation in salt content at PVC is considered to be due to the operators being busy and forgetting to take the cheese out of brine precisely after 90 minutes. As all the

cheese made on the pilot plants was brined for 90 minutes, the significant difference in the salt content between the pilot plant cheeses and reference could be due to the wide range of concentrations of brine solutions used to salt the cheeses as stated earlier (section 7.1.1). The volume ratio of brine to cheese also contributes significantly to salt intake as mentioned above (Walstra et al, 1999).

The total solids percentage of cheese made from spiral wound concentrates at transmembrane pressures of 1.0 bar and 1.3 bar were 40.0 ± 1.0 % and 39.8 ± 0.7 % by weight. The reference sample had a total solids content of 42.2 ± 0.1 % by weight. The lower total solids in the pilot plant cheeses were considered to be due to the variation in total solids content of concentrated milk, different fat to protein ratio of the milk used to make cheese and the lower salt content. At PVC the total solids in the cheese is aimed to be in the range of 36 – 44 %. The total solids percentage for the pilot plant cheeses was slightly lower than that of the reference cheese sample but still within the acceptable range required by PVC.

7.2 Texture

The most significant factor affecting the texture of cheese is the fat content, as the fat globules act as filler material preventing formation of cross linking between the casein micelles in cheese (Gunasekaran and Mehmet, 2003 and Walstra et al, 1999). This decrease in cross linking results a cheese that is less firm.

Mallatou et al. (1993) reported that the fracturability force can be used as a measure of firmness or the brittleness of the sample. As fracturability is a measure of the force required to fracture the sample, higher fracturability forces are found in more firm or less brittle samples. The hardness is the force required to compress the sample to the required height.

7.2.1 Comparison of cheese made using Hollow fibre unit against PVC cheese

The fracturability and hardness of the cheese made using the concentrated milk from the hollow fibre unit was 17.6 ± 1.2 N and 28.6 ± 0.5 N, respectively. The reference feta cheese from PVC had a fracturability of 15.3 ± 0.2 N and hardness of

27.7 ± 0.9 N. The higher fracturability and hardness values for the hollow fibre feta cheese than the PVC cheese shows that the hollow fibre cheese was slightly more firm (but not significantly) and required more force than that of the reference feta cheese sample from PVC to compress to 70% of the original height.

The most important factor affecting the firmness of the feta cheese was the differences in the composition of the milk used at PVC and for the hollow fibre pilot plant prior to making the cheese. As mentioned above fat entrapped by the casein structure of the cheese gives the cheese its softness (Madadlou et al., 2007). Therefore, the composition of milk used as raw material for cheese has an influence on the final texture of the cheese. The presence of less fat in the milk before concentration leads to less fat in the subsequently concentrated milk and the final cheese made from this milk. Hence, as the milk concentrated in the pilot plant hollow fibre unit had a slightly lower level of fat, the cheese made was firmer than the reference cheese from PVC.

7.2.2 Comparison of cheese made using Spiral wound unit against PVC cheese

The fracturability of the cheese made on the spiral wound UF unit at the transmembrane pressure of 1.0 bar (Spiral DP1) was 18.8 ± 1.1 N. The fracturability of the spiral wound cheese made with the transmembrane pressure of 1.3 bar (Spiral DP2) and the reference cheese from PVC were 17.9 ± 0.9 and 15.3 ± 0.2 N, respectively. The firmer samples require greater force to fracture the sample beyond the recovery point; hence the cheeses made from concentrated milk from spiral wound unit are more firm. The hardness was 30.6 ± 1.4, 29.9 ± 1.5 and 27.7 ± 0.9 N for spiral wound cheeses made at 1.0 and 1.3 bar transmembrane pressure and PVC reference cheese, respectively. The spiral wound cheese at 1.0 bar transmembrane showed the highest firmness and the highest hardness.

The differences in the composition of the milk used at PVC and for the spiral wound pilot plant UF units to make the cheese contributed to the changes in texture of the cheese made, especially in terms of the firmness. The fat to protein ratio in the PVC

reference cheese is greater than that of the spiral wound cheeses, hence the PVC cheese was softer or less firm than the spiral wound pilot plant cheeses.

7.3 Lipase activity

7.3.1 Comparison of cheese made using Hollow fibre unit against PVC cheese

The lipase activity for the cheese from the hollow fibre concentrated milk was higher than the reference from PVC. Voutsinas et al (1995) states that the heat treatment of milk and concentrate before, during and after ultrafiltration affects the lipase activity in the cheeses. They also consider that severe heat treatment causes greater lipase inactivation.

The cheese making process using pilot plants was investigated thoroughly to identify any likely areas of contamination when some batches of cheeses showed very high lipase activity. It was identified that there was potential for cross contamination while brining as experiments with meat were conducted close to the brining area. As mentioned earlier to prevent this brining was set up in an enclosed sterilised area. Therefore, the high lipase activity in the cheeses made from milk concentrated on the pilot plant units was considered to be due to the incomplete inactivation of the lipase enzyme present in the milk.

The lipase activity after four months of shelf life was 3.35 ± 0.1 and 2.00 ± 0.04 ADV for cheese from hollow fibre concentrated milk and the reference cheese from PVC respectively. The lipase activity in both cheeses made with hollow fibre concentrate and at PVC showed an increase from two to four months of storage time. Mallatou et al. (1994) showed that there was a slight but constant increase in lipase activity in feta cheese made from sheep's, goats' and a mixture of both milk when stored for 120 days at -20 °C. Although they do not provide an explanation for the gradual increase in lipase activity, Georgala et al (2005) and Svensson et al (2005) state that lipase activity increases due to fat hydrolysis during ripening.

Deeth and Fitzgerald (1976) state that cheese greater than ADV of 3 is rancid. Hence the feta cheese from the hollow fibre milk concentrate would be considered to be

rancid after 4 months storage at 5 °C. However, they base their results on cheddar cheese and do not specify if the milk used was cow's milk. Mallatou et al. (1994) state that there was data reported where ADV between 4.7 and 22.6 for sheep's milk feta cheese was considered to be not rancid. The panel of judges from the sensory evaluation did not perceive the cheeses to be rancid up to an ADV value of 5. Therefore, the cheese made from milk concentrated on hollow fibre unit could be considered as acceptable in terms of lipase activity.

7.3.2 Comparison of cheese made using Spiral wound unit against PVC cheese

The lipase activity after two months of shelf life has an acid degree value (ADV) of 4.7 ± 1.0 and 3.0 ± 0.2 for cheese made from milk concentrated using the spiral wound unit at transmembrane pressure of 1.0 bar and 1.3 bar, respectively. The reference cheese from PVC after two months shelf life had an acid degree value of 1.500 ± 0.004 as mentioned earlier. The ADV values of all cheeses made using the spiral wound pilot plant to concentrate the milk are higher than that obtained for the reference from PVC.

Deeth and Fitz-Gerald (1976) state that greater that an ADV of 3.0 in any cheese is to be considered to have a detectable level of rancidity, they also state that good quality cheddar cheese is to have an ADV between 1.2 – 1.8. The ADV of fresh feta cheese on day one made from goat's milk is reported to be 1.8, after 60 days the ADV has increased to 2.2.

The reference samples were found to have an ADV of 2.00 ± 0.04 after four months of storage. As mentioned earlier, Mallatou et al. (1994) found cases where 4.7 – 22.6 of ADV in sheep and goats' milk feta cheese were perceived to be not rancid. All cheese made using the concentrated milk from spiral wound pilot plant and the reference cheese show an increase in the lipase activity with increase in storage time from two to four months.

The high lipase activity in the pilot plant cheeses may be due to contamination through other products such as meat from other experiments in the laboratory (where the pilot

plants were located) that have lipase or more likely due to incomplete inactivation of the lipase enzyme present in milk that was concentrated. The lack of complete inactivation of the lipase enzyme in concentrate resulted in higher lipase activity in the cheese made.

7.4 Sensory

The ripening process that takes place over time consists of proteolysis, lipolysis and glycolysis. Proteolysis is the breakdown of proteins into lower molecular weight compounds and amino acids which give the cheese its texture and structure (Gunasekaran and Mehmet, 2003). Lipolysis the process of breaking down the fat into free fatty acids which are precursors for flavour compounds in cheese. Glycolysis is the production of lactic and citric acids (Fox, 2004). Proteolysis and lipolysis are significant in terms of sensory as they contribute to the texture and flavour compounds.

The most significant differences identified between the pilot plant cheeses and the PVC cheeses by the panellists in terms of sensory were the firmness, creaminess and the saltiness. The pilot plant cheeses were perceived to be more firm, creamier and less salty than the reference cheese from PVC. The attribute differences measured were qualitative, they were detected as either more or less than the reference. Therefore, the results from the sensory evaluation cannot be used to determine magnitude of the difference between the pilot plant cheeses and PVC cheese.

7.4.1 Comparison of cheese made using Hollow fibre unit against PVC cheese

The average degree of difference between the cheese made using hollow fibre concentrated milk and the reference cheese from PVC remained at a constant of 3, which represented a moderate difference on the sensory scale. The same degree of difference over the storage time of four months suggested that the changes occurring in the PVC cheese due to ripening were similar to that of hollow fibre concentrated milk cheese.

The statistical analysis of the data collected for sensory from the panellists shows that there was a significant difference in the cheese made using the concentrated milk

from the hollow fibre pilot plant unit and the ultrafiltration system at PVC. The difference between the hollow fibre concentrated milk cheese and the cheese from PVC was defined by the judges in terms of colour, brittleness, bitterness, sour or acidic, firmness, creaminess and saltiness attributes. The most significant attribute differences in the cheese made from the milk concentrated by the hollow fibre and the PVC cheese were saltiness, creaminess and firmness.

7.4.2 Comparison of cheese made using Spiral wound unit against PVC cheese

The sensory differences defined in terms of the attributes of the cheese made from concentrating milk on spiral wound unit at differential pressure of 0.3 and 1.0 bar were that the cheese was firmer, more creamy and less salty than the reference.

The panellists perceived the presence of an after taste with some replicates of cheese made by milk concentration using spiral wound unit and they were found to have very high lipase activity. Fox (2004) states that lipase activity affects the flavour and textural aspects of cheese. It was observed that the cheese with higher lipase activity caused textural defects such as softness in cheese, hence making the cheese appear creamier or less brittle when consumed. All the cheese made from the concentrated milk from spiral wound unit had higher lipase activity than the reference, hence all the cheese made using the spiral wound pilot plant was perceived by the judges to be creamier than the reference from PVC.

Cheese contains various compounds, which contribute to the flavour, texture, and how the consumer perceives it. The cheese texture and flavour perceived by the panellists is the balance of the various compounds rather than the quantity of each individual compound present in cheese (Delahunty, 2002). One of the factors effecting creaminess is the fat content, increase in fat content would cause cheese to be creamier (Fox, 2004). However, the results show that although pilot plant cheeses have a lower fat content than PVC cheese they are creamier. This shows that other factors affect the creaminess of cheese.

The results from the texture analysis show that the cheeses made from both hollow fibre and spiral wound concentrated milk were firmer than the cheese from PVC. As a decrease in fat content contributes to an increase in the firmness and hardness of the cheese (Sipahioglu et al, 1999), the texture results are in accordance with chemical analysis results in terms of composition. The chemical analysis for salt content in the cheeses was in agreement with the sensory results, the judges perceived the cheese from the milk concentrated on hollow fibre and spiral wound units were lower in salt content than the PVC cheese. The panellists perceived that the cheese made utilising milk concentrated by both pilot plant units were firmer than the PVC cheese; this is in agreement with the chemical analyses and the texture results.

In conclusion, the differences between the cheese made from concentrated milk by either hollow fibre or spiral wound ultrafiltration units when they were compared against the reference cheeses from PVC, showed that there was a difference in terms of texture, fat and salt content, lipase activity and sensory evaluation. However, these differences were the same in both the pilot plant cheeses, this shows that the cheese made from hollow fibre concentrated milk was very similar to the cheese from concentration of milk using the spiral wound unit.

Making cheese on the hollow fibre and spiral wound pilot plants using milk with complete lipase inactivation and a higher fat content, plus salting with a higher volume of brine to cheese should result in the production of the same quality of cheese as at PVC.

8.0 Conclusions

Feta cheese was successfully produced using the concentrated milk from both hollow fibre and spiral wound pilot plant units. The operating performances of both the pilot plant units were very similar to that of PVC hence the milk was treated similarly as at PVC during concentration. The hollow fibre and both spiral wound pilot plant cheeses were very similar to each other in terms of protein, fat, moisture, lipase activity, texture and sensory evaluation. The similarity in quality of cheeses made on the spiral wound unit at both the differential pressures and the hollow fibre pilot plant cheese suggests that the effect of differential pressure and TMP are insignificant. However, the cheese made on the hollow fibre pilot plant was different from that made on the hollow fibre ultrafiltration system at PVC.

The hollow fibre pilot plant cheese differs from the cheese made using the PVC ultrafiltration system in terms of fat to protein ratio, salt and moisture content, lipase activity, texture and sensory. The difference in fat to protein ratio is considered to due to the difference in fat to protein ratio in the milk concentrated on the hollow fibre pilot plant and the milk used at PVC. The lower salt content in hollow fibre pilot plant cheese was considered to be due to the low ratio of brine volume to the cheese volume used during brining. The lipase activity in the hollow fibre pilot plant cheese was higher than the PVC cheese. The high lipase activity was believed to be due to incomplete inactivation of the lipase enzyme present in the milk that was concentrated on the hollow fibre pilot plant.

The texture results show that the hollow fibre pilot plant cheese is similar to the spiral wound cheeses but is more firm than the PVC cheese. The sensory results show that the panellists perceived that the salt content in the pilot plant cheeses is lower than the PVC cheese and the pilot plant cheeses are more firm than PVC cheese. The results from the texture and sensory are in agreement with the fat, salt and moisture differences detected from the chemical analysis.

The cheeses made on the spiral wound unit at both differential pressures and the hollow fibre unit are the same in quality in terms of composition, texture and sensory evaluation.

The same quality of cheese as made at PVC should be able to be produced using both the pilot plant units provided that the milk that was to be concentrated is of the same composition and had the same heat treatment as that of PVC milk and the brine volume to cheese volume during brining was similar to that of PVC.

9.0 Recommendations for further work

Hollow fibre membranes at PVC could be replaced with spiral wound membranes as the same quality of cheese can be made using both membranes. To ensure that the same quality of cheese made during every batch, it is recommended that milk should be standardised to maintain the fat to protein ratio and eliminate any texture discrepancies. It is also highly recommended that it is checked that complete inactivation of lipase enzyme takes place during pasteurisation and that the volume of brine is five times larger than the cheese volume during brining of cheese.

The desired concentration of total solids can be achieved using both hollow fibre and spiral wound membranes. The performance of the hollow fibre and spiral wound pilot plant membranes cannot be directly compared due to different membrane surface areas. However, the pressure drop per unit length was found to be greater when the hollow fibre membrane was used. The lower pressure drop in the spiral wound runs results in better transmembrane pressures and less loading on the pumps, hence reducing the run time and the operating costs. Therefore, it is recommended that hollow fibre membranes at PVC be switched with spiral wound membranes as the decrease in run time and lower pumping loads would reduce the operating costs.

The spiral wound membranes are liable to less fouling due to the presence of the feed spacer which induces turbulence and lower run time potentially reduces the exposure of the membranes to high total solids hence, less fouling.

If the spiral wound membranes are more economical in terms of price than hollow fibre membranes, it is strongly recommended that the hollow fibre membranes be replaced.

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RB 08.05.01	Feta Batchsheet	Version 10	February 2007	Page

FETA	5	DAY 2 1 1	RUN A	PASTEURISER OPERATOR	Silo 3	Delivery date 29/7	Vol 10000
					Silo	Delivery date	Vol

Fat	Protein	TS	TA	pH	Conc Factor 22-40	Theo Yield	Final Volume	Cook Time	Cook Temp	Cool Temp	Homo Press
4.36	3.68	13.29	0.144	6.67		2040 l	2160 l	9.50	79.2	30.1	400 P
↓	↓	↓	↓	↓				10.00	77.6	31.2	"
UF	Time	Feed Press	Boost Pres	Temp	Perm Flow	Per Volume		10.10	80.5	30.1	"
	6.30	1.10	2.10	42.07	1.27/1.50			10.20	25.7	29.8	"
	9.43	1.10	2.10	46.35	0.37/0.42						

Start Up Sanitise	Xy12 200ppm	Temp Aim 50°c	Time Aim 15 min	Water Rinse	Wash Post Production	Caustic Wash 2l	Time Aim 15m	Temp Aim 60°c	Xy12 200ppm	Temp Aim 50°c	Time Aim 15m	Water Rinse	Sign O
Vat 2	200 ppm	53°C	15 m	✓	Vat 2								
Vat 3	X	X	X	X	Vat 3								
Ricotta	X	X	X	X	Ricotta								
Siteglass	✓	✓	✓	✓	Siteglass								

Inoculation/incubation of starter R704 — **Cooking operator:**

Starter R704 Batch Number	Addition rate Units/litres	Actual amount	Milk volume	Milk temp at set	Bulk starter Set time	Conc. temp	Time bulk Starter added	Incubation time
2741228	500 u/L	142 g	2l	30.5	10.10	29.7	10.30	30 mins

Pour operators: — **Cut operators:**

Type	Size Required	Aim	Actual tubs	Ingredients		Batch code	Time Added
Basil	200 g	9 tubs	9 tubs	1 bag per tub	Basil pesto 1.25%	230707	11.00
Capsicum				1 bag per tub	Chargrilled capsicum		
Garlic/Cumin				1 bag per tub	Garlic/cumin pr2.8		
Tomato				1 bag per tub	Sundried tomato 3.33%		
Lemon/Corian				1 bag per tub	Coriander/lemongrass		
Olive				1 bag per tub	Olive Tapenade		
Rocket/pesto				1 bag per tub	Rocket/pesto.		
Plain cow	200 g	9 tub	8 tubs		[redacted]		
Plain cow					Sign		
Plain cow					**RELEASE TO PACKING**		
Plain goat					**SIGN**		
Chevre Salade					**Batchsheet Complete**		

DIP BASE = 37.71 — RENNET 1ML 4% RENNET/LITRE CONCENTRATE

Pour Start Time:	11.00	pH Start of vat:	6.54	Total solids:	pH End of vat:	6.50	Pour Finish Time:	11:50

Appendix – B. Ultrafiltration operating procedure

Start up

- Switch on power on the wall
- Fill feed tank with pure de-ionised water
- Check that the permeate and bleed off valves are closed
- Open the valve connecting the feed tank to the feed pump
- Open the bleed off valve
- Turn the feed and boost pump on
- Open the permeate valve
- Flush the ultrafiltration unit for 10 minutes with pure de-ionised water
- Check and record the inlet and outlet pressure, permeate flux of the pure water

Sanitising

- Half fill the tank with de-ionised water
- Close the bleed off and permeate valve
- Add appropriate amount of Ultrasil 91 (Caustic solution; Ecolab) to bring the pH to 10.5 – 11.
- Add XY12 (Sodium hypochlorite sanitiser solution; Ecolab) to obtain 180 - 200 ppm.
- Open the bleed off valve and turn on the feed and boost pumps
- Open the permeate valve
- Place the permeate outlet stream and bleed off stream back into the feed tank (to operate in 100% recycle mode)
- Run the ultrafiltration unit with the caustic sanitising solution for 30 minutes.
- Turn off the feed and boost pumps after 30 minutes.
- Close the bleed off and permeate valves.
- Drain the caustic sanitiser solution from the feed tank.
- Fill the tank with de-ionised water
- Open the bleed off valve and turn on the feed and boost pumps
- Open the permeate valve
- Flush out the sanitising solution with de-ionised water for 10 minutes.
- Turn off the feed and boost pump

- Close the bleed off and permeate valves.

Milk concentration on hollow fibre unit

- Drain any de-ionised water left in the feed tank
- Pre heat the milk to 45 - 50ºC using the steam jacketed vessel and pour gently into the feed tank to avoid formation of froth.
- Connect the hot water supply (55 ± 5 ºC) to the water jacket to maintain the temperature of the milk.
- Open the bleed off valve and turn on the feed and boost pumps
- Open the permeate valve
- Drain the bleed off outlet stream for a couple of minutes to avoid dilution of milk from the hold up volume of de-ionised water in the unit.
- Return the bleed off stream to the feed tank once the milk starts coming out, ensure that it is placed 5 cm below the surface of the milk.
- Place the permeate outlet stream into a container and measure the total permeate collected during the run
- Adjust the pressure in and out using the feed valve and recirculation valve on the pumps.
- Adjust the recycle rate using the bleed off valve.
- Record the flow into the UF unit, pressure in and out, permeate flow rate, and recycle rate, temperature and the total solids every 20 minutes.
- Adjust the recycle stream such that the differential pressure is maintained as constant through out the run.

NOTE: As viscosity increases over time during the run, the volumetric flow rate increases, which increases the pressure in and out, altering the differential pressure. Hence, it is important that the recycle rate is adjusted as appropriate to maintain the differential pressure.

- Once the required total solids are achieved, shut off the feed and boost pumps, bleed off and permeate valves.
- Drain the concentrate from the feed tank into sanitised containers.

- Rinse the feed tank and ultrafiltration unit with de-ionised water to remove the traces of concentrate by flushing the unit with de-ionised water for 10 minutes.
- Rinse the ultrafiltration unit with water until clear water is observed coming out of the bleed off stream.
- Turn off the feed and boost pumps and close the permeate and bleed off valves

Cleaning

- Half fill the tank with de-ionised water, add Ultrasil 91 to adjust the pH to 10.5 – 11.
- Add 7 mL of E2001 (enzyme cleaner; Orica Chemnet) for every 1 mL of Ultrasil 91 added to the feed tank.
- Mix well and turn on the feed and boost pumps and open the bleed off and permeate valves.
- Operate the UF unit on 100% recycle mode for 30 minutes.
- Turn off the feed and boost pumps after 30minutes, close permeate and bleed off valves.
- Drain the enzyme caustic solution and rinse with de-ionised water for further 10 minutes.
- Follow the sanitising procedure mentioned above, record the permeate flux, pressure in and out and leave the membrane sitting in pure de-ionised water until the next time it is operated.

The above mentioned operating procedure is for hollow fibre pilot plant, however it can be used for spiral wound pilot plant with the following changes.

- Hot water supply (55 ± 5 ºC) to be connected to the heat exchanger instead of the water jacket.
- The pumps on the spiral wound unit are on variable speed drive (VSD), the pumping capacity of the pumps can be adjusted using the VSD.
- There is no feed valve on the spiral wound unit, there is a recirculation valve which can be used to adjust the pressures.

- There is no permeate valve on the spiral wound unit, therefore the permeate flow rate cannot be shut off.
- The flow into the UF cannot be measured on the spiral wound unit due to a fault in the flow meters.

Appendix – C. Methods

Protein in cheese (Kjeldhal method, AOAC 2001.14 (33.7.12A)) (AOAC International, 2005)

Equipment:

Weighing scale (Mettler AE 260, Deltarange)

Grater

Filter papers (Whatman 50, Hardened, 12.5 cm)

Spatula

Kjeldahl digestion tubes

Kjeldahl digestion unit (2006 Digestor, Foss tecator)

Kjeldahl distillation unit (2100 Kjeltec ™ Distiller, Foss tecator)

50mL Burette

250mL Conical glass flasks

500 mL Volumetric glass flasks

Solutions:

Kjeltabs (Foss, 1000 Kjeltabs Se/3.5, 3.5g K_2SO_4, 3.5mg Se)

95-98% Sulphuric acid (BDH, Analytical reagent grade, specific gravity 1.84)

40% Sodium hydroxide solution (BDH, Analytical reagent grade, 99.0% purity)

4% Boric acid solution (Sigma, Sigma Ultra grade, 99.5% purity)

0.1M Hydrochloric acid (BDH, Analytical reagent grade, specific gravity 1.18)

Distilled water

Method:

1. Sample preparation.
    - Cut the cheese into strips
    - Chop the strips into cubes
    - Grate the cubes of cheese using the grater

2. Digestion
    - Accurately weigh 1g of the blended cheese onto a pre-folded filter paper

- Fold the filter paper such that the cheese sample is completely enclosed and does not fall out.

NOTE: While folding make sure none of the sample falls out as this would give inaccurate results.

- Place the enclosed sample along with the filter paper in the digestion tube.
- Prepare two blanks for each set of samples being digested; each blank consists of the filter paper without the sample with all the reagents.
- Clean the Kjeldahl digestion unit, especially the heating surface and the fume collection area.
- Add two kjeltabs to the digestion tube
- Add 18mL of concentrated sulphuric acid to each digestion tube inside the fume hood.
- Swirl the digestion tube to ensure the kjeltabs and all of the filter paper with sample inside is wetted completely by the sulphuric acid.
- Set the digestion tubes onto the digestion unit and lower the stoppers.
- Turn on the heating and set the time to 5hours, temperature to 250 ± 5 °C.
- Over 1 hour gradually increase the temperature to 420 ± 2 °C.
- After 4 hours, check the digestion tubes, the solution should be a clear solution without any of the digest left.
- If any digest noticed further heat for 2 hours.
- Once the clear solution is achieved, cool the digested solution to room temperature.

NOTE: digestion and distillation can be done on different days.

3. Distillation
    - Add 70mL of distilled water to the digestion tube.
    - Start the distillation unit steam supply.
    - Carefully place the digestion tube on the tube holder and ensure the stopper is in the correct position by giving the tube a twist once in position.
    - Measure 50mL of boric acid in a 250mL conical flask and place it on the receiving platform with the tube from the condenser.

- Lower the hood over the digestion tube for the steam distillation process to start.
- The sodium hydroxide solution is added automatically by the distillation unit as per the settings.
- The distillation is performed for 3 minutes after which the distillation unit stops automatically.
- Wear rubber gloves and push down the tube holder slightly to remove the digestion tube.
- Empty the contents of the digestion tube into the sink
- The flask is removed from the receiving platform to be titrated.
- Fill the burette with 0.1M HCl solution.
- Titrate the contents of the flask with the 0.1M HCl solution until an end point of grey-mauve colour is achieved.
- Record the required amount of HCl to reach the end point.
- Calculate the % Nitrogen using the following equation:

$$\% \text{ Nitrogen} = \frac{m \times (V_s - V_b) \times M}{W} \times 100$$

Where m is the molecular weight of nitrogen (g/mol)

V_s is the volume of HCl used in the titration of the sample (mL)

V_b is the volume of HCl used in the titration of the blank (mL)

M is the molarity of the HCl solution (mol/L)

W is the weight of the cheese sample (g)

- The % Protein can be calculated by using the following correlation:

$$\% \text{ Protein} = \% \text{ Nitrogen} \times 6.38$$

4. Cleaning
    - Wash all the glassware using hot water and a scrub.

NOTE: Ensure that none of the glassware to be used for the Kjeldahl method is washed with dishwashing liquid as it contains nitrogen hence would produce inaccurate results.

- Sterilise all the glassware.

Fat in cheese (Modified Schmid-Bondzynski-Ratzlaff method, ISO 1735: 198 E) (Ceirwyn, 1995)

Equipment:

Mojonnier flask

250mL Conical flasks

Water bath (Naake W19 Fisons, Type: KA/TA)

Weighing scale (Mettler AE 260, Deltarange)

Measuring cylinder

Grater

Centrifuge (Funke Gerber, Super Vario-N, 1998)

Solutions:

Hydrochloric acid (prepared by diluting 675mL of concentrated hydrochloric acid to 1L with water) (BDH, Analytical reagent grade, specific gravity 1.18)

Diethyl ether (BDH, Analytical reagent grade)

Petroleum ether (40-60) (BDH, Analytical reagent grade)

Ethanol (ASCC (Australasian solvents & chemicals company Pty Ltd), 99.8% ethanol solution denatured with 2% methanol, SDA-3A)

Method:

- Grate cheese and place 1g of cheese into the Mojonnier tube.
- Add 8-10mL of hydrochloric acid to the tube and mix well.
- Heat tube in boiling water bath until all particles are dissolved completely.
- Leave tube in boiling water bath for further 20-30min.
- Cool and add 10mL ethanol, mix gently for 30 seconds.
- Add 25mL of diethyl ether, mix well for 1 minute
- Add 25mL of petroleum ether, mix well for 1 minute.
- Centrifuge for 3 minutes at 600 rpm at room temperature.
- Pour the top layer into a pre-weighed conical flask without disturbing the bottom layer.

- Repeat the second extraction with 5 mL of ethanol, mix well for 30 seconds and 25mL of each solvent, mix well for 1 minute and add the top layer to the conical flask.
- Repeat the third extraction with 25mL of each solvent, mix well for 1 minute and add the top layer to the conical flask.
- Distill off the solvent and dry the flask for 4 hours at 100°C and reweigh the flask with extracted fat.
- Calculate the fat content using the following equation

$$\% \text{ Fat content of cheese} = \frac{w_2 - w_1}{w_3} \times 100$$

Where w_1 is weight of empty flask (g)

w_2 is weight of flask + fat (g)

w_3 is weight of cheese taken (g)

Total solids in Cheese (AOAC official method 926.08) (AOAC International, 2005)

Equipment:

Weighing scale (Mettler AE 260, Deltarange)

Metal dish (>= 5 cm in diameter) with tight fitting lid

Dry air oven

Desiccator

Method:

- Weigh the metal dishes and the tight fitting lids.
- Record the weight of dish and lid (to 3 significant figures).
- Weigh 3-4 g of cheese sample into the dish and cover with lid.
- Label the dish and lid accordingly.
- Place the dish with lid and sample in the oven set at 105°C.
- Remove the lid once the dish is placed inside the oven.
- Place the lid back on the dish after 4 hours and remove the dish from oven.
- Cool the dish for half hour in the dessicator.
- Check weight of the dish with sample and lid and record.
- Place the dish with lid and sample back in the oven.
- Repeat the above procedure until a constant weight is achieved.

$$\text{TS (\%)} = \frac{(w_2 - w_1) - (w_3 - w_1)}{w_2} \times 100$$

Where w_1 weight of dish and lid

w_2 weight of cheese, dish and lid before placing in oven

w_3 weight of cheese, dish and lid after taking out of oven

Moisture Content (%) = 100 - TS (%)

Salt in Cheese (AOAC Official method 935.43)

Equipment:

Weighing scale (Mettler AE 260, Deltarange)

200mL Conical glass flasks

Measuring cylinder

Hot plate (IEC (Industrial equipment & control Pty Ltd) cat no. CH2092-001)

Filter paper (Whatman 50, Hardened, 12.5 cm)

Solutions:

0.1M $AgNO_3$ (Ajax chemicals, Analytical reagent grade (Univar))

Concentrated HNO_3 (Ajax chemicals, Analytical reagent grade (Univar), 70% $^{w}/_{w}$)

0.1M KSCN (Potassium thiocyanate) (Sigma, ReagentPlus ™ grade, $\geq$ 99.0%)

Ferric alum (indicator, saturated solution) (BDH, Analytical reagent grade)

Method:

1. Sample preparation.
    - Cut the cheese into strips
    - Chop the strips into cubes
    - Blend the cheese cubes in the blender for 2-5 minutes or until a homogenous mixture is obtained.

    NOTE: Occasionally stop and spoon the cheese towards the blades.

2. Titration.
    - Weigh 3g of the prepared cheese sample into the 200mL flask.
    - Add 10mL of distilled water.
    - Add 25mL of 0.1M $AgNO_3$.
    - Add 10mL of HNO_3.
    - Boil the contents of the flask.
    - Cool the contents of the flask and add 50mL of H_2O.
    - Titrate excess $AgNO_3$ with 0.1M KSCN, using 2mL saturated solution of ferric alum as indicator to a reddish-brown end-point.

3. Blank.
    - Add all the reagents as above.

4. Determination of the salt content.
    - Calculate Cl found to determine the NaCl in cheese using the following equation.

$$\% \text{ Salt} = \frac{(B - S)\,\text{ml} \times 0.1 \times 0.0585 \times 100}{\text{Weight of food sample}}$$

Where B is the blank titre

S is the sample titre

Lipase activity

Equipment:

Weighing Scale (Mettler AE 260, Deltarange)

Babcock flasks (bottle, cream test, size: 50% 9G., in ½%, Kimble productions USA)

Water bath (Naake W19 Fisons, Type: KA/TA)

5mL Burette

10mL Pipettes

250mL Conical glass flasks

Centrifuge (Funke Gerber, Super Vario-N, 1998)

Solutions:

Triton™ X100 (iso-octylphenoxypolyethoxyethanol) (BDH chemicals, 98-100% purity)

Calgon (Sodium hexametaphosphate) (BDH chemicals, flake, Laboratory reagent)

Methanol (Biolab, Analytical grade (Labserv))

0.02 N Potassium hydroxide made up with methanol instead of water (Scientific supplies Ltd, pellets, Analytical reagent grade)

5% Phenolphthalein (indicator)

Hexane (Merck, n-Hexane for liquid chromatography, 98%)

1-Propanol (BDH chemicals, Analytical reagent grade, 99.5%)

2% Sodium citrate solution (Pfizer, Food grade)

Method:

- Make up BDI (Bureau of Dairy Industries) reagent by weighing 30g of Triton X100 and 70g of Calgon made up to 1 litre with water.
- Weigh 5g of cheese, add 37.5mL of 2% sodium citrate solution, heat to 50 - 60ºC to emulsify the cheese.
- Place 35mL of the cheese milk into the babcock flask, add 10mL of BDI reagent and mix well.
- Heat the flask in a gently boiling water bath for 15 – 20 minutes, shake well after 5 and 10 minutes.
- Centrifuge the flask for 1 minute at 600 rpm.

- Add methanol - water (1:1) solution to the flask to bring the fat level into the neck of the flask.
- Centrifuge further for 1 minute at 600 rpm.
- Carefully remove around 0.2 – 0.4g of the fat from the neck and place into pre-weighed conical flask.
- Record the fat weight placed into the conical flask.
- Make solvent solution made of hexane:1-Propanol (4:1), add 5mL of the solvent solution to the conical flask with the extracted fat.
- Add 5 drops of phenolphthalein indicator to the flask.
- Titrate the fat solution with 0.02N methanolic potassium hydroxide solution until the first definite colour change is observed.
- A blank titration is required to obtain the background titration.
- The acid degree value (ADV) is calculated using the following equation.

$$\text{ADV} = \frac{S \times N \times 10^2}{\text{Weight of fat}}$$

Where S is the net titration volume of sample

N is the normality of KOH

Texture

Equipment:

Wire cutter

Stable Micro systems texture analyzer (model: TA.XT.Plus)

Method:

- Cut cheese in to 20cm cubes, ensure that the surface is smooth.
- Open the texture analyzer and set the load to 50 kg.
- Screw in the 50mm diameter plunger and set the distance from the base to the plunger appropriately to ensure that the cheese blocks can be placed with ease on the base.
- Set the base as zero distance from the plunger.
- Set the compression distance to be 70% of the cheese block.
- Set the units on the graph to force in Newtons on the y-axis and distance in mm on the x-axis.
- Place the cheese block on the circle in the centre of the base and Start compression to generate a compression curve.
- Remove the crushed cheese sample and clean the base with a soft tissue.
- Label the compression curve and save in a file.

Screening Test

Name _____ Date _____

Instructions
Please rinse your mouth between the samples with the water provided.
Taste each solution and match the provided descriptors with the solutions.

Descriptors: Salty, Sweet, Bitter, Sour.

Code	**Descriptor**
079	_____
318	_____
992	_____
467	_____

Instructions
Please rinse your mouth between the samples with the water provided.
Taste the salty taste solutions provided in the coded cups in ascending order of saltiness.

	Code
Least salty	_____
	_____
	_____
Most salty	_____

Instructions

Please rinse your mouth between the samples with the water provided.
Taste the sweet taste solutions provided in the coded cups in ascending order of saltiness.

	Code
Least sweet	_____
	_____
	_____
Most sweet	_____

Instructions

Please rinse your mouth between the samples with the water provided.
Taste the bitter taste solutions provided in the coded cups in ascending order of saltiness.

	Code
Least bitter	_____
	_____
	_____
Most bitter	_____

Instructions

Please rinse your mouth between the samples with the water provided.
Taste the sour taste solutions provided in the coded cups in ascending order of saltiness.

	Code
Least sour	_____
	_____
	_____
Most sour	_____

DIFFERENCE FROM CONTROL

Name _____ Date _____

Taste the REFERENCE sample before tasting samples.
Please rinse your mouth between the samples with the water provided.
Taste the samples, from left to right, and rate them against the REFERENCE using the scale 1-7:

1	2	3	4	5	6	7
	Same as REFERENCE	Moderately different from REFERENCE			Extremely different from REFERENCE	

Sample						
Score						

If the sample scored 1 or 2 (not different from REFERENCE) **STOP** here and proceed with tasting the next sample.
If the sample scored 3 or MORE (different from REFERENCE) describe the difference and the intensity of the difference below:

Sample	Much less than	Less than	Attribute	More than	Much more than

Attributes to choose from: Salty, Firm, Creamy, Bitter, Brittle, Colour, Acidic/Sour.

Appendix – D. Raw Data

The average protein content expressed as percentage in cheese made on hollow fibre at differential pressure of 0.3 bar, spiral wound at differential pressure of 0.3 and 1.0 bar and reference from PVC for all batches.

	Hollow fibre	Spiral 0.3 bar	Spiral 1.0 bar	Reference
Batch 1	15.1	15	14.9	14.3
Batch 2	14.2	15.6	14.6	13.7
Batch 3	14.5	15.2	15.7	14.3
Batch 4	14.3	14.3	14.9	13.8

Fat content as percentage in cheese made on hollow fibre at 0.3 bar differential pressure, spiral wound with differential pressure of 0.3 and 1.0 bar and the reference from PVC for that batch.

	Hollow fibre	Spiral 0.3 bar	Spiral 1.0 bar	Reference
Batch 1	18.6	18.3	17.7	20.7
Batch 2	16.9	19.2	20.1	20.9
Batch 3	17.1	18.7	18.8	21.3
Batch 4	17.2	17.3	17.1	20.1

Total solids percentage in cheese made on hollow fibre at differential pressure 0.3 bar, spiral wound with differential pressure of 0.3 bar and 1.0bar and the corresponding reference for that batch.

	Hollow fibre	Spiral 0.3 bar	Spiral 1.0 bar	Reference
Batch 1	41.2	38.9	38.5	42.5
Batch 2	40	43	39.5	41.9
Batch 3	39.8	40.2	42	42.2
Batch 4	39.6	37.7	39.2	42

The salt percentage of cheese made on hollow fibre at differential pressure of 0.3 bar, spiral wound unit at differential pressure of 0.3 and 1.0 bar and the corresponding reference for all batches.

	Hollow fibre	Spiral 0.3 bar	Spiral 1.0 bar	Reference
Batch 1	3.1	2.3	2.8	3.8
Batch 2	2.6	3.1	2.3	4
Batch 3	2.9	2.9	3	3.9
Batch 4	2.7	2.8	2.8	3.6

The lipase activity after 3 months in all cheese made on hollow fibre at differential pressure of 0.3 bar, spiral wound unit at differential pressure of 0.3 and 1.0 bar and the reference from PVC. The Lipase activity is expressed as acid degree value (ADV).

	Hollow fibre	Spiral 0.3 bar	Spiral 1.0 bar	Reference
Batch 1	3.1	7.8	2.4	1.5
Batch 2	2	2.4	3.2	1.5
Batch 3	3.1	3.5	12.8	1.5
Batch 4	3	5	3.4	1.5

The lipase activity after 5 months in cheese made on hollow fibre at differential pressure of 0.3 bar, spiral wound unit with differential pressure of 0.3 and 1.0 bar and the reference for batch 1 and 2.

	Hollow fibre	Spiral 0.3 bar	Spiral 1.0 bar	Reference
Batch 1	3.2	11	3.9	2
Batch 2	3.5	3.7	6	2

The average degree of difference detected by the panel of judges between all batches of cheese made on the hollow fibre pilot plant and the reference from PVC over a storage period of 5 months.

	Degree of difference			
	Month 1	**Month 2**	**Month 3**	**Month 4**
Batch 1	4	3	3	3
Batch 2	3	3	3	3
Batch 3	3	3	3	-
Batch 4	3	3	3	-

The degree of difference distinguished between the all batches of cheese made on spiral wound unit at differential pressure of 0.3 bar and the reference from PVC over 5 months of shelf life.

	Degree of difference			
	Month 1	**Month 2**	**Month 3**	**Month 4**
Batch 1	3	3	3	4
Batch 2	3	3	3	3
Batch 3	4	3	3	-
Batch 4	3	3	3	-

The degree of difference determined by the panel of judges between all batches of cheese made on spiral wound unit operating at differential pressure of 1.0 bar and the reference over storage time of 5 months.

	Degree of difference			
	Month 1	**Month 2**	**Month 3**	**Month 4**
Batch 1	3	3	2	3
Batch 2	5	5	6	5
Batch 3	3	3	3	-
Batch 4	3	4	3	-

All total solids data from the pilot plant runs represented below is determined using the refractometer. The total solids from the refractometer are accurate to ±2% of the total solids determined using the air drying method.

Raw data of Total solids (%) as obtained from four duplicate runs using Hollow fibre pilot plant. Expt is the set of data obtained from a trial run conducted using the hollow fibre pilot plant.

Time	Expt	Dup 1	Dup 2	Dup 3	Dup 4	Average	Std dev	Std error
0	12	12	12	12	12	12	0	0
15	13	12	13	13	12	12.6	0.49	0.22
30	14	14	13	14	13	13.6	0.49	0.22
45	15	15	14.5	15	14	14.7	0.40	0.18
60	16	16	15	16	15	15.6	0.49	0.22
75	18	18	16	17	17	17.2	0.75	0.33
90	19	19	18	19	19	18.8	0.40	0.18
105	20.5	23	19	21	21	20.9	1.28	0.57
120	25.5	25.5	21.5	25	25	24.5	1.52	0.68
135	28.5	29	23	26		26.625	2.38	1.19
150	30	30.5	25.5	30		29	2.03	1.02
165	34	34.5	28.5	33		32.5	2.37	1.19
180	37	36.5	33.5	37.5		36.125	1.56	0.78
185			37			37	0	0

Raw data of Total solids (%) as obtained from four duplicate runs using Spiral wound pilot plant operated at differential pressure of 0.3bar.

Time	Dup 1	Dup 2	Dup 3	Dup 4	Average	Std dev	Std error
0	10.5	11	11	11	10.875	0.22	0.11
15	13.5	13	12	12	12.625	0.65	0.32
30	14	14	13	13	13.5	0.50	0.25
45	16	15	15	14	15	0.71	0.35
60	15	16	17		16	0.82	0.47
75	17	18.5	18	16	17.375	0.96	0.48
90	19	21.5	23	21	21.125	1.43	0.72
105	23	23	25	23	23.5	0.87	0.43
120	25	26.5	30	26	26.875	1.88	0.94
135	28.5	30.5	31	29	29.75	1.03	0.52
150	30	31.5	34	31	31.625	1.47	0.74
165	32	36	36	33	34.25	1.79	0.89
180	34			36	35	1.00	0.71
185	36				36	0	0

Raw data of Total solids (%) as obtained from four duplicate runs using Spiral wound pilot plant at differential pressure of 1.0bar. Expt is the set of data obtained from a trial run conducted using the Spiral wound pilot plant.

Time	Expt	Dup 1	Dup 2	Dup 3	Dup 4	Average	Std dev	Std error
0	12	12	12	12	13	12.2	0.40	0.18
15	13	14	14	13	14	13.6	0.49	0.22
30	14	14	15	15	16	14.8	0.75	0.33
45	15.5	15.5	17.5	17	18	16.7	1.03	0.46
60	16.5	21	20	20	21	19.7	1.66	0.74
75	21	27.5	25	27	25	25.1	2.29	1.02
90		31	30.5	32	28	30.375	1.47	0.74
105	30	36.5	34	36	32	33.7	2.44	1.09
120	36		36.5		37	36.5	0.41	0.24

Raw data of permeate flux in LMH as obtained from four duplicate runs using Hollow fibre pilot plant. Expt is the set of data obtained from a trial run conducted using the hollow fibre pilot plant.

Time	Expt	Dup 1	Dup 2	Dup 3	Dup 4	Average	Std dev	Std error
0	28.16	33.61	32.70	32.70	32.70	31.98	1.94	0.87
15	30.89	32.70	32.70	32.70	32.70	32.34	0.73	0.33
30	28.16	29.98	28.16	30.89	30.89	29.62	1.23	0.55
45	28.16	27.25	23.62	30.89	29.07	27.80	2.41	1.08
60	24.98	23.62	15.44	29.07	26.34	23.89	4.59	2.05
75	22.26	20.89	16.35	20.89	23.62	20.80	2.44	1.09
90	19.53	19.08	16.35	18.17	20.89	18.80	1.51	0.67
105	14.99	17.26	13.63	17.26	17.26	16.08	1.51	0.67
120	12.72	11.81	13.63	13.63	14.53	13.26	0.93	0.41
135	9.99	8.18	11.81	12.72		10.67	1.74	0.87
150	7.95		9.99	10.90	9.08	9.48	1.09	0.55
165			7.27	7.27	6.36	6.96	0.43	0.25

Raw data of permeate flux in LMH as obtained from four duplicate runs using Spiral wound pilot plant operated at differential pressure of 0.3bar.

Time	Dup 1	Dup 2	Dup 3	Dup 4	Average	Std dev	Std error
0	12.9	19.9	20.9	19.9	18.38	3.18	1.59
15	12.9	18.9	19.9	17.9	17.39	2.68	1.34
30	12.9	17.9	19.9	17.9	17.14	2.57	1.28
45	11.9	15.9	17.9	15.9	15.40	2.17	1.08
60	11.9	14.9	15.9	13.9	14.16	1.47	0.73
75	11.9	11.9	13.9	12.9	12.67	0.82	0.41
90	9.9	9.9	10.9	9.9	10.18	0.43	0.22
105	8.9	7.0	7.0	7.9	7.70	0.82	0.41
120	7.0	5.0	5.0	6.0	5.71	0.82	0.41
135	5.0	4.0		4.0	4.31	0.47	0.27
150	4.0	3.0	3.0	3.0	3.23	0.43	0.22
165	3.0	2.0	2.0	2.0	2.24	0.43	0.22

Raw data of permeate flux in LMH as obtained from four duplicate runs using Spiral wound pilot plant at differential pressure of 1.0bar. Expt is the set of data obtained from a trial run conducted using the Spiral wound pilot plant.

Time	Expt	Dup 1	Dup 2	Dup 3	Dup 4	Average	Std dev	Std error
0	21.9	10.9	25.8	21.9	21.9	22.85	1.72	0.86
15	18.9	9.9	25.8	21.9	21.9	22.11	2.47	1.24
30	17.9	10.9	23.8	19.9	19.9	20.37	2.17	1.08
45	16.9	10.9	20.9	17.9	17.9	18.38	1.49	0.75
60	14.9	10.9	16.9	14.9	15.9	15.65	0.82	0.41
75	12.9	7.0	11.9	8.9	12.3	11.51	1.52	0.76
90		5.0	6.0	5.0	7.9	6.29	1.24	0.72
105	7.9	3.0	3.0	3.0	4.0	4.47	2.05	1.02
120	3.0		3.0		2.0	2.65	0.47	0.27

Raw data of permeate flux in LMH as obtained from ten runs at PVC.

Time	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	Run 8	Run 9	Run 10	Average	Std dev	Std error
10	-	-	23.36	21.86	24.18	22.68	21.17	16.26	19.26	5.60	21.25	2.52	0.80
20	24.86	29.37	-	24.73	23.63	17.49	21.99	24.45	21.31	9.56	23.48	3.20	1.07
40	26.64	26.37	-	25.14	23.63	9.43	21.72	24.59	21.72	13.39	22.40	5.21	1.74
60	25.27	-	20.63	24.18	23.09	-	21.31	23.77	21.31	12.57	22.79	1.61	0.51
80	23.36	24.18	19.54	22.27	22.27	-	20.49	22.95	20.63	14.89	21.96	1.49	0.47
100	21.31	19.67	18.31	15.85	20.22	-	19.81	21.31	19.40	13.80	19.48	1.66	0.52
120	18.99	16.94	16.67	18.44	19.40	-	18.44	19.81	18.72	15.30	18.43	1.03	0.33
140	12.98	11.61	14.07	17.35	17.35	-	17.90	16.94	17.35	13.52	15.69	2.27	0.72
160	8.74	8.33	8.61	14.75	14.75	-	15.71	10.93	11.61	8.74	11.68	2.85	0.90
180	6.83	-	3.96	7.65	6.15	-	9.43	5.87	6.42	7.10	6.62	1.55	0.49
190	6.28	5.46	5.87	7.38	6.01	-	5.33	5.33	5.33	6.01	5.87	0.66	0.21
200	-	-	5.87	6.56	-	-	5.46	5.46	5.46	5.74	5.77	0.43	0.13

Raw data of Total solids (%) as obtained from ten runs at PVC.

Time	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	Run 8	Run 9	Run 10	Average	Std dev	Std error
0	13.80	12.63	13.51	13.36	13.99	14.28	12.97	12.65	13.86	13.26	13.43	0.53	0.17
10	16.46	15.72	14.96	-	14.38	19.20		14.11	14.71		15.65	1.63	0.52
20		17.34	-	16.90	16.68	27.78	16.56	15.49	15.62		18.05	4.02	1.34
40			-	19.73	20.46	34.96	17.54	17.67	17.95	20.61	21.27	5.72	1.91
60	21.72	21.45	22.26	21.42	20.86	-	18.88	19.60	19.48	19.08	20.53	1.20	0.38
80			20.98	24.40	22.97	-	21.05	21.42	21.68	19.91	21.77	1.37	0.43
100	24.79	26.60	22.69	30.67	25.42	-	22.27	23.20	22.93	20.15	24.30	2.87	0.91
120			25.82	26.91	26.60	-	24.78	24.66	24.20	21.58	24.94	1.66	0.53
140	28.77	28.78	28.58	29.34	29.45	-	24.99	28.49	27.60	23.05	27.67	2.07	0.65
160			33.66	32.30	33.78	-	29.92	32.82	32.90	26.14	31.65	2.54	0.80
180			34.69	35.80	37.44	-	33.88	36.44	36.35	30.97	35.08	2.00	0.63
190	37.90		36.67	36.84	37.68	-	37.26	36.70	36.60	35.83	36.94	0.62	0.20
200		-	37.29	37.52	-	-	37.50	37.60	37.50	36.94	37.39	0.22	0.07