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Influence of processing temperature on flux decline during skim milk ultrafiltration

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ABSTRACT

The flux decline behaviour during skim milk ultrafiltration (UF) was investigated at 10 °C, 30 °C and 50 °C. Despite higher fluxes, UF processing at higher temperatures resulted in higher magnitudes and rates of irreversible fouling. Fouling in this temperature range was primarily proteinaceous, consisting of mainly peptides and alpha-lactalbumin (α-LA), with minor amounts of beta-lactoglobulin (β-LG) present only at 50 °C. The increase in fouling resistance with processing temperature was concluded to be mainly due to increased pore fouling by α-LA deposition, and in part to β-LG deposition at 50 °C. These are attributed respectively to the thermal expansion of membrane pores and reversible conformational changes of β-LG.

1. Introduction

Skim milk UF is widely used in dairy processing as means of concentration and purification of milk proteins for the production of cheese [1–5], milk protein concentrates (MPC) [3–7], and for protein standardisation [3–6,8]. During filtration, additional permeation resistance is contributed by the inherent accumulation of retained particles at the membrane surface (concentration polarisation, or CP) and particle deposition on the membrane surface or inside membrane pores (fouling) [1,9]. As a result, filtration throughput is reduced and product quality is altered. In addition, chemical cleaning is necessary for removing fouling, incurring significant water and chemical consumption, and process downtime [10]. To further optimise filtration and cleaning, a deeper understanding of CP and fouling in skim milk UF is required, but to date these is not completely understood. In particular, the effect of processing temperature has yet to be fully investigated.

Industrial skim milk UF is typically conducted at either ~10 °C or ~50 °C, each with its advantages and drawbacks. Operation at the higher temperatures (~50 °C) is generally favoured due to higher fluxes resulting from lower permeate viscosity. However, the growth of thermophiles is also promoted [1,6]. Conversely, thermophile growth is hindered at ~10 °C, at the expense of lower fluxes due to higher permeate viscosity. Processing temperature also affects the physicochemical properties of skim milk, which can in turn influence filtration behaviour. For instance, the structure and composition of casein micelles (CMs) are influenced by hydrophobic interactions and calcium phosphate solubility, both of which are temperature-sensitive [2]. At lower temperatures, calcium phosphate solubility increases and hydrophobic interactions weaken, resulting in the solubilisation of micellar calcium phosphate and dissociation of β-casein [2,11,12]. Meanwhile, whey proteins are more prone to thermally-induced conformational changes at elevated temperatures [13]. Filtration behaviour has been observed to be affected by changes in milk physicochemical properties due to alterations in pH [14–18], ionic strength (via mineral addition) [17,19] and thermal pre-treatment [20]. However, despite the temperature-sensitive nature of milk and the common practice of skim milk UF at ~10 °C in some parts of the world, the vast majority of the skim milk UF fouling characterisation studies have been conducted at 50 °C, which is more widely used. There are very few studies pertaining to skim milk UF at low temperatures, limited to investigations on flux [21,22], protein rejection [22], compositional changes [23–25], and microbial growth [26]. To our knowledge, the fouling behaviour during skim milk UF at 10 °C has not been properly examined, nor has any comparison or validation with what is known for UF at 50 °C been made. This also raises the question of how much of the knowledge established for UF at 50 °C is applicable to UF at ~10 °C.

Membrane fouling in the dairy industry has generally been attributed to adsorption of proteins and precipitation of calcium phosphate [1,6]. Accordingly, chemical cleaning typically involves alkaline and acid cleaning for the removal of organic and mineral fouling respectively [27]. However, it has been shown that fouling in skim milk UF performed at 50 °C is predominantly caused by proteins, with minerals only accounting for about 0.4% of the foulant material [28–30]. More recent studies investigating the effectiveness of various cleaning chemicals for rejuvenating UF membranes fouled by skim milk (at 50 °C) have also demonstrated the ineffectiveness of acid cleaning in protein

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Nomenclature

Abbreviations

ATR-FTIR	attenuated total reflection Fourier transform infrared spectroscopy
BSA	bovine serum albumin
CFV	cross-flow velocity
CM	casein micelle
CN	casein
CP	concentration polarisation
ICP-OES	inductively coupled plasma optical emission spectrometry
LA	lactalbumin
LG	lactoglobulin
MPC	milk protein concentrate
MW	molecular weight
NF	nanofiltration
PES	polyethersulfone
PSf	polysulfone
SDS-PAGE	sodium dodecyl sulfate polyacrylamide gel electrophoresis
SEM-EDS	scanning electron microscopy-energy dispersive X-ray spectroscopy
TMP	transmembrane pressure
UF	ultrafiltration

removal [27,31–33]. The omission of acid cleaning has been suggested as this can potentially reduce chemical, water and chemical consumption as well as cleaning time, especially if the alkaline cleaning formulation is optimised [34]. The applicability of these findings to skim milk UF at 10 °C is subject to the composition of the fouling layer formed at 10 °C, but this is yet to be reported. It is not known whether changes in the equilibrium of calcium and casein between the micelles and serum affect the contribution of minerals or protein to the fouling.

In this study, the influence of processing temperature on flux decline behaviour during skim milk UF was investigated in detail using a custom-built filtration rig. Respective resistance contributions of CP and fouling were evaluated using the resistance-in-series approach [35]. The temperature-dependent fouling behaviour was studied, including a comprehensive analysis of the composition of the fouling layers. An explanation of the underlying mechanisms for the observed differences was also provided.

2. Materials and methods

2.1. Filtration feed fluids and water

Fresh pasteurised (72 °C/15 s) skim milk and whole milk were purchased from the local supermarket and stored at 6 °C. The skim milk contained 37 g/L protein and 1 g/L fat, while the whole milk contained 35 g/L protein and 34 g/L fat, as per manufacturer specifications. Double distilled water (ddH_2O) and RO water were used for solution preparation and membrane rinsing respectively.

2.2. Membranes

10 kDa polyethersulfone (PES) flat sheet membranes were used in this study. The membrane sheets were cut from a Koch HFK-131 spiral wound module (Koch Membrane Systems, Massachusetts, USA) and stored in 1% Ultrasil 73 (Ecolab, NSW, Australia) at 6 °C to prevent bacterial contamination. Feed and permeate spacers used were also cut to size from the same membrane module.

2.3. Cross-flow filtration rig

Filtration experiments were carried out on a custom built filtration rig (Fig. 1). The filtration cell consisted of feed and permeate plates constructed out of grade 316 stainless steel, with a 0.046 in. feed spacer and permeate carrier inserted along the feed and permeate channels respectively. The filtration surface dimensions were 0.21 m × 0.060 m, giving a filtration area of 0.0126 m^2. The assembled cell plates were secured with stainless steel screws.

A gear pump (Micropump, USA) was used to deliver the feed solution to the filtration cell through a set of cooling coils immersed in a water bath. The water bath temperature was controlled by a thermoregulator connected to a chiller. Filtration was conducted under constant pressure, and the desired flow rate and pressure were obtained by adjusting the backpressure valve (Swagelok, Victoria, Australia) and pump speed. Permeate was collected in a beaker placed on a mass balance (Mettler Toledo, Switzerland) to measure the mass of permeate collected at regular time intervals. The mass balance and variable speed drive were connected to a computer via an I/O interface (National Instruments, USA). NI Labview was used to adjust the pump speed and to record permeate mass every 30 s.

Pressure gauges (Swagelok, Victoria, Australia) and digital thermometers (Comark Instruments, USA) were placed in-line near the feed-side inlet and outlet of the filtration cell to monitor the feed and retentate streams respectively. 3/8 in. high-pressure nylon tubing and

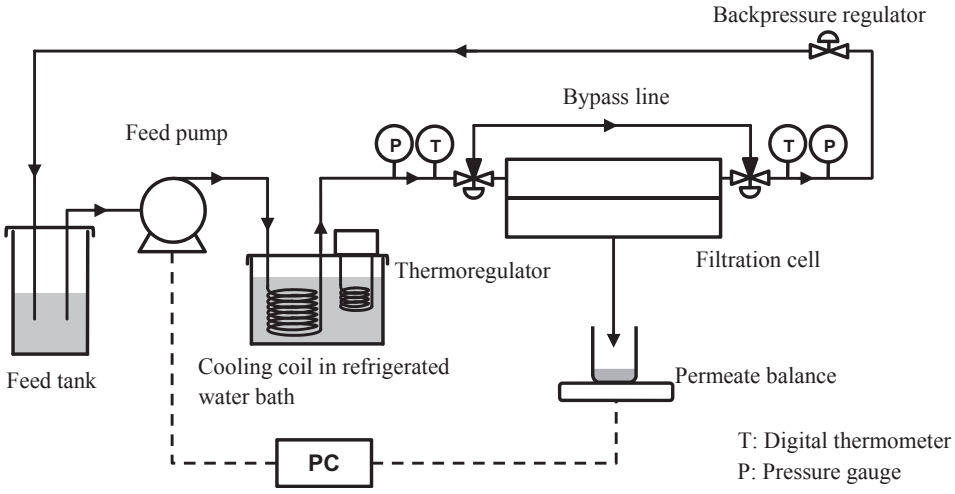

Fig. 1. Schematic of the cross-flow filtration rig.