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## Efficiency of serum protein removal from skim milk with ceramic and polymeric membranes at 50°C1

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#### **ABSTRACT**

Raw milk (2,710 kg) was separated at 4°C, the skim milk was pasteurized (72°C, 16 s), split into 3 batches, and microfiltered using pilot-scale ceramic uniform transmembrane pressure (UTP; Membralox model EP1940GL0.1μA, 0.1 μm alumina, Pall Corp., East Hills, NY), ceramic graded permeability (GP; Membralox model EP1940GL0.1μAGP1020, 0.1 μm alumina, Pall Corp.), and polymeric spiral-wound (SW; model FG7838-OS0x-S, 0.3 μm polyvinylidene fluoride, Parker-Hannifin, Process Advanced Filtration Division, Tell City, IN) membranes. There were differences in flux among ceramic UTP, ceramic GP, and polymeric SW microfiltration membranes (54.08, 71.79, and 16.21 kg/m<sup>2</sup> per hour, respectively) when processing skim milk at 50°C in a continuous bleed-and-feed 3× process. These differences in flux among the membranes would influence the amount of membrane surface area required to process a given volume of milk in a given time. Further work is needed to determine if these differences in flux are maintained over longer processing times. The true protein contents of the microfiltration permeates from UTP and GP membranes were higher than from SW membranes (0.57, 0.56, and 0.38%, respectively). Sodium-dodecyl-sulfate-PAGE gels for permeates revealed a higher casein proportion in GP and SW permeate than in UTP permeate, with the highest passage of casein through the GP membrane under the operational conditions used in this study. The slight cloudiness of the permeates produced using the GP and SW systems may have been due to the presence of a small amount of casein, which may present an obstacle in their use in applications when clarity is an important functional characteristic. More β-lactoglobulin passed through the ceramic membranes than through the polymeric membrane. The efficiency of removal of serum proteins in a continuous bleed-and-feed 3× process at  $50^{\circ}$ C was 64.40% for UTP, 61.04% for GP, and 38.62%for SW microfiltration membranes. The SW polymeric membranes had a much higher rejection of serum proteins than did the ceramic membranes, consistent with the sodium-dodecyl-sulfate PAGE data. Multiple stages and diafiltration would be required to produce a 60 to 65% serum protein reduced micellar casein concentrate with SW membranes, whereas only one stage would be needed for the ceramic membranes used in this study. tein fractionation

**Key words:** microfiltration, flux, serum protein, pro-

#### INTRODUCTION

Microfiltration (MF) is a pressure-driven separation process using membranes with a pore-size diameter ranging from 20 to 0.1  $\mu$ m (Maubois, 2002). The 2 most common applications of MF in the dairy industry (Saboya and Maubois, 2000) are removal of bacteria and spores from skim milk and separation of micellar case from serum proteins (SP). Separation of micellar casein from SP in skim milk relies on the difference in size between casein micelles  $(0.2 \mu m)$  and SP (0.0036μm) when using ceramic membranes with a pore-size diameter around 0.1 µm. The separation of casein and SP by 0.1-µm ceramic membranes is accomplished by a combination of the selectivity of the membrane (Brans et al., 2004) and the lift on particles in the feed stream created by very high shear turbulent flow (Altmann and Ripperger, 1997).

Separation of casein and SP with spiral-wound polymeric membranes depends more on the combination of properties of the membrane and the fouling layer (Riedl et al., 1998). Microfiltration produces a permeate with a composition similar to that of sweet whey with the main components being β-LG, α-LA, lactose, minerals, and NPN. Microfiltration permeate is clear, sterile, free of phage, and is an excellent starting material for production of SP concentrates or isolates (Saboya and Maubois, 2000). The casein obtained by MF of skim milk shows many interesting properties especially for

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<sup>&</sup>lt;sup>1</sup>Use of names, names of ingredients, and identification of specific models of equipment is for scientific clarity and does not constitute any endorsement of product by authors, University of Warmia and Mazury, Cornell University, or the Northeast Dairy Foods Research

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the cheese industry (Caron et al., 1997; Saint-Gelais et al., 1998; Neocleous et al., 2002a, b).

Most ultrafiltration in the dairy industry uses polymeric membranes, which cost less than ceramic membranes and require less energy but have relatively low chemical stability (Jost and Jelen, 1997) and a shorter life compared with ceramic membranes (Cheryan, 1998). Ceramic membranes are very resistant to extreme chemical and physical conditions and can withstand a pH range from 0.5 to 13.5 and temperatures >100°C (Baruah et al., 2006). However, there is a need to change temperature gradually (<10°C per min) with ceramic membranes to avoid cracking of the membrane.

Brans et al. (2004) has reviewed the mechanism of fouling of MF membranes. To overcome a rapid fouling of MF membranes when processing skim milk caused by large transmembrane pressure, Sandblom (1978) developed a uniform transmembrane pressure (UTP) system in which the MF permeate circulates in a loop parallel to the direction of the retentate flow. The specific conditions (flow) are chosen to ensure low and uniform transmembrane pressure from the retentate inlet to outlet end of the membrane. In the method patented by Alfa Laval, the permeate side of the membrane is filled with polymeric beads (Holm et al., 1990), which aids in control of transmembrane pressure to reduce fouling. A ceramic UTP MF system consists of a feed pump and 2 recirculation pumps, 1 for retentate and 1 for permeate. Recent attempts to achieve the benefits of UTP in alternative ways and reduce electrical operating cost have resulted in the development of graded permeability (GP) ceramic membranes (e.g., Membralox GP, Pall Corporation, East Hills, NY). The GP membranes have the uniform thickness and porosity of the selective membrane layer and a difference in permeability built into the ceramic support structure, with the support structure providing more resistance to permeate flow at the retentate inlet end of the membrane and less resistance to flow at the retentate outlet end of the membrane (Garcera and Toujas, 2002). In addition, restriction of the permeate removal rate controls pressure and the rate of permeate flow throughout the length of a GP membrane. The GP approach claims to maintain a constant and uniform flux along all parts of the membrane, while eliminating the need for a permeate recirculation pump and the associated electrical costs. Another approach used to overcome the higher operational cost of the permeate recirculation loop of UTP systems while controlling fouling uses a continuous gradient in the thickness of MF membrane selective layer along the membrane length (Saboya and Maubois, 2000) and a uniform porosity of the ceramic support structure. The commercial name of the system patented by Grangeon et al. (2002) is "Isoflux" and it

is manufactured by TAMI Industries (Nyones, France). Ceramic membrane systems are typically in multichannel configuration with either cylindrical channels or various cross-section forms (Saboya and Maubois, 2000) to increase amount of the filtration area per module.

Recently, spiral-wound polymeric MF membranes have become available and provide a large amount of membrane surface area per module at a lower cost per square meter than ceramic membranes, but with a more frequent need for replacement. Zeman (1996) listed 17 polymers commonly used in commercial MF and UF applications. The physical and chemical conditions used during processing, as well as the characteristics of the feed material, determine to a large extent the separation properties of polymeric membranes. Approaches used to minimize fouling of polymeric spiral-wound (SW) membranes include the use of polymeric spacer materials of various thicknesses in the retentate flow path to create turbulent flow. Spiral-wound membranes use much lower retentate side pressures than ceramic membranes to avoid compaction of the membrane and to keep transmembrane pressure low. In contrast to the use of back pressure on the permeate side for operation of ceramic membranes, back pressure from the permeate side in a SW polymeric membrane system cannot be used because it will damage the membrane. The polymeric membrane formation technology and types of polymer blends must produce a membrane with reproducible quality and performance (Zeman, 1996).

The type of MF membrane used for processing of skim milk may affect the efficiency of transmission of SP, protein composition of the retentate and permeate, the level of residual casein in the permeate, and the cost of the process. Differences in composition may influence the functional and sensory properties of MF retentate and permeate products. The differences in efficiency of SP removal (kg of SP removed/m<sup>2</sup> per hour) will also influence the amount of membrane surface area required to accomplish the process and to obtain retentate and permeate with the desired composition. A higher rejection of SP by an MF membrane will require additional diafiltration and membrane surface area to achieve a given percentage of SP removal and will increase processing cost. The only data available in the literature for efficiency of removal of SP from skim milk is for a UTP ceramic membrane system (Nelson and Barbano, 2005), which shows that in a 3-stage continuous bleed-and-feed process with a 3×concentration factor, it was possible to remove  $95 \pm 1.1\%$  of SP from skim milk at 50°C. No data are available in the literature on the efficiency of removal of SP from skim milk by using polymeric membranes or graded permeability membranes. The objective of our research was to compare the flux and SP removal for 3 different MF

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