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A process efficiency assessment of serum protein removal from milk using ceramic graded permeability microfiltration membrane

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ABSTRACT

Microfiltration (MF) is a well-known process that can be used in the dairy industry to separate caseins from serum proteins (SP) in skim milk using membranes with a pore diameter of 0.1 μm . Graded permeability ceramic membranes have been studied widely as means of improving milk fractionation by overcoming problems encountered with other MF membranes. The ideal operating parameters for process efficiency in terms of membrane selectivity, permeate flux, casein loss, SP transmission, energy consumption, and dilution with water remain to be determined for this membrane. Our objective was to evaluate the effects of transmembrane pressure (TMP), volumetric concentration factor (VCF), and diafiltration on overall process efficiency. Skim milk was processed using a pilot-scale MF system equipped with 0.72-m² graded permeability membranes with a pore size of 0.1 μm . In the first experiment, in full recycle mode, TMP was set at 124, 152, 179, or 207 kPa by adjusting the permeate pressure at the outlet. Whereas TMP had no significant effect on permeate and retentate composition, 152 kPa was found to be optimal for SP removal during concentration and concentration or diafiltration experiments. When VCF was increased to 3 \times , SP rejection coefficient increased along with energy consumption and total casein loss, whereas SP removal rate decreased. Diafiltering twice allowed an increase in total SP removal but resulted in a substantial increase in energy consumption and casein loss. It also reduced the SP removal rate by diluting permeate. The membrane surface area required for producing cheese milk by blending whole milk, cream, and MF retentate (at different VCF) was estimated for different cheese milk casein concentrations. For a given casein concentration, the same quantity of permeate and SP would be produced, but less membrane surface area would be needed at a lower retentate VCF. Mi-

crofiltration has great potential as a process of adding value to conventional cheesemaking processes, but its cost-effectiveness at a large scale remains to be demonstrated.

Key words: microfiltration, graded permeability membrane, process efficiency, energy consumption, mass balance

INTRODUCTION

Microfiltration (MF) is a pressure-driven, membrane-based process used to concentrate particles in the 0.1 to 10 μm size range and to separate them from the suspending liquid (Saboya and Maubois, 2000). The first major application of MF in the dairy industry was for the removal of bacteria from milk using a membrane having a pore size of 1.4 μm (Trouvé et al., 1991). A pore size of 0.1 μm allows the separation of casein micelles from SP of skim milk (Saboya and Maubois, 2000). The casein-enriched retentate thus obtained can be used to standardize cheese milk, whereas the clear, fat-free, and sterile serum protein (SP)-enriched permeate has a composition similar to cheese whey. Serum proteins contained in the MF permeate are functionally and nutritionally superior to SP from cheese whey, which gives it an excellent potential for production of SP concentrates or isolates (Saboya and Maubois, 2000). This process also allows the production of micellar casein concentrate (MCC), a novel dairy ingredient obtained by diafiltering the casein-enriched retentate. This high-protein ingredient can be used in the formulation of different products due to its nutritive and functional value, as well as its stability during processing.

Ceramic is a popular choice of membrane material for MF applications due to its resistance to back-pressure and steam sterilization, its narrow pore size distribution, and the high permeate flux obtainable (Zulewska et al., 2009; Fernández García and Riera Rodríguez, 2015). First-generation ceramic membranes fouled quickly, decreasing selectivity, permeate flux, and overall performance (Saboya and Maubois, 2000). To overcome this problem, Sandblom (1978) developed

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the uniform transmembrane pressure (**UTP**) system, whereas Alfa Laval (Kolding, Denmark) improved and patented this process a few years later (Holm et al., 1990). In this system, MF permeate is recirculated in a loop parallel to the retentate flow, which allows a lower and more UTP from the retentate inlet to the outlet and reduces membrane fouling. Although the UTP system performed well, it was not adopted widely, due mainly to high operating costs associated with the permeate recirculation pump and the difficulty of adjusting pressure drops on the permeate and retentate sides (Hu and Dickson, 2015). In recent years, several attempts were made to achieve the benefits of UTP system, but without the permeate recirculation pump (Saboya and Maubois, 2000). One promising alternative to UTP systems is the graded permeability (**GP**) ceramic membrane (Membralox GP, Pall Corp., East Hills, NY). With GP membranes, uniform permeate flux is obtained by incorporating a variation in the porosity of the membrane support along the length of the filtration element (Garcera and Toujas, 2002). This allows MF with less energy expenditure and simpler operation than with UTP systems (Hu and Dickson, 2015).

A study comparing UTP and GP systems for separating SP from skim milk has shown that UTP allows greater passage of SP, whereas permeate flux is greater through the GP membrane (Zulewska et al., 2009). Spiral-wound polymeric membrane (0.3 μm pore size) and Isoflux (TAMI, Nyons, France) ceramic membrane have been found less effective than UTP or GP systems (Zulewska et al., 2009; Adams and Barbano, 2013). Previous work by Zulewska and Barbano (2014) evaluated the effect of diafiltration (**DF**) on the efficiency of SP removal during MF of skim milk. Most studies of the performance of GP membranes have been conducted under feed-and-bleed conditions, which allow continuous production of retentate with a volumetric concentration factor (**VCF**) of $3\times$ by adjusting permeate and retentate removal rates during the process. Whereas this process is used widely by dairy processors, it is prone to fluctuating operating pressures and Starling flow (permeate backflow into the retentate), which increases membrane fouling and reduces the effective filtration surface area (Zulewska et al., 2009).

Efficiency can be defined as the link between results obtained and the amount of resources used. For a dairy processor, the ideal MF process would yield retentate sufficiently enriched in casein to be useful for standardizing cheese milk, plus permeate containing high-value SP and as little casein as possible would require low energy input and minimal dilution with water and would have a high permeate flux (Noble and Agrawal, 2005). To maximize process efficiency, MF should be

performed at a transmembrane pressure (**TMP**) at or below the critical flux defined as the mean flux at which a fouling layer begins to accumulate on the membrane (Bacchin, 2004). Raising the TMP above the critical flux increases the thickness of the fouling layer and decreases SP transmission (Gésan-Guiziou et al., 1999). Zulewska and Barbano (2014) determined that the critical flux of skim milk in a continuous feed-and-bleed process with a VCF of $3\times$ using a 0.1- μm GP membrane was 64 L/m² per hour. Under the same conditions, the maximal flux attainable (limiting flux) was estimated at 96 L/m² per hour. Sustainable flux is defined as the flux that can be maintained for a long time with minimal fouling, and is usually in between the critical and limiting fluxes (Hurt et al., 2015a). It was determined that this flux can be achieved at a TMP of 150 kPa regardless of the target protein concentration or membrane channel diameter. During MF of skim milk at a VCF of $3\times$, and using a 4-mm channel diameter GP membrane, this flux corresponds to 80 to 85% of the limiting flux (77–82 L/m² per hour; Hurt et al., 2015a). Other parameters, such as pressure drop, cross-flow velocity, operating temperature, membrane properties, type of pasteurization, feed concentration, and DF, have an influence on the efficiency of skim milk MF (Bacchin et al., 2006; Hu and Dickson, 2015).

Whereas the effects of channel diameter (Hurt et al., 2015a) and retentate protein concentration (Hurt et al., 2015b) on the limiting flux and the SP removal using GP membrane have been studied, no study has focused on attempts to perform batch filtrations at constant operating pressures. This would provide the truest assessment of GP membrane performance at different VCF and TMP. To the best of our knowledge, little attention has been devoted to processing efficiency in terms of overall mass balance under different conditions and, more importantly, to the in situ energy requirements of those conditions. The goal of our study was to characterize the performance of a 0.1- μm pore size GP membrane at different TMP and different VCF and to evaluate the effect of DF on the separation of SP from skim milk and on overall process efficiency.

MATERIALS AND METHODS

Skim Milk Preparation

Whole raw bovine milk was purchased from the bulk of a local dairy. Raw milk was heated to 45°C in a heat exchanger then separated using a centrifugal cream separator (model MP1254, Westfalia, Oelde, Germany). The cream was discarded and the skim milk (<0.05% fat) was HTST-pasteurized in a heat exchanger (APV, Goldsboro, NC) at 74°C for 16 s. The pasteurized skim

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