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Effect of high-hydrostatic-pressure-treated skim milk on permeate flux and fouling during ultrafiltration

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ABSTRACT

Ultrafiltration (UF) is largely used in the dairy industry to generate milk and whey protein concentrate for standardization of milk or production of dairy ingredients. Recently, it was demonstrated that high hydrostatic pressure (HHP) extended the shelf life of milk and improved rennet coagulation and cheese yield. Pressurization also modified casein micelle size distribution and promoted aggregation of whey proteins. These changes are likely to affect UF performance. Consequently, this study will determine the effect of skim milk pressurization (300 and 600 MPa, 5 min) on UF performance in terms of permeate flux decline and fouling. The effect of HHP on milk proteins was first studied and UF was performed in total recycle mode at different transmembrane pressures to determine optimal UF operational parameters and to evaluate the effect of pressurization on critical and limiting fluxes. Ultrafiltration was also performed in concentration mode at a transmembrane pressure of 345 kPa for 130 or 140 min to evaluate the decline of permeate flux and to determine fouling resistances. It was observed that average casein micelle size decreased by 32 and 38%, whereas β-lactoglobulin denaturation reached 30 and 70% at 300 and 600 MPa, respectively. These results were directly related to UF performance because initial permeate fluxes in total recycle mode decreased by 25% at 300 and 600 MPa compared with nonpressurized milk, critical flux, and limiting flux, which were lower during UF of milk treated with HHP. During UF in concentration mode, initial permeate fluxes were 30% lower at 300 and 600 MPa compared with the control, but the total flux decline was higher for nonpressurized milk (62%) compared with pressure-treated milk (30%). Fouling resistances were similar, whatever the treatment, except at 600 MPa where irreversible fouling was higher. Characterization of the fouling layer showed that caseins and β-lactoglobulin were mainly involved in membrane fouling after UF of pressure-treated milk. Our results demonstrate that HHP treatment of skim milk drastically decreased UF performance.

Key words: ultrafiltration, high hydrostatic pressure, permeate flux decline, membrane resistance, protein fouling

INTRODUCTION

In the dairy industry, UF is widely used to concentrate caseins and whey proteins to produce milk protein concentrate (Marcelo and Rizvi, 2008; Pouliot, 2008; Mohammad et al., 2012; Meyer et al., 2015). Milk protein concentrates are used in the formulation of a wide range of food products due to their nutritional value and their functional properties, such as foaming, emulsifying, solubilizing, and gelling. At this time, the main application of milk protein concentrate is to standardize milk to the desired casein to fat ratio for cheese production (Huffman and Harper, 1999; Gesan-Guiziou, 2012).

As largely reviewed for all pressure-driven membrane processes, the main drawback in using UF for skim milk concentration is the decline in permeate flux due to concentration polarization and membrane fouling. Concentration polarization is defined as the accumulation of particles at a membrane surface, whereas fouling results from protein adsorption and protein-protein interactions on the membrane surface (formation of a deposit layer) and in membrane pores (complete or partial pore plugging). Consequently, concentration polarization and fouling can drastically reduce permeate flux and membrane selectivity, increase energy consumption, therefore negatively affecting UF performance and efficiency (Chen et al., 1997; Bacchin et al., 2006; Shi et al., 2014).

Previous studies evaluated fouling species during milk UF as a function of the initial matrix (whole/skim milk, raw/pasteurized/reconstituted) and filtration operating parameters [hydrodynamic parameters, membrane material, molecular weight cut-off (**MWCO**),

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and so on]. Proteins are the major membrane foulants during UF of whole or skim milk (pasteurized or not) on polyethersulfone (5 and 10 kDa; Bégoin et al., 2006) or on ceramic membranes (zirconia material, MWCO of 10 and 150 kDa; Daufin et al., 1991). However, it is still unclear whether whey proteins, especially α-LA or casein micelles, represent the major protein foulant of membranes during UF of milk. Indeed, Tong et al. (1988) showed that fouling of polysulfone membrane (MWCO of 10 kDa) was mainly composed of whey proteins, especially α-LA. Moreover, after UF (M5 Carbosep type, 5–10 kDa) of UHT and reconstituted skim milk, Rabiller-Baudry et al. (2005) demonstrated that the deposit layer was mainly composed of casein micelles and its permeability was governed by electrostatic interactions. Consequently, and as a function of filtration conditions, casein and whey proteins must be characterized as fouling species after skim milk UF.

Recently, high hydrostatic pressure (**HHP**) was studied as an emerging technology and alternative treatment for skim milk pasteurization. Pressure-treated milk was demonstrated to be comparable to pasteurized milk (72°C, 15 s) in terms of microbial quality (Trujillo et al., 2002, 2016). More specifically, isostatic pressures of 400 MPa for 15 min or 600 MPa for 3 min at 20°C extended the milk shelf-life for 10 d (Rademacher and Kessler, 1997; Chawla et al., 2011). Although it has been established that HHP treatment had little effect on flavor compounds and the integrity of vitamins and other nutrients, the technology induced drastic modifications of milk color and turbidity as well as colloidal and soluble protein fractions (Balasubramaniam et al., 2016). Indeed, and depending on HHP parameters (pressure, time, and temperature), pressure treatment can have a major effect on casein micelle size distribution. For example, a treatment at 250 MPa for 15 min increased micelle casein size due to casein aggregation, whereas a treatment at pressures greater than 300 MPa irreversibly reduced the micelle size to approximately 50% of its initial diameter (Huppertz et al., 2002, 2006a,b; Voigt et al., 2015). The loss of micellar integrity observed after the pressure treatment was explained by solubilization of calcium phosphate, and consequently, disruption of casein micelles (Huppertz et al., 2002, 2004; Huppertz and de Kruif, 2007). Denaturation of the whey protein, β-LG was observed from 100 MPa and increased with pressure to reach 90% at 400 MPa. However, pressurization above 400 MPa had only a minor effect on β-LG denaturation (Considine et al., 2007a; Balasubramaniam et al., 2016). Contrary to β-LG, α-LA in whey is more resistant to pressurization because α-LA is denatured from 400 MPa with a maximal denaturation rate of 70% after 30 min at 800 MPa (Trujillo et al., 2002; López-Fandiño, 2006; Naik et al., 2013). The higher stability of α-LA is probably due to its higher number of intra-molecular disulfide bonds and the absence of a free sulfhydryl group in its structure (Huppertz et al., 2006a). The denaturation of whey proteins combined with disruption of casein micelles generates specific milk protein interactions under isostatic pressure. It was reported that β-LG interacted with other β-LG and α-LA through SH/S–S exchange reactions, which induced the formation of whey protein aggregates. It was also observed that β-LG can also form specific complexes with κ-CN due to their free thiol groups (Kolakowski et al., 2001; Considine et al., 2007a,b).

Consequently, considering the protein structure modifications observed on pressurization, it is conceivable that HHP-treated milks will exhibit different UF performance in terms of permeate flux decline and fouling mechanisms. The study of HPP-treated milks provides an unprecedented opportunity to better understand the contribution of casein micelles and whey proteins to membrane fouling. Thus, the aim of this study was (1) to evaluate the effect of HHP on casein micelle size distribution and whey protein aggregation in skim milk; (2) to study the effect of pressure-treated skim milk (300 and 600 MPa for 5 min) on permeate flux decline during UF in total-recirculation and concentration modes; and (3) to compare the composition of the membrane fouling layer after pressure-treated and control skim milk concentration by UF.

MATERIALS AND METHODS

Milk Supply

Pasteurized (HTST, 72°C for 16 s) skim milk was obtained from a local distributor (Québon, Agropur Natrel Division, Longueuil, QC, Canada) and was stored at 4°C until used in pressurization experiments.

High Hydrostatic Pressure Treatment

High hydrostatic pressure treatments of skim milk were performed at 300 and 600 MPa for 5 min at room temperature in a discontinuous hydrostatic pressurization unit Hiperbaric 135 system (Hiperbaric, Burgos, Spain) with water as the pressure transmission medium. The stainless-steel pressure vessel measured 0.30 m in diameter and 2.20 m in length with a working volume of 135 L. A nonpressurized skim milk was used as a control. Pressurization at 300 and 600 MPa was specifically chosen to observe the reversible and the irreversible effects of HHP on skim milk proteins. Indeed,