J. Dairy Sci. 99:1–16
http://dx.doi.org/10.3168/jds.2016-11090

Optimization of protein fractionation by skim milk microfiltration: Choice of ceramic membrane pore size and filtration temperature

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ABSTRACT

The objective of this study was to investigate how ceramic membrane pore size and filtration temperature influence the protein fractionation of skim milk by cross flow microfiltration (MF). Microfiltration was performed at a uniform transmembrane pressure with constant permeate flux to a volume concentration factor of 2.5. Three different membrane pore sizes, 0.05, 0.10, and 0.20 μm, were used at a filtration temperature of 50°C. Furthermore, at pore size 0.10 μm, 2 different filtration temperatures were investigated: 50 and 60°C. The transmission of proteins increased with increasing pore size, giving the permeate from MF with the 0.20-μm membrane a significantly higher concentration of native whey proteins compared with the permeates from the 0.05- and 0.10-μm membranes (0.50, 0.24, and 0.39%, respectively). Significant amounts of caseins permeated the 0.20-μm membrane (1.4%), giving a permeate with a whitish appearance and a casein distribution (α_{S2}-CN: α_{S1}-CN: κ-CN: β-CN) similar to that of skim milk. The 0.05- and 0.10-μm membranes were able to retain all caseins (only negligible amounts were detected). A permeate free from casein is beneficial in the production of native whey protein concentrates and in applications where transparency is an important functional characteristic. Microfiltration of skim milk at 50°C with the 0.10-μm membrane resulted in a permeate containing significantly more native whey proteins than the permeate from MF at 60°C. The more rapid increase in transmembrane pressure and the significantly lower concentration of caseins in the retentate at 60°C indicated that a higher concentration of caseins deposited on the membrane, and consequently reduced the native whey protein transmission. Optimal protein fractionation of skim milk into a casein-rich retentate and a permeate with native whey proteins were obtained by 0.10-μm MF at 50°C.

Key words: protein fractionation, ceramic membrane pore size, filtration temperature, uniform transmembrane pressure, constant flux

INTRODUCTION

The main proteins in milk, the caseins and whey proteins, differ in their functional and nutritional characteristics, and it is of interest to the dairy industry to separate these proteins. The caseins can be used to produce cheese, and high protein beverages and fermented milks. Whey proteins derived from microfiltration (**MF**) of milk are commonly referred to as native whey, ideal whey, or virgin whey. Native whey, as opposed to cheese whey, is free from somatic cells, lactic acid bacteria, bacteriophages, remnants of rennet (Maubois, 2002), cheese fines, and the glycomacropeptide from κ-CN (Brans et al., 2004). The neutral taste and pH, native protein conformation, and nutritional quality of whey proteins make native whey an excellent end product or ingredient in products addressed to infant, elderly, or sports nutrition.

Casein micelles and whey proteins can be separated by the use of MF with membranes with pore sizes in the range of 0.05 to 0.20 μm (Brans et al., 2004). The MF membrane material (i.e., ceramic, polymeric) and the membrane design and system [i.e., ceramic gradient, ceramic uniform transmembrane pressure (**UTP**), polymeric spiral-wound] influence the efficiency of whey protein removal, but also overall costs and cleaning procedures. Ceramic membranes in a UTP system give significantly better whey protein removal than ceramic graded permeability membranes and polymeric spiral-wound membranes (Zulewska et al., 2009). Optimal separation of caseins and whey proteins is of interest to the dairy industry. Therefore, the focus of this paper is on ceramic membranes in a UTP system. The composition of retentates and permeates from MF of skim milk is also influenced by several other factors:

Received February 26, 2016.
Accepted April 29, 2016.
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the composition of the skim milk, the pretreatment of the skim milk (Brandsma and Rizvi, 1999; Hernández and Harte, 2009; Svanborg et al., 2014), membrane pore size (Punidadas and Rizvi, 1999), channel geometry (Adams et al., 2015a), filtration temperature (Van Hekken and Holsinger, 2000; Hurt et al., 2015; Seibel et al., 2015), concentration factor (Punidadas and Rizvi, 1999; Kersten, 2001), wall shear stress (Le Berre and Daufin, 1996), and fouling (Le Berre and Daufin, 1996; Gésan-Guiziou et al., 1999, 2000; Jimenez-Lopez et al., 2008; Lawrence et al., 2008; Adams et al., 2015b).

Ceramic MF of skim milk to separate caseins and whey proteins is usually carried out at temperatures ranging from 50 to 55°C. Operating at higher filtration temperatures (>50°C) gives the potential benefit of reducing microbial growth (Walstra et al., 2006) and increasing flux as reported by Kersten (2001). However, Kersten (2001) observed a flux decline at temperatures above 55°C, explained by the precipitation of calcium phosphate. Hurt et al. (2015) reported, as opposed to Kersten (2001), that calcium phosphate precipitation did not cause membrane fouling when increasing the MF temperature from 50 to 65°C. They observed, however, a decrease in whey protein transmission with increasing filtration temperature, partly explained by the possible denaturation of whey proteins. Thus, the possible denaturation of whey proteins may be another disadvantage of MF of skim milk at higher temperatures. Significant denaturation of α-LA and β-LG occurs on heating milk above about 70°C (Anema, 2009), although conformational changes of β-LG has been reported to take place already at temperatures of 40°C (Qi et al., 1995, 1997). The separation of milk into cream and skim milk is usually carried out at around 57°C (range 55–65°C). In a continuous milk treatment process with ceramic MF at 60°C, the skim milk can be fed directly to the filtration process without temperature adjustment. The pumping energy and the friction forces arising from the flow of feed through the MF channels contribute to a temperature increase of the feed, and the temperature is likely to rise from 57 to 60°C. Thus, ceramic MF at 60°C might be relevant for the dairy industry.

Ceramic membranes with different pore sizes are available on the market. The effect of membrane pore size on the composition of MF retentate and permeate could possibly influence the choice of membrane pore size in an industrial MF application. Optimal fractionation of caseins and whey proteins is of interest to the dairy industry due to their different functional properties. Optimization of native whey protein removal and utilization of native whey proteins in value-added products may be a key to increase profitability of a ceramic MF process. For instance, relatively small differences in whey protein concentration in the MF permeate could have a major effect on the economic feasibility of an MF process with the goal to produce a native whey protein concentrate. Punidadas and Rizvi (1999) investigated the effect of gradient membranes with pore sizes of 0.05 and 0.20 μm on the composition of retentates and permeates from MF of skim milk. They reported that a portion of casein passed through the 0.20-μm membrane, whereas almost all the caseins were retained by the 0.05-μm membrane at the same time that whey proteins permeated the membrane. According to Zulewska et al. (2009), the permeate from MF of skim milk with gradient membranes has a higher casein proportion compared with permeate from MF of skim milk with ceramic membranes operated at a UTP. Information concerning the effect of different pore size on the protein fractionation of skim milk in a UTP MF system seems to be lacking and should be further investigated.

Milk proteins are the most valuable constituent of milk, and quantitative determination is important. Electrophoresis and column liquid chromatography are the main techniques used to separate and quantify milk proteins (Dupont et al., 2013). Protein compositions as found with capillary electrophoresis and reversed-phase HPLC are often given as relative values (Miralles et al., 2003; Heck et al., 2008; Svanborg et al., 2014) because of the difficulties in using standard curves for quantification of protein concentrations due to the impurity of the protein standards. Important information may get lost with the interpretation of relative values. A useful procedure to calculate real protein concentration values based on capillary electrophoresis is therefore presented in the present study.

Kersten (2001) and Hurt et al. (2015) studied the effect of temperature on protein fractionation using an MF system run in recycle mode at a constant transmembrane pressure (**TMP**) resulting in a minimal change in flux with filtration time. Investigation of the effect of MF temperature in recycle mode cannot preclude potential effects of the recycle time on the protein fractionation. Industrially, it is common to run MF processes at flux values above the critical flux to maximize the utilization of the membrane area, although operation above the critical flux causes fouling and can reduce operating time (Gésan-Guiziou et al., 2000). In an industrial MF process, the flux is kept constant because downstream unit operations in a continuous process are depending on a constant flow.

The objective of this study was to investigate the effect of membrane pore size and filtration temperature on protein fractionation of skim milk by MF to optimize fractionation of caseins and whey proteins.