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Effect of partial whey protein depletion during membrane filtration on thermal stability of milk concentrates

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ABSTRACT

Membrane filtration technologies are widespread unit operations in the dairy industry, often employed to obtain ingredients with tailored processing functionalities. The objective of this work was to better understand the effect of partial removal of whey proteins by microfiltration (MF) on the heat stability of the fresh concentrates. The micellar casein concentrates were compared with control concentrates obtained using ultrafiltration (UF). Pasteurized milk was microfiltered (80 kDa polysulfone membrane) or ultrafiltered (30 kDa cellulose membrane) without diafiltration (i.e., no addition of water) to 2× and 4× concentration, based on volume reduction. The final concentrates showed no differences in pH, casein micelle size, or mineral concentration in the serum phase. The micellar casein retentates (obtained by MF) showed a 20 and 40% decrease in whey protein concentration compared with the corresponding UF milk protein concentrates for 2× and 4× concentration, respectively. The heat coagulation time decreased with increasing protein concentration, regardless of the treatment; however, MF retentates showed a higher thermal stability than the corresponding UF controls. The average diameter for casein micelles increased after heating in UF but not MF concentrates. The turbidity (measured by light scattering) increased after heating, but to a higher extent for UF retentates than for MF retentates at the same protein concentration. It was concluded that the reduced amount of whey protein in the MF retentates caused a significant increase in the heat stability compared with the corresponding UF retentates. This difference was not due to ionic composition differences or pH, but to the type and amount of complexes formed in the serum phase.

Key words: microfiltration, heat stability, milk protein

INTRODUCTION

The use of concentrated milk protein ingredients has become increasingly widespread for high-protein beverages because of the high demand in the marketplace (Agarwal et al., 2015). Milk contains caseins and whey proteins, and these proteins have very different heat denaturation behavior. Whey proteins in their native form show a globular structure, which unfolds at temperatures >62°C (Singh and Havea, 2003). Caseins, on the other hand, are relatively stable to heating in their monomeric form because of their flexible structure.

The interactions between whey proteins and caseins have been widely studied in skim milk and are affected by time, temperature, rate of heating, pH, and protein concentration (Anema et al., 2006; Anema, 2009; Li et al., 2015). The complexes formed mostly with κ-CN and α_{S2}-CN may be soluble in the serum or associated with the micelles, according to the pH of the initial milk (Alexander and Dalgleish, 2005; Dalgleish and Corredig, 2012). The details on the formation of soluble and colloidal heat denatured aggregates in milk concentrated by filtration are not fully understood. Concentration increases the amount of protein in the soluble phase and leads to calcium phosphate solubilization, with a consequent increase in the formation of unsedimentable protein and soluble aggregates (Ferrer et al., 2011; Li and Corredig, 2014).

In designing high protein milk beverages, concentrates obtained by membrane filtration are often used. Whereas with UF both caseins and whey proteins are concentrated in the retentate, when using larger pore size membranes, it is possible to selectively concentrate caseins while transmitting whey proteins in the permeate. Depending on the extent of diafiltration, it is possible to achieve up to 95% of serum protein removal (Nelson and Barbano, 2005). Because of their sensitivity to heat, whey protein removal has been proposed as a way to produce more heat-stable beverages (Sauer and Moraru, 2012) and a solution for shelf-stable high-protein beverages (Sauer and Moraru, 2012).

Studies have been conducted on the stability of reconstituted dispersions of micellar micellar casein (from

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powders with less than 5% of total protein being whey proteins). These dispersions have been studied at different pH values, with variable calcium concentration and colloidal calcium distribution, with or without chelators [e.g., uridine monophosphate, disodium hydrogen phosphate, sodium hexametaphosphate, and trisodium citrate (de Kort et al., 2012; Sauer and Moraru, 2012)]. These studies have demonstrated poor heat stability of protein concentrates at pH lower than 6.9, with visible aggregation after sterilization, both by UHT or retort processes (Sauer and Moraru, 2012). Whereas at pH 6.9 the average casein micelle size increases after heating, this is not the case at higher pH (Sauer and Moraru, 2012). The positive effect of increasing the pH in the range of 6.7 to 7.3 on the heat stability of micellar casein concentrates was also reported by de Kort et al. (2012), who also demonstrated that calcium chelators increase heat stability of the concentrates to varying degrees depending on the chelator type and concentration.

As most of the research reported on the heat stability of milk concentrates has been conducted on reconstituted suspensions, in this research, it is hypothesized that the results may not apply to fresh concentrates. Profound changes occur in the calcium and phosphate equilibrium during drying and reconstitution. The objective of this work was to understand the effect of a reduction in whey protein in the heat stability of milk concentrates. The use of microfiltration (**MF**) to reduce the concentration of whey proteins and to increase the casein-to-whey ratio in the retentate may provide a solution to problems related to heat stability of milk concentrates, as well as an opportunity to create novel functional ingredients. The heat stability of fresh concentrates with a reduced concentration of whey proteins (referred to as MF retentates) was then compared with that of concentrates prepared by UF, still containing the original whey-to-protein ratio. It is important to note that by avoiding the diafiltration step during concentration, it was possible to maintain a comparable ionic composition in the serum phase among the fresh concentrates.

MATERIALS AND METHODS

Sample Preparation

Pasteurized skim milk (Sealtest/Agropur, supplied by Crown Dairy Ltd., Guelph, Canada) was 4× concentrated in a plate and frame membrane system (PUROSEP LT-2, SmartFlow Technologies, Apex, NC) as previously described (Li et al., 2015). The membranes used were either 30 or 80 kDa molecular cutoff. The plate and frame system differs from common spiral wound systems by allowing low transmembrane pressure and better molecular weight cut off due to low fouling of the membranes. Therefore, it was possible to achieve a reduction in the whey protein-to-casein ratio with a 80 kDa cutoff membrane. Samples obtained were referred as UF or MF retentates. Samples were collected at 2× and 4× concentration in both processes, based on volume reduction, by measuring the volume of permeate. Retentates concentrated 4× were also diluted back to 2× concentration with permeate obtained by UF. With this procedure the samples obtained had the same protein volume fraction, but with a different ratio of casein to whey proteins. Using UF permeate in the dilution experiments, it was possible to maintain a similar ionic composition of the serum phase for all the samples.

In total, 7 samples were examined: skim milk control; microfiltered milk 2× and 4× concentrated (**2×MF**; **4×MF**); UF milk 2× and 4× concentrated (**2×UF**; **4×UF**); and 4× MF and UF rediluted to 2× with UF permeate (**4×MFD**; **4×UFD**). In separate experiments, the concentrates were also subjected to dialysis with UF permeate at 4°C overnight to further equilibrate the serum ionic composition. A cellulose dialysis membrane (Fisher Scientific, Whitby, ON, Canada) with a molecular mass cutoff of 6 to 8 kDa was used. Dialysis was conducted at a ratio of 1 L of permeate per 50 mL of sample.

Heat Treatment and Heat Stability

The thermal stability of retentates was determined by heat coagulation time (**HCT**), defined as the time required during heating at 120°C to induce visible coagulation. The system used was a silicone oil bath (Haake AC200, Thermo Fisher Scientific, Newington, NH) fitted with a custom-made circulation device. Three-milliliter aliquots were transferred to a heat-resistant screw-cap test tube and immersed in the oil bath at 120°C (Eshpari et al., 2014). The samples were kept under agitation, and the elapsed time between the immersion and the first visible precipitation was recorded as the HCT.

To further characterize the effect of heating on the various concentrates, aliquots (9 mL) of retentate were transferred to a glass tube and heated at 120°C for 10 min in the silicone oil bath (see above). The heating time of 10 min was arbitrarily chosen, as, at this time, most samples did not show coagulation and could then be further analyzed. After heating, the samples were cooled immediately to room temperature by immersion in an ice bath.