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Investigating cold gelation properties of recombined highly concentrated micellar casein concentrate and cream for use in cheese making

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ABSTRACT

Highly concentrated micellar casein concentrate (HC-MCC), a potential ingredient for cheese making, contains ~20% casein with ~70% of serum proteins removed by microfiltration and diafiltration of skim milk, followed by vacuum evaporation. Our objective was to investigate cold gelation properties of recombined concentrated milk (RCM) by mixing thawed frozen HC-MCC and cream under different casein levels, pH, and protein-to-fat ratios, and with addition of sodium citrate or calcium. The HC-MCC was recombined with cream using low shear at 50°C for 30 min, and rheological measurements were conducted. Cold-gelling temperature [the temperature at which storage modulus (G') = loss modulus (G'')] was linearly correlated with casein levels from 8.6 to 11.5% ($R^2 = 0.71$), pH from 6.6 to 7.0 ($R^2 = 0.96$), and addition of sodium citrate from 0 to 0.36 mmol/g of casein ($R^2 = 0.80$). At pH 7.0, gelation occurred at 12, 26, and 38°C with 9, 10, and 11% casein, respectively. At pH 6.6, 6.8, and 7.0, RCM with 12% casein gelled at a mean temperature of 12, 26, and 37°C, respectively. Adding calcium chloride at 0.17 mmol/g of casein significantly increased cold-gelling temperature from 18 to $\geq 50^\circ\text{C}$, whereas no significant change was observed at levels up to 0.12 mmol/g of casein. Different protein to fat ratios ranging from 0.8 to 1.2 did not significantly influence gelling temperature. In transmission electron micrographs of RCM with 12% casein, casein micelles were nonspherical and partially dissociated into small protein strands. Upon addition of calcium chloride at 0.21 mmol/g of casein, casein micelles were more spherical and retained colloidal structure with the presence of aggregated casein micelles. These gelation processes of RCM with or without addition of trisodium citrate were both reversible. We propose that cold gelation of RCM occurs when protein strands that have been partially released from the casein micelles entangle, restrict their mobility, and

form a fine-stranded gel network. Upon addition of high levels of calcium, cold gelation was promoted presumably through direct aggregation of casein micelles. Understanding cold gelation properties can facilitate potential use of RCM in cheese making.

Key words: micellar casein, microfiltration, gelation, microstructure

INTRODUCTION

Ultrafiltration technology has been used in cheese making to increase cheese yield and daily milk processing capacity since the 1970s (Ernststrom et al., 1980; Kosikowski et al., 1985; Govindasamy-Lucey et al., 2004). The practical limit for concentration by UF is about $5\times$ (van Leeuwen et al., 1990). Hard and semi-hard cheeses can be made using diafiltrated and preacidified UF retentate ($\sim 5\times$) to reduce lactose and calcium phosphate contents, resulting in sufficient whey expulsion after rennet coagulation to achieve the final moisture content (Sutherland and Jameson, 1981). Concentrating milk to this level requires use of specialized equipment to handle the higher viscosity compared with milk, and because it is more difficult to expel whey from curd made from a 4 to $6\times$ retentate. An additional challenge is that having more serum proteins retained in the cheese slows changes in texture and flavor development during aging (Creamer et al., 1987; Lelievre et al., 1990; Bastian et al., 1991).

In the 1990s, microfiltration (MF) was used to concentrate skim milk and produce phosphate caseinate (Pierre et al., 1992), which is commonly called micellar casein concentrate (MCC) in the United States (Saboyainsta and Maubois, 2000; Nelson and Barbano, 2005; Hurt et al., 2010). Unlike UF, MF only concentrates micellar casein and does not concentrate serum proteins, up to 95% of which can be removed with extensive diafiltration (Pierre et al., 1992; Hurt et al., 2010; Marella et al., 2013). Therefore, MCC obtained through a MF process is potentially more suitable for cheese making than UF retentate.

In a similar way to using UF retentate, MF retentate with a low concentration level of 1.2 to $1.8\times$ is

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suitable for increasing cheese yield (Neocleous et al., 2002a,b; Govindasamy-Lucey et al., 2004). Typically, MCC manufactured using MF is only concentrated to 3 to 4 \times (Nelson and Barbano, 2005; Amelia et al., 2013; Hurt et al., 2015), whereas higher concentrations of 7 to 8 \times can be achieved by using milk acidification or further concentrating using UF or evaporation (Brandsma and Rizvi, 1999; Amelia and Barbano, 2013; Lu et al., 2015). Cheese making using milk retentate with high concentration factors such as 8 \times MF retentate is difficult and requires specialized equipment in manufacture, and therefore is not widely used in industry (Brandsma and Rizvi, 1999; Fox et al., 2000; Brandsma and Rizvi, 2001). Medium concentration factor (4 to 5 \times) of UF retentate is used commercially to produce high-moisture cheese such as quark, cream, and Feta cheese but is not used widely for semi-hard or hard rennet-curd cheese due to changes in cheese texture, flavor, and functionality (Green et al., 1981; Fox et al., 2000). Without the interference of serum protein, MF retentate with medium concentration has the potential to make cheese using conventional cheese making equipment while retaining cheese quality.

Because of the fouling issues caused by the fat when microfiltering whole milk, it is more feasible to use an MF skim milk concentrate and recombine it with cream for use in cheese making (Brandsma and Rizvi, 2001; Neocleous et al., 2002b; Govindasamy-Lucey et al., 2007). There has been some research using recombined MF retentate and cream to make Cheddar (St-Gelais et al., 1995; Neocleous et al., 2002a,b), Mozzarella (Garem et al., 2000; Brandsma and Rizvi, 2001), and pizza cheese (Govindasamy-Lucey et al., 2007). A highly concentrated micellar casein concentrate (**HC-MCC**) containing ~23% (wt/wt) protein has been manufactured through MF, diafiltration, and vacuum evaporation of skim milk (Lu et al., 2015). The recombined concentrated milk (**RCM**) obtained by mixing HC-MCC with cream is very suitable for cheese making

because of its high casein level (~20% wt/wt) and low serum protein level (<2% wt/wt). However, HC-MCC tends to gel at temperatures below 50°C, which can make it problematic for use in cheese making (Lu et al., 2015). Our objective was to investigate the effect of casein levels, pH, protein-to-fat ratios, and addition of citrate or calcium on cold gelation properties of RCM. Thermal reversibility and microstructure of RCM gel were also studied to better understand the factors that affect cold gelation.

MATERIALS AND METHODS

HC-MCC Manufacture

The HC-MCC was manufactured by MF and diafiltration of skim milk into MCC, followed by further condensation through vacuum evaporation as described by Lu et al. (2015). Pasteurized skim milk (72°C for 20 s) was processed into MCC in a 4-vessel, continuous MF unit (Filtration Engineering Inc., Champlin, MN) at feed temperature of 18 to 20°C, base line pressure of 35 kPa, differential pressure of 103 kPa, and a volume reduction of 4.0. Diafiltration was carried out at a level of 100% (based on volume of skim milk) with 20, 30, 30, and 20% of the diafiltration water added at vessels 1, 2, 3, and 4, respectively. During start-up, the feed and circulating boost pumps were sequentially started and the combined MF concentrate obtained from all 4 vessels was recycled back to the balance tank until the desired concentration factor was achieved. After about 10 min, diafiltration was started and after about 30 min, when volume reduction of 4.0 was reached, the MF concentrate was collected in a 2,000-L double-jacketed tank. Based on the mass of serum protein collected in the permeate relative to the mass of serum protein in the pasteurized skim milk, 70 to 75% of the serum protein present in the skim milk was removed during the MF process. The composition of the skim milk and MF retentate are shown in Table 1. The HC-MCC was transferred to 1.89-L containers, frozen at -20°C, shipped from South Dakota State University (Brookings) to Utah State University (Logan), and stored at -20°C until further analysis. The composition of the HC-MCC is shown in Table 1.

Composition Analysis

Compositions of pasteurized skim milk, MF retentate, and HC-MCC were analyzed. Total solids, total fat, and ash were analyzed as described by Hooi et al. (2004). Kjeldahl analysis was used to determine total N, noncasein N, and NPN (Hooi et al., 2004). Lactose

Table 1. Composition of highly concentrated micellar casein concentrate (HC-MCC) made using microfiltration and vacuum evaporation

Component	HC-MCC
Total solids %	30.14
Fat, %	0.94
Total N, %	23.02
Noncasein N, %	2.30
NPN, %	0.32
Casein N/total N	0.90
Lactose, %	3.79
Organic acids, %	0.39
Ash, %	2.33
Calcium, %	0.72

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