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Changes in the physical properties, solubility, and heat stability of milk protein concentrates prepared from partially acidified milk

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ABSTRACT

A limiting factor in using milk protein concentrates (MPC) as a high-quality protein source for different food applications is their poor reconstitutability. Solubilization of colloidal calcium phosphate (CCP) from casein micelles during membrane filtration (e.g., through acidification) may affect the structural organization of these protein particles and consequently the rehydration and functional properties of the resulting MPC powder. The main objective of this study was to investigate the effects of acidification of milk by glucono-δ-lactone (GDL) before ultrafiltration (UF) on the composition, physical properties, solubility, and thermal stability (after reconstitution) of MPC powders. The MPC samples were manufactured in duplicate, either by UF (65% protein, MPC65) or by UF followed by diafiltration (80% protein, MPC80), using pasteurized skim milk, at either the native milk pH (~pH 6.6) or at pH 6.0 after addition of GDL, followed by spray drying. Samples of different treatments were reconstituted at 5% (wt/wt) protein to compare their solubility and thermal stability. Powders were tested in duplicate for basic composition, calcium content, reconstitutability, particle size, particle density, and microstructure. Acidification of milk did not have any significant effect on the proximate composition, particle size, particle density, or surface morphology of the MPC powders; however, the total calcium content of MPC80 decreased significantly with acidification (from 1.84 ± 0.03 to 1.59 ± 0.03 g/100 g of powder). Calcium-depleted MPC80 powders were also more soluble than the control powders. Diafiltered dispersions were significantly less heat stable (at 120°C) than UF samples when dissolved at 5% solids. The present work contributes to a better understanding of the differences in MPC commonly observed during processing.

Key words: milk protein concentrate, partial acidification, physical properties, solubility, heat stability

INTRODUCTION

Milk protein concentrate (**MPC**) powders are manufactured from skim milk by membrane filtration and spray drying. The major components of these shelf-stable food ingredients are protein, lactose, fat, and minerals. The proportion of these components varies depending on the extent of concentration and the type of membrane separation used. The proteins in MPC consist of caseins and whey proteins, present at the same ratio (4:1) as in milk.

Milk protein concentrate powders may present poor solubility (Mistry and Pulgar, 1996; McKenna, 2000; Anema et al., 2006; Havea, 2006; Baldwin and Truong, 2007; Mimouni et al., 2010; Sikand et al., 2011). Poor solubility has been attributed to the slow release of casein micelles from powder particles; it is shown that the slowly solubilizing material is almost entirely made up of caseins, whereas the whey proteins, minerals, and lactose are more soluble (Anema et al., 2006; Havea, 2006; Mimouni et al., 2010). It is widely believed that the processes involved in manufacture of MPC maintain the overall original state of casein micelles (Mulvihill and Ennis, 2003). The delicate ionic equilibrium between serum and micelles in milk (Holt et al., 1981) is shifted during membrane filtration. This change may cause a lasting effect on the casein micelles, affecting their rehydration and the functional properties of the resulting concentrates. It is known that certain processing steps can improve the rehydration properties of MPC powders; for example, heating the milk after pH adjustment before membrane filtration (Blazey et al., 2000), drying at lower temperatures (Schuck et al., 1994), adding salts before or after UF (Carr, 2002; Schuck et al., 1999, 2002; Mao et al., 2012; Sikand et al., 2013), adding sodium caseinate to the retentate before spray-drying (Schokker et al., 2011), adding polydextrose and whey proteins (Davenel et al., 1997), or partly depleting calcium by adding chelating agents, using cation exchange chromatography, or acidifying the milk (Bhaskar et al., 2001).

Little has been written about the effect of partial acidification of milk before UF on the composition, physical properties, and rehydration behavior of the re-

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sulting MPC powders. Investigating the effects of such manipulations on the thermal stability of the resulting powders after reconstitution is also of considerable interest, as these ingredients are often subjected to extensive heat treatments when incorporated into beverages. In such applications, the ability to withstand heating regimens as high as, for example, 121°C for 15 min, is very important.

In this study, we explored the effect of acidification to pH 6 by addition of glucono-δ-lactone (**GDL**), on the physical properties, reconstitutability, and thermal stability (after reconstitution). Milk pH was modified before ultrafiltration, which was then carried out with or without a subsequent diafiltration (**DF**) stage. This work attempted to use methods for reconstitution and observations commonly used in the industry to depict the properties of the powders under conditions relevant to processors.

MATERIALS AND METHODS

Materials

Pasteurized skim milk was obtained from Producer's Dairy Foods Inc. (Fresno, CA). Analytical-grade reagents were from Sigma-Aldrich Chemical Co. (St. Louis, MO). Glucono-δ-lactone was purchased from Roquette America Inc. (Geneva, IL). When added to an aqueous solution, GDL dissolves rapidly but hydrolyses progressively to gluconic acid, leading to a controlled decrease of pH, which suited the purposes of this research. Ultrapure water (Milli-Q Ultrapure Water Purification Systems, Billerica, MA) was used to prepare all the solutions.

Preparation of MPC Powders Acidified with GDL

Milk protein concentrate powders were manufactured in duplicate using pasteurized skim milk, either by UF to achieve an MPC with 65% protein (**MPC65**) or by UF followed by DF to achieve an MPC with 80% protein (**MPC80**). Controls were prepared at the native milk pH (~pH 6.6) or GDL was added (at 3.25 g/L) to reach pH 6.0 before starting membrane filtration; GDL gradually decreases the pH of milk and is often used as a model for lactic acid fermentation in milk. The pH value of 6.0 was selected because little physical and chemical changes occur to the casein micelles at this pH (Alexander and Dalgleish, 2004). By doing so, the effect of limited acidification could be studied. Figure 1 illustrates the various stages of MPC manufacture. The MPC65 and MPC80 powders were manufactured in the pilot plant of Dairy Products Technology Center at California Polytechnic State University (San Luis Obispo). Pasteurized skim milk (300 kg) was ultrafiltered by using a model R12 cross-flow membrane recirculatory pilot plant unit (Niro Inc., Hudson, WI) equipped with dual 10-kDa-cut-off, spiral-wound polyethersulfone membranes (Snyder Filtration, Vacaville, CA). Ultrafiltration began at 5.8°C ± 1.5°C. During UF, the temperature was allowed to increase in such a way that by the end of the UF process, the temperature was 20 ± 1.6°C. Milk was concentrated up to 5× concentration, resulting in 60 kg of retentate.The UF retentate was then divided into 2 equal portions, 30 kg of which was spray-dried and the other 30 kg was diafiltered to achieve 6× concentration using the same membrane pilot-plant unit, mentioned above. At the end of DF, 20 kg of retentate was collected and spray-dried. Both UF and DF retentates were spray-dried with a pilot Niro Filtermat Spray Dryer (Niro Inc.) to approximately 3.5% moisture, and the obtained MPC powders were immediately collected and sealed in airtight bags for further analysis. Inlet temperature was about 210°C and outlet temperature was 82°C. Four MPC powders were produced (Figure 1), 2 MPC65 and 2 MPC80. The control samples were named **UF-C** and **DF-C** and the GDL-treated samples were named **UF-G** and **DF-G**.

Composition

Powder samples were analyzed for proximate composition (AOAC International, 1995). Total protein was determined by Kjeldahl. Ash content was determined by ignition at 550°C in an electric muffle furnace (AOAC International, 1995; method 945.46; 33.2.10). Fat content was determined by the Mojonnier method (AOAC International, 1995; method 989.05; 33.2.26), and free moisture content by oven-drying method (AOAC International, 1995; method 990.20; 33.2.44). Lactose was determined by difference.

Total Ca content of the powders was determined by using Hewlett Packard 4500 ICP-MS (Agilent Technologies, Santa Clara, CA), according to EPA method 6020 A (US EPA, 2007).

Solubility Determination

The MPC powders were reconstituted in ultrapure water (5% wt/wt) for 3 h using a laboratory stage mixer (R010 Power, IKA Works, Wilmington, NC) at 960 rpm (speed setting #8) at 23°C ± 1.0°C. Aliquots (13 mL) of these samples were transferred to a series of 15-mL Falcon tubes and centrifuged at 700 × g for 10 min at 23°C. The supernatant was separated from the pelleted material by withdrawing the supernatant into a pipette. Total solids (TS) contents of both bulk