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Lactose crystallization in milk protein concentrate and its effects on rheology

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ABSTRACT

This work investigated the impact of lactose crystallization in milk protein concentrate and its effects on rheology. Crystallization was conducted with varying levels of total solids (44, 46, and 48%), temperature (15, 20, and 25 °C), and α-lactose monohydrate powder size (40 and 100 mesh) as seed crystals. Concentration of total solids was the most important factor affecting crystallization rate. MPC 42 concentrates exhibited a non-Newtonian shear thinning behavior. The difference in viscosities among the total solids content was caused by the amount of crystal mass and not by the total solids itself. Samples seeded with 100 mesh lactose seed exhibited a significantly higher consistency index, higher yield stress, and lower flow behavior index than samples seeded with 40 mesh seeds. A second order polynomial model provided empirical relationships between rheology and volume fraction.

1. Introduction

Milk protein concentrate (MPC) is any type of concentrated milk product manufactured from skim milk that contains 40–89% milk proteins (Mulvihill, 1992). As the protein content of MPC represented by caseins and whey proteins increases, the lactose levels decrease. A lower lactose content provides a whiter and blander flavor, thus making MPC suitable for the formulation of products where flavor and aroma compounds are considered defects (Mistry, 2002).

MPC is manufactured by ultrafiltration and diafiltration of skim milk to partially remove soluble minerals and lactose, followed by vacuum evaporation until the concentrate reaches 32–35% total solids (Gelter et al., 1996) and spray drying of the retentate. During spray drying, atomized feed in the form of fine droplets comes into contact with hot air in the drying chamber and dries within a fraction of a second, giving insufficient time for the droplets to crystallize (Das and Langrish, 2012). As a result, lactose forms a solid-like amorphous glass directly from the liquid state. Amorphous components are in a nonequilibrium state, so a drive toward thermodynamic equilibrium will occur (Huppertz and Gazi, 2016), especially under sufficient conditions of temperature (24 °C) and moisture content (relative humidity of 37%) (Roos, 2002). Crystallization is preceded by moisture sorption from air and the formation of liquid bridges between powder particles, which leads to an increase in the cohesiveness, causing them to readily adhere to the walls of spray dryers, and caking during storage (Fitzpatrick et al., 2007).

It is necessary to control the behavior of lactose in dairy ingredients powders in order to ensure desired powder handling properties and powder stability (Huppertz and Gazi, 2016). Commercially available spray-dried dairy products usually contain 10–15% amorphous lactose (Hourigan et al., 2013). To avoid this problem, it is necessary to precrystallize lactose as α-lactose monohydrate, which is nonhygroscopic, prior to spray drying. This ensures that a high proportion (typically 75% or higher) of the lactose in the spray-dried product is in the nonhygroscopic α-lactose monohydrate form, making it more stable to humidity (Darcy and Buckton, 1998). Up to 50–75% crystallinity may be achieved in whey powder by precrystallization (Paterson, 2009).

During precrystallization, lactose content in solution decreases to eventually reach the equilibrium concentration. Solute transfers through the bulk solution to the crystal interface. With the decrease in lactose concentration in the aqueous phase, the solution phase viscosity decreases (Pancoast and Junk, 1980). However, with progress of crystallization, the presence of crystals contributes to an

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increase in the overall slurry viscosity. During the spray drying process, higher viscosity causes the concentrate to resist agitation, tending to prevent its breakup and leading to a larger average droplet size. Because large droplets have a lower ratio of surface area to volume, they require a longer time to dry and will affect the properties of the final powder. Additionally, the spray dryer is easily fouled by the large droplets, resulting in short runtimes (Schuck et al., 2005). The viscosity of skim milk concentrate should not exceed 100 cP at 40 °C measured on the feed to be atomized (Morrison, 2001).

There are numerous reports on the rheology of milk based products, including raw milk and acidified products such as yogurt and cheese (Almanza-Rubio et al., 2016; Diezhandino et al., 2016; Sah et al., 2016; Sharma et al., 2016). However, there have been few studies on concentrated systems (Hadde et al., 2015; Meletharayil et al., 2015; Trinh et al., 2007; Karlsson et al., 2005). In the case of MPC, no literature has been published on the pre-crystallization step. The information gained through the rheological characterization of reconstituted MPC will be useful for process optimization. The objective of this investigation is to study lactose crystallization in MPC 42 and its effects on rheology. The rheology with respect to the effects of total solids content, temperature, and lactose seed size on lactose crystallization was investigated.

2. Materials and methods

2.1. Materials

Low heat MPC 42 powder, containing 42% protein, was obtained from United Dairymen of Arizona (Tempe, AZ). α-Lactose monohydrate powder was obtained from Leprino Foods (Denver, CO). Anti-foaming agent was obtained from Masson Group Company Limited (Guangzhou, China). Powdered sodium azide was obtained from Agropur (La Crosse, WI).

2.2. Sample preparation

MPC 42 was reconstituted to solids content of 44, 46, and 48% with distilled water at ambient temperature. A hand-held blender was used to stir and mix the concentrate for 15 min. The solution was heated to 35 °C in a hot water bath. The concentrate was then cooled to crystallization temperatures of 15, 20 and 25 °C, after which it was filtered using a sieve of 841 microns opening (U.S. Standard Sieve Series, Fisher Scientific Company) to remove any remaining large particulates.

2.3. Crystallization procedure

Batch isothermal lactose crystallization runs were carried out in a 500 mL glass vessel maintained at crystallization temperatures of 15, 20 and 25 °C using a water bath (Fischer Scientific Isotemp 2150). Agitation was provided by an anchor impeller (diameter between paddles = 3.2 cm) operated at 15 rpm. The concentrates were seeded at time $t = 0$, by adding α-lactose monohydrate powder at a concentration of 0.1% w/w. Seed size was selected by using two commercial lactose powders of size designation of 40 and 100 mesh (420 and 149 μm) (Leprino Foods, Denver, CO). An anti-foaming agent was added at a volume of 100 μL and powdered sodium azide (NaN_3) was added at a final concentration of 0.05% w/v to prevent microbial growth during the experiments. Experiments lasted 7 h and were performed in triplicate.

2.4. Analytical methods

2.4.1. Concentration

An Abbe 3L BenchTop Refractometer (Thermo Scientific, Waltham, MA) was used to determine the dissolved lactose on an hourly basis. The effect on the refractive index (RI) of the presence of various components in MPC 42 being unknown, a calibration curve was constructed to convert the RI of different MPC 42 concentrations (10, 20, 30, 40, and 50% total solids) into lactose concentration expressed as g lactose per 100 g water. For this purpose, 44.1% was used as the percentage of lactose in MPC 42 powder, based on the manufacturer's specifications, in order to calculate the amount of lactose (in grams) in each MPC 42 concentration. A plot of refractive index against lactose concentration over the whole concentration range of interest was obtained (Fig. 1). This calibration curve was used to derive the mass of crystallized lactose. The temperature of the refractometer was maintained at 20 °C by circulating water with a water bath circulator (VWR Scientific, model 1157, Rochester, NY) through the refractometer. Sampling was performed by extraction of 100 μL from the concentrate.

2.4.2. Calculation of crystal mass

The crystal mass at any time during crystallization was calculated by a model developed by Mimouni et al. (2005), as shown in the following equations:

$$\text{M } H_2O\,(0) = \text{M } H_2O\,(t) + 0.05 \cdot \text{M Crystal}\,(t) \quad (1)$$

$$[C\,(0)/100] \cdot \text{M } H_2O\,(0) = [C\,(t)/100] \cdot \text{M } H_2O\,(t) + 0.95 \cdot \text{M Crystal}\,(t) \quad (2)$$

$$\text{M Crystal}\,(t) = \text{M } H_2O\,(0) \cdot [C\,(0) - C\,(t)/95 - 0.05 \cdot C\,(t)] \quad (3)$$

Equ. (1) represents the mass balance of water; Equ. (2) represents the mass balance of lactose between $t = 0$ and t; and Equ. (3) represents the crystal mass obtained at time t. In these equations, C is the dissolved lactose concentration expressed as g.100 g^{-1} water, $C(t)$ is the lactose solubility value at a respective temperature, while

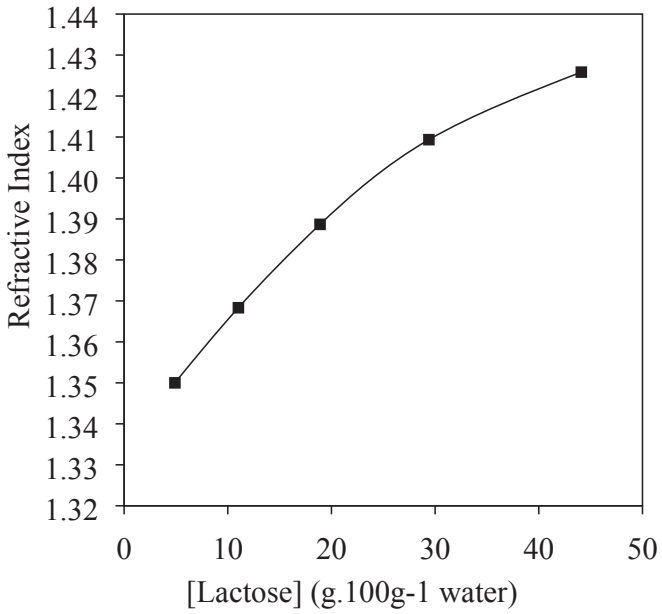

Fig. 1. Refractive index as a function of lactose concentration in milk protein concentrate.