International Dairy Journal 69 (2017) 19–22

Contents lists available at ScienceDirect

International Dairy Journal

journal homepage: www.elsevier.com/locate/idairyj

Influence of milk pre-heating conditions on casein–whey protein interactions and skim milk concentrate viscosity

Suresh G. Sutariya [a], Thom Huppertz [a, b], Hasmukh A. Patel [a, c, *]

[a] Dairy Science Department, South Dakota State University, Brookings, SD, USA
[b] NIZO Food Research, P.O. Box 20, 6710 BA, Ede, The Netherlands
[c] Dairy Foods Research and Development, Land O'Lakes, Inc., Arden Hills, MN, USA

ARTICLE INFO

Article history:
Received 18 December 2016
Received in revised form
16 January 2017
Accepted 16 January 2017
Available online 4 February 2017

ABSTRACT

The viscosity of concentrates (50–55% total solids) prepared from skim milk heated (5 min at 80 or 90 °C) at pH 6.5 and 6.7 was examined. The extent of heat-induced whey protein denaturation increased with increasing temperature and pH. More denatured whey protein and κ-casein were found in the serum phase of milk heated at higher pH. The viscosity of milk concentrates increased considerably with increasing pH at concentration and increasing heating temperature, whereas the distribution of denatured whey proteins and κ-casein between the serum and micellar phase only marginally influenced concentrate viscosity. Skim milk concentrate viscosity thus appears to be governed primarily by volume fraction and interactions of particles, which are governed primarily by concentration factor, the extent of whey protein denaturation and pH. Control and optimization of these factors can facilitate control over skim milk concentrate viscosity and energy efficiency in spray-drying.

1. Introduction

Evaporation and spray drying are two major steps involved in the manufacture of skim milk powder. Since removal of water by evaporation requires ~10–20 times less energy than by spray-drying (Fox, Akkerman, Straatsma, & De Jong, 2010), maximizing the solids content of concentrate prior to spray-drying is desired economically and environmentally. Commonly, skim milk powder manufacturers concentrate skim milk to 45–50% total solids prior to drying. At higher solids content, viscosity increases dramatically with further increases in total solids.

The viscosity of concentrated milk depends primarily on the solids content and composition of the milk, heating conditions, evaporation temperature and holding time of the concentrate (Bloore & Boag, 1981; Fernandez-Martin, 1972; Karlsson, Ipsen, Schrader, & Ardö, 2005; Snoeren, Damman, & Klok, 1982; Vélez-Ruiz & Barbosa-Cánovas, 1998). Snoeren et al. (1982) showed that increases in viscosity as a result of heating of milk are related to an increase in milk protein voluminosity, because denaturation increases the voluminosity of the whey proteins; as a result, concentrate viscosity was found to correlate strongly with the extent of whey protein denaturation (Snoeren et al., 1982). More recently, Anema, Lowe, Lee, and Klostermeyer (2014) suggested that not only the extent of denaturation, but also the distribution of denatured whey proteins, and κ-casein, between the serum and micellar phase affects the viscosity of milk concentrates for solids content <40%, but that at higher total solids content, viscosity was less influenced by the distribution of denatured whey proteins and κ-casein. Hence, for reducing skim milk concentrate viscosity in industrial operations, tailoring casein–whey protein interactions does not appear to be a key route based on the results of Anema et al. (2014). However, the studies performed by Anema et al. (2014) only studied solids contents up to 45%, which are at the lower end of industrial processing. For investigating options to improve spray-drying efficiency by reducing milk concentrate viscosity, effects at solids contents >50% should be considered.

Another factor that is crucial to consider in these studies is inter-particle interactions. In unconcentrated milk, where the inter-particle distance is several times larger than the particle diameter, such effects may appear to be limited. However, in concentrated milk the inter-particle distance becomes considerably smaller than particle diameter and particle interactions become much more important in governing rheological behavior (Karlsson et al., 2005), particularly in milk concentrates of >50% total solids.

* Corresponding author. Tel.: +1 651 375 1497.
E-mail address: HPatel@Landolakes.com (H.A. Patel).

http://dx.doi.org/10.1016/j.idairyj.2017.01.007
0958-6946/

Hence, the objectives of the current work were to evaluate the role of whey protein distribution, as well as pH, on the rheological behavior of skim milk concentrates (>50% dry matter) and evaluate whether protein distribution or other factors govern the rheological behavior of the concentrates.

2. Materials and methods

2.1. Sample preparation

Low-heat skim milk powder (Associated Milk Producers Inc., New Ulm, MN, USA; whey protein nitrogen index > 6; 36%, w/w, protein on a dry matter basis) was reconstituted in deionized water at 10% (w/v) for 4 h at room temperature. Sodium azide (0.02%) was added to prevent bacterial growth and the pH was subsequently adjusted to 6.5 and 6.7 by addition of 1 M HCl or 1 M NaOH with continuous stirring. The pH-adjusted skim milk samples were then stored overnight at 4 °C. Samples were brought to room temperature the next day and pH was checked and readjusted if required. Samples were the heated in a hot water bath to 80 or 90 °C, held at this temperature for 5 min and subsequently cooled to room temperature in an ice-water bath. For samples at pH 6.5 and 6.7 that were heated at 80 °C, subsamples were subsequently adjusted to pH 6.7 and 6.5, respectively. For each sample variant described above, 3 replicate samples were prepared.

2.2. Determination of whey protein denaturation and whey protein distribution

To determine the level of whey protein denaturation in heated milk samples, the pH 4.6-soluble fraction of milk samples was prepared as described by Gazi and Huppertz (2015). The pH 4.6-soluble fraction and whole sample were subsequently analyzed by sodium dodecylsulfate-polyacrylamide gel electrophoresis (SDS-PAGE) as described by Meletharayil, Patel, and Huppertz (2015) and the stained gels were scanned using a Bio-5000 Microtek scanner (Microtek, Hsinchu, Taiwan) and the images were analyzed to quantify the intensity of whey protein bands using the Bio-image Intelligent Quantifier v 3.3.7 system (Bio Image Systems, Jackson, MI, USA). The level of whey protein denaturation was calculated from the difference in band intensities of the pH 4.6-soluble fraction unheated milk sample (residual native whey protein in the unheated samples) and the pH 4.6-soluble fraction of the heated sample (residual native whey protein in the heated sample). To determine the distribution of whey proteins in heated milk, samples were centrifuged at 25,000× *g* for 1 h at 20 °C, followed by SDS-PAGE analysis, scanning and image quantification of the supernatants as described above.

2.3. Viscosity of concentrated milk samples

Samples of 1.5 L of skim milk were concentrated at 60 °C under vacuum using a rotary evaporator (Rotovapor Hei-VAP Value/G3, Heidolph, Schwabach, Germany) with an absolute pressure of 180–190 mbar. Concentration was continued until a solids content of 50–55% (m m^{-1}) was reached. Solids content was determined by oven drying. Immediately after concentration, apparent viscosity was measured at 55 °C, over a shear rate profile of 50–500 s^{-1}, using a STRESSTECH rheometer (ATS Rheosystems, Bordentown, NJ, USA), fitted with an CC 25 CCE SS cup and bob attachment. Shear rate was increased by 50 s^{-1} at 10 s intervals. Since milk concentrates showed non-Newtonian viscosity behavior, viscosity profiles were evaluated using a Power Law model:

$$\sigma = K^{*}\gamma^{n} \quad (1)$$

where, σ is the shear stress (Pa), γ is the shear rate (s^{-1}), *K* is the consistency coefficient (Pa s^{n}) and n is the dimensionless flow behavior index. *K* and n were determined from a plot of log σ versus log γ, which has log *K* value as the intercept and the n as slope. For shear thinning liquids, $0 \leq n \leq 1$, with lower values indicating a greater degree of shear thinning.

2.4. Statistical analysis

The results were analyzed by SAS (version 9.3; SAS Institute Inc., Cary, NC, USA). Analysis of variance (ANOVA) was performed to examine the statistical difference between the samples, and the differences were considered significant when *p*-values were less than 0.05. All experiments were replicated on three individual milk samples.

3. Results

3.1. Whey protein denaturation and distribution in heated milk samples

Heat treatment of samples at 80 °C denatured ~43% and 50% of native whey protein present in samples at pH 6.5 and 6.7, respectively, whereas heat treatment of the same samples at 90 °C denatured ~84% and 93% of total native whey protein present, respectively (Table 1). In addition, the distribution of whey proteins and κ-casein between the serum phase and colloidal phase was also affected by the pH at which heat treatment was carried out. Heat treatment at pH 6.7 resulted in higher levels of whey proteins and κ-casein in the serum phase of milk than heat treatment at pH 6.5 (Table 1); this, again, is in agreement with previous studies (Anema, 1998; Anema & Klostermeyer, 1997; Anema et al., 2014; Cassandra & Dalgleish, 2006; Renan et al., 2006; Vasbinder & de Kruif, 2003).

3.2. Viscosity of concentrated milk samples

Samples were concentrated to a solids content of 50–55% dry matter by evaporation (Table 2) which was accompanied by a reduction in pH of the samples, by ~0.4–0.5 pH units, irrespective of starting pH. These decreases in pH are in agreement with previous studies and can be attributed to a concentration of acids by

Table 1
Extent of whey protein (WP) denaturation and individual proteins in the serum phase of milk heated at pH 6.5 or 6.7 at 80 or 90 °C for 5 min.[a]

pH at heat treatment	Temperature (°C)	Denatured WP (% total)	β-Lactoglobulin (band intensity)	α-Lactalbumin (band intensity)	κ-Casein (band intensity)
6.5	80	43 ± 5[a]	0.80 ± 0.01[a]	0.47 ± 0.01[a]	0.37 ± 0.05[a]
6.7	80	50 ± 2[a]	1.06 ± 0.02[b]	0.74 ± 0.01[b]	0.62 ± 0.05[b]
6.5	90	84 ± 7[b]	0.32 ± 0.00[c]	0.24 ± 0.01[c]	0.28 ± 0.04[c]
6.7	90	93 ± 2[b]	0.79 ± 0.05[a]	0.33 ± 0.04[d]	0.55 ± 0.09[d]

[a] Values are means ± standard deviation of measurements on three individual milk samples; means within the same column not sharing a common superscript letter are significantly different ($P < 0.05$). Band intensity values are in arbitrary units.