J. Dairy Sci. 90:57–65

The Formation of Calcium Lactate Crystals is Responsible for Concentrated Acid Whey Thickening

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ABSTRACT

The use of spray drying for dehydration of acid whey is generally limited by the appearance of uncontrolled thickening and solidifying of the whey mass during the lactose crystallization step. The origin of this physical change is still unknown and probably linked to complex interactions between physical properties and chemical composition of these products. To understand this phenomenon, we simulated the thickening of concentrated acid whey on a laboratory scale by measuring the flow resistance changes as a function of time and whey composition. The thickening process was characterized by an amplitude of torque and a lag time (induction time). Thickening of lactic acid whey concentrate occurred regardless of the presence of whey proteins or lactose crystals. Moreover, this work clearly demonstrated that the thickening process was due to the formation of filamentous structures corresponding to calcium lactate crystals and showed a large dependence on calcium and lactate contents, pH, and phosphate concentration.
Key words: acid whey, rheology, thickening, calcium lactate

INTRODUCTION

Spray drying is one of the most convenient techniques for producing milk, whey, and derivative powders. However, to maximize energy efficiency, these products must be concentrated at the highest possible TS content during a previous evaporating step, which is usually conducted in falling film vacuum evaporators. Additionally, between the evaporation and drying steps, whey powder manufacturing includes a lactose crystallization step that is often performed in a stirred tank over a few hours (Jensen and Oxlund, 1988). In the case of lactic acid whey powder manufacturing, unpredictable and drastic increases in the viscosity of the concentrate occur during the crystallization step, which prevent the concentrate from being pumped to the dryer and often lead to its solidification.

A number of researchers investigated the so-called phenomenon of age-thickening or age-gelation in either concentrated (skim) milk or condensed sweetened milk (Noda et al., 1976; Snoeren et al., 1984; Aoki et al., 2002; Bienvenue et al., 2003). Their results indicated that preheating of milk, storage temperature, and TS content of the concentrate or whey proteins appeared to play an important role in the thickening process. But they mainly indicated that the marked increases in viscosity observed during storage of concentrated (skim) milk and condensed sweetened milk had to be attributed to casein micelle transformation and aggregation. In contrast, to our knowledge, very few studies have analyzed thickening of whey concentrates. Mikelsone (1984) studied the behavior of ammoniated whey concentrate neutralized either by calcium hydroxide or calcium carbonate. He pointed out sharp increases in viscosity before solidification after 90 min of storage with calcium hydroxide compared with after 45 min with calcium carbonate. Hargrove et al. (1974, 1976) investigated rheological properties of deproteinized concentrates. They observed that above 60% (wt/wt) TS, the concentrates were very viscous and solidified into a firm mass on standing for 30 min to 1 h. However, no explanation of the phenomenon was proposed.

The purpose of the present study was to understand the causes and origin of lactic acid whey concentrate thickening. In the following, a method was used in which whey concentrates were stirred under controlled conditions (temperature, agitation rate) and changes in rheological properties were measured in real time via the monitoring of flow resistance. Explanations of these changes were then proposed and, finally, the effects of some chemical parameters on thickening of lactic acid whey concentrates were investigated.

MATERIALS AND METHODS

Preparation of Concentrates

Preparation of Lactic Acid Whey Concentrate. Lactic acid whey concentrate was prepared by dissolv-

Received June 27, 2006.
Accepted August 31, 2006.
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Table 1. Chemical compositions of lactic acid whey concentrate and deproteinized concentrated acid whey supernatant (DCAWS) prepared at 30°C

	Lactic acid whey concentrate		DCAWS	
	g/kg of product	g/100 g of H_2O[1]	g/kg of product	g/100 g of H_2O
TS	579.9	—	372.0	—
Total N	53.3	12.7	22.9	3.6
NPN	26.9	6.4	22.4	3.6
Ash	73.9	17.6	70.5	11.2
Lactose	379.8	90.4	184.0	29.3
Lactate	71.2	16.9	108.7	17.3
Phosphate	12.5	3.0	12.0	1.9
Chloride	10.4	2.5	11.8	1.9
Sulfate	1.0	0.2	1.1	0.2
Calcium	12.8	3.0	10.5	1.7
Magnesium	1.1	0.3	1.5	0.2
Sodium	3.1	0.7	4.3	0.7
Potassium	13.6	3.2	22.7	3.6

[1]These values were calculated according to the following formula: g/100 g of H_2O = g/kg of product/(1,000 − TS) × 100.

ing 100 g of lactic acid whey powder, supplied by Euroserum (Saint Martin Belle Roche, France), in 70 g of deionized water at room temperature. Complete lactose crystal dissolution was ensured through heating at 80°C under agitation for 20 min. Chemical composition of the concentrated lactic acid whey is reported in Table 1. This specific powder to water ratio was set up to mimic the TS content of industrial lactic acid whey concentrates at the end of the vacuum evaporation step.

Preparation of Deproteinized Concentrated Acid Whey Supernatant. The overall steps of the preparative procedure are given in Figure 1. First, whey proteins were removed from industrial acid whey by selective protein concentration with an 8-kDa ultrafiltration membrane (ceramic membrane, Tami Industries, Nyons, France) as described by Fauquant et al. (1988). Then, the concentration and spray drying of the protein-free permeate were performed at Bionov (Rennes, France) in a 3-stage pilot-plant spray dryer (GEA, Niro Atomizer, St Quentin en Yvelines, France) according to Schuck et al. (2002). Crystallized lactose deproteinized concentrated lactic acid whey was prepared by mixing the above powder (100 g) with 70 g of deionized water at 20 or 30°C with gentle shaking for 5 min. Centrifugation (160 × g, 10 min, 20 or 30°C; Funke Gerber, Berlin, Germany) of this suspension and filtration (0.45-μm syringe filters, Pall Gelman Laboratory, Ann Arbor, MI) of the supernatant resulted in a soluble phase that contained dissolved lactose in slight supersaturation and other soluble components including lactate, phosphate, and calcium in high concentrations (Table 1). This solution, the deproteinized concentrated acid whey supernatant (**DCAWS**), contained nei-

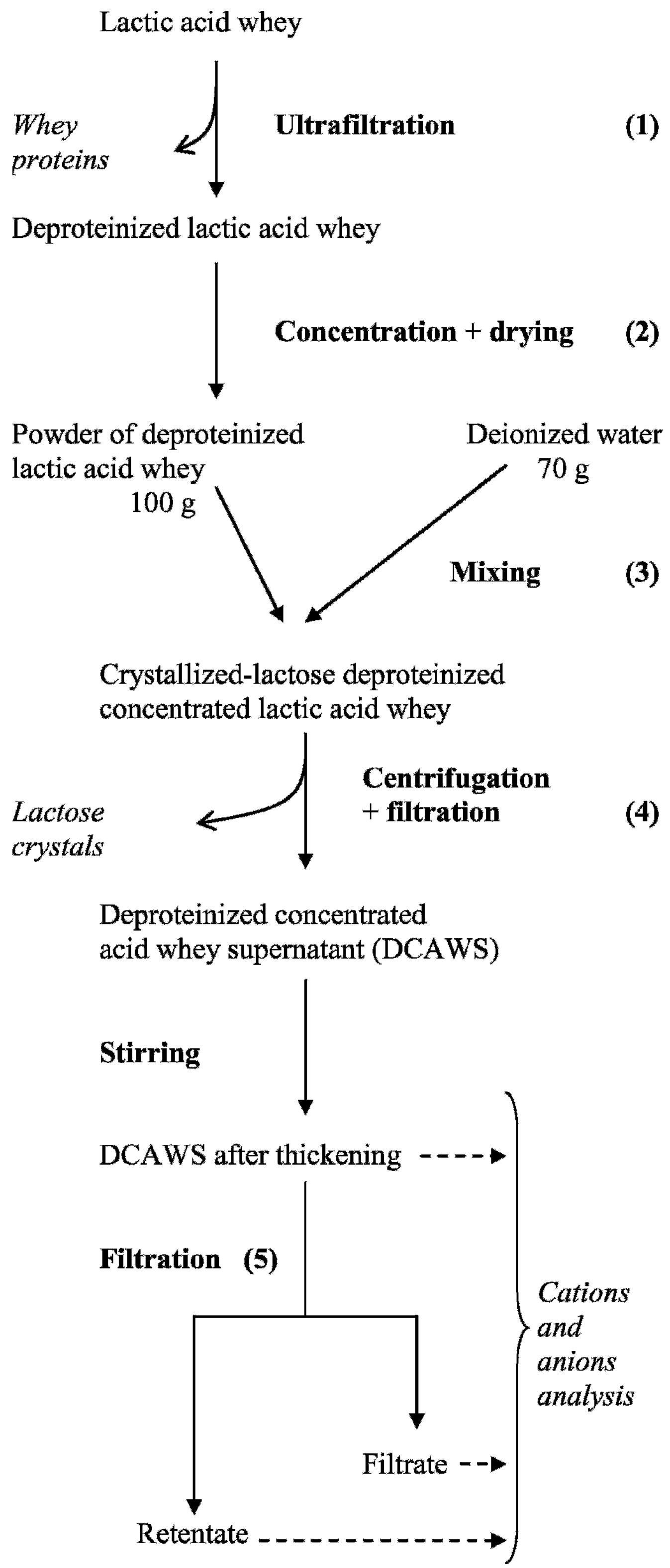

Figure 1. Schematic description of the various steps of the preparation of deproteinized concentrated acid whey supernatant (DCAWS) and the separation of insoluble fraction of thickened DCAWS for cations and anions analysis.