

## Production of Ultrafiltered Skim Milk Retentate Powder. 2. Functional Properties<sup>1</sup>

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### ABSTRACT

High protein skim milk retentate powders with modified functional properties were produced using UF and spray drying. Different heat treatments (65°C for 30 min, 75°C for 28 s, and 85°C for 28 s) and pH adjustments (6.4, 6.7, and 7.0) were applied before spray drying. The pH adjustment affected gel water-holding capacity, acid gel strength, emulsifying capacity, and foaming capacity. Heat treatment also affected gel water-holding capacity, acid gel strength, and emulsifying capacity, but not foaming capacity. The interaction of heat treatment and pH adjustment affected gel water-holding capacity, acid gel strength, emulsifying capacity, and foaming capacity.

(**Key words:** skim milk retentate powder, heat treatment, pH, functional properties)

### INTRODUCTION

Retentate from ultrafiltered milk can be dried and used to make cheese (23), fermented milk products (22, 26), or other products based on milk protein (16). During UF of milk, proteins are retained in the retentate, and lactose and mineral constituents pass into the permeate. The true digestibility, biological value, and net protein utilization of low lactose milk powder produced from UF retentate are better than those of regular skim milk powder (7). Also, when membrane filtration is used to concentrate protein and to remove lactose from milk prior to spray drying, some functional proper-

ties, such as foaming capacity of the retentate powder, are improved (25).

The unique physicochemical characteristics of UF retentate powder are desirable in food (21). Emulsifying capacity, gel water-holding capacity, acid gel strength, and foaming capacity of high protein milk powder may influence processing and quality of dairy products such as cheese, yogurt, ice cream, and dairy-based desserts.

Heat treatment of UF skim milk can result in powders with desirable functional properties (2). The extent of heat treatment can be altered to prepare powders for different products [e.g., low heat powders in yogurt, ricotta cheese, and rennet casein and high heat powder in bakery products (20, 27)]. Heat treatment of whey proteins or  $\beta$ -lactoglobulin can result in products for which the rheological and textural properties range from soft curd to hard gels (6, 14, 21, 24, 30).

When UF retentate was modified by heat treatment, the functional properties (i.e., water absorption, whippability, foaming capacity, and acid gel strength) of the resulting high protein powder improved as protein content increased (2). In UF retentate, rennin action strongly depends on the severity of heat treatment. Curd consistency and syneresis are influenced by both heat treatment and protein concentration (3). Syneresis decreases as protein concentration increases and is strongly affected by pasteurization and UHT treatment. High protein retentate powder has low bacterial count and low acidity and retains excellent flavor when stored for up to 4 wk at five temperatures ranging from 4 to 60°C. Foaming and heat stability were also better than those of commercial skim milk powder (18).

The functional behavior of milk can be altered by heat treatment (not as extensive as in evaporation) and pH adjustment. The objective of this study was to evaluate how heat treatments and pH changes prior to drying affected

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the functional properties of spray-dried skim milk retentate powders.

## MATERIALS AND METHODS

### Production of UF Retentate Powders

Skim milk retentate powders were prepared as described previously (9). A raw skim milk retentate with 20% solids was divided into portions and heated prior to spray drying. The four heat treatments were no heat, 65°C for 30 min, 75°C for 28 s, and 85°C for 28 s. The pH was adjusted to 6.4, 6.7, and 7.0. Three different lots of milk were used to prepare three replicates for each treatment.

### Analytical Procedures

**Gel Water-Holding Capacity.** A method developed to determine the water-holding capacity of gels of reconstituted milk powders (Y. A. El-Samragy and C. L. Hansen, 1993, unpublished data) was used to measure the gel water-holding capacity of the retentate powders. Retentate powder was reconstituted in .01 M CaCl<sub>2</sub> to 12% total solids (1) and held for 20 h at 5°C for equilibrium (10). The reconstituted milk was then held at 30°C for 30 min. A purified calf rennet solution (The New Zealand Cooperative Rennet Co., Ltd., Eltham, New Zealand) was diluted with distilled water to nominal clotting activity of 100 rennet units and maintained at 2°C for no longer than 1 h prior to use. Two milliliters of diluted rennet were added to 50 ml of sample in a 250-ml beaker and kept at 30°C in a water bath for 30 min. The curd that formed was then cut around the edge and into four quarters with a thin, straight spatula. Whey was drained by pouring curd into a glass funnel containing a number 1 filter paper (12.5-cm diameter; Whatman, Maidstone, England) set on a 100-ml measuring cylinder. The gel water-holding capacity was calculated after 1 h of filtration as follows: gel water-holding capacity (milliliters) = 50 - drained whey volume.

**Acid-Gel Strength.** Retentate powders were reconstituted in distilled water (10% total solids), and those solutions were acidified with D-glucono- $\delta$ -lactone with the procedure described by Harwalkar et al. (15). One hundred milliliters of reconstituted retentate powder in

a 150-ml beaker were heated to 90°C for 5 min. One gram of crystalline D-glucono- $\delta$ -lactone was quickly stirred into the mixture, which was then held quiescently at 90°C for 5 min during which time gelation occurred. The gels were cooled to 5°C and held at this temperature for 2 h prior to testing. The beaker was placed on a digital electronic balance (Denver Instrument Co., Denver, CO), and a motor-driven 1.5 cm cylindrical steel rod was lowered at a rate of 1 cm/min (19). The acid gel strength was the force required to break that gel, i.e., the highest recorded balance in grams.

**Emulsifying Capacity.** The emulsifying capacity was estimated using the procedures outlined by Satterlee and Free (33) and Webb et al. (36). Corn oil was delivered continuously and mixed into the reconstituted sample (1% total solids). The delivery of oil was stopped at the point of emulsion collapse, as measured by electrical resistance; the amount of oil at that point was the emulsifying capacity.

**Foaming Capacity.** The method described by Phillips et al. (32) was used to determine the foaming capacity of the retentate powder. The overrun measurement was made after 10 min of whipping using 75 ml of reconstituted sample (5% total solids).

**Heat Stability.** The retentate powders were tested for heat stability as described by Hall and Hedrick (13) and Jimenez-Flores (17); 20-ml samples reconstituted to 10% total solids were added to 50-ml test tubes, covered with aluminum foil, and placed in a boiling water bath for 120 min. Observations for coagulation were made every 5 min without retrieving the tubes from the water.

### Statistical Analysis

The experimental variables were studied by ANOVA using a split-plot randomized block design, where heat treatment was the whole plot treatment, pH adjustment was the subplot treatment, and the three lots of milk were the blocks. The least significant difference test was used to calculate significant differences.

## RESULTS AND DISCUSSION

An ANOVA table for the experimental results is given as Table 1. The interaction

TABLE 1. Analysis of variance: some functional properties of skim milk retentate powders.

Source of variance	df	Mean squares			
		Gel water-holding capacity	Acid gel strength	Emulsifying capacity	Foaming capacity
Replicates	2	1.083	2.8	.2	13,556
Heat treatment	3	92.741***	757.8***	10,714.3***	10,548
Error (a)	6	1.824	5.1	6.7	8201
pH	2	438.583***	8129.4***	17.0*	18,561*
pH × Heat treatment	6	6.769*	236.5***	22.1***	14,194*
Error (b)	16	1.931	2.9	3.3	4054
Total	35				

\**P* < .05.\*\*\**P* < .001.

effects of heat treatment and pH adjustment on all functional properties were significant to varying degrees (Table 1).

Data presented in Table 2 indicate the effects of heat treatment and pH adjustment on functional properties of reconstituted skim

milk retentate powders. The gel water-holding capacity was significantly higher for the heated retentate than for the unheated retentate at the same pH. These results are consistent with those of Casiraghi et al. (3), who also found that heat treatment and protein concentration in

TABLE 2. Means for some functional properties of skim milk retentate powders.<sup>1</sup>

pH	Gel water-holding capacity	Acid gel strength	Emulsifying capacity	Foaming capacity
	(ml)	(g)	(ml of oil)	(% overrun)
No heat treatment				
6.4	24 <sup>c,z</sup>	135 <sup>a,x</sup>	148 <sup>a,x</sup>	1056 <sup>b,x</sup>
6.7	27 <sup>b,y</sup>	122 <sup>b,x</sup>	146 <sup>a,x</sup>	1281 <sup>a,x</sup>
7.0	37 <sup>a,z</sup>	95 <sup>c,x</sup>	140 <sup>b,x</sup>	1104 <sup>b,x</sup>
65°C for 30 min				
6.4	28 <sup>c,y</sup>	133 <sup>a,x</sup>	140 <sup>a,y</sup>	1036 <sup>a,x</sup>
6.7	32 <sup>b,x</sup>	122 <sup>b,xy</sup>	137 <sup>ab,y</sup>	1072 <sup>a,y</sup>
7.0	41 <sup>a,y</sup>	73 <sup>c,y</sup>	134 <sup>b,y</sup>	1102 <sup>a,x</sup>
75°C for 28 s				
6.4	27 <sup>c,y</sup>	136 <sup>a,x</sup>	88 <sup>a,z</sup>	1027 <sup>b,x</sup>
6.7	32 <sup>b,x</sup>	97 <sup>b,y</sup>	89 <sup>a,z</sup>	1145 <sup>a,x</sup>
7.0	39 <sup>a,yz</sup>	75 <sup>c,y</sup>	91 <sup>a,z</sup>	1075 <sup>ab,x</sup>
85°C for 28 s				
6.4	34 <sup>b,x</sup>	120 <sup>a,y</sup>	74 <sup>a,x</sup>	1138 <sup>a,x</sup>
6.7	33 <sup>b,x</sup>	94 <sup>b,y</sup>	75 <sup>a,x</sup>	1065 <sup>a,y</sup>
7.0	43 <sup>a,xy</sup>	74 <sup>c,y</sup>	76 <sup>z,x</sup>	1067 <sup>a,x</sup>
LSD <sup>a,b,c</sup>	2.4	2.9	3.1	110
LSD <sup>x,y,z</sup>	2.5	3.5	3.9	138

<sup>a,b,c</sup>Least significant difference for the difference between two pH at same heat treatment; means not sharing the same superscript are significantly different (*P* < .05).

<sup>x,y,z</sup>Least significant difference for the difference between two heat treatments at same pH; means not sharing the same superscript are significantly different (*P* < .05).

<sup>1</sup>Mean of three replicate trials.

combination affected the curd consistency and syneresis of the rennet-coagulated UF reconstituted retentate powders.

Acid gel strength was altered significantly ( $P < .05$ ) by pH change within each heat treatment. Acid gel strength decreased significantly ( $P < .05$ ) for the heat treatment at 85°C for 28 s compared with that for the first three heat treatments at pH 6.4. At pH 6.7, the effect of the first two heat treatments differed significantly ( $P < .05$ ) compared with that of the last two heat treatments. Acid gel strength of the unheated samples was significantly higher ( $P < .05$ ) than that of the samples heated at the last three heat treatments when the pH was adjusted to 7.0 (Table 2).

Emulsifying capacity decreased significantly ( $P < .05$ ) when the pH increased to 7.0 in unheated samples. Also, pH change affected emulsifying capacity when heat treatment at 65°C for 30 min was applied. Change in pH did not significantly change emulsifying capacity ( $P > .05$ ) of the samples heated at 75 or 85°C for 28 s prior to spray drying. At each pH, heat treatment decreased emulsifying capacity significantly ( $P < .05$ ) as the temperature of the heat treatment increased (Table 2). Others (16, 28, 31, 35) found that the type of protein affected the ability to form an emulsion.  $\alpha$ -Lactalbumin is more easily denatured and apparently interferes with the emulsifying capacity of the  $\beta$ -lactoglobulin.

Change in pH caused foaming capacity to change in unheated samples and when heat treated at 75°C for 28 s. At each pH, the effect of heat treatment was not significant ( $P < .05$ ), except at pH 6.7, when samples were heated at 65°C for 30 min and at 85°C for 28 s ( $P < .05$ ) (Table 2), probably because of changes in the functional behavior of protein [e.g., solubilization, aggregation, and unfolding (5, 29, 34)]. These pH effects have also been noted in whey protein concentrate powders (12), delactosed high protein milk powders (25), and other high protein products (32). A foam consists of a gas bubble phase surrounded by a liquid phase (11), which results in a large gas-liquid interface. A protein film must be viscous and elastic to create a stable form (4, 20).

All reconstituted retentate powders remained stable when heated at 100°C for 2 h.

## CONCLUSIONS

Using UF to concentrate skim milk to 20% total solids, followed by application of a heat treatment and control of pH before spray drying, makes it possible to produce high protein skim milk retentate powder with specific functional properties. Thus, retentate powders can be modified to be more beneficial for use in food products.

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## REFERENCES

- Berridge, N. J. 1952. Some observations on the determination of the activity of the rennet. *Analyst* 77:57.
- Butterick, J. K., and J. J. Higgins. 1982. Functional properties of milk powders from ultrafiltered skim milk. Page 438 in *Brief Commun.*, 21th Int. Dairy Congr., Moscow. Book 2. Mir Publ., Moscow, USSR.
- Casiraghi, E., M. Lucisano, and C. Peri. 1989. Rennet coagulation of milk retentate. 2. The combined effect of heat treatments and protein concentration. *J. Dairy Sci.* 72:2457.
- Cherry, J. P., and K. H. McWatters. 1981. Whippability and aeration. Page 149 in *Protein Functionality in Foods*. J. P. Cherry, ed. Am. Chem. Soc. Symp. Ser., Am. Chem. Soc., Washington, DC.
- de Wit, J. N. 1981. Structure and functional behavior of whey proteins. *Neth. Milk Dairy J.* 35:47.
- de Wit, J. N., and G. Klarenbeek. 1988. Page 211 in *Milk Proteins: Nutritional, Clinical, Functional and Technological Aspects*. C. A. Barth and E. Schlimme, ed. Steinkopff Verlag Darmstadt, Germany.
- Edelsten, D., M. Meersohn, P. Friis, E. W. Neilsen, K. L. Sorensen, and E. Gudmen-Hoyer. 1983. Production of skim milk powder with lactose content reduced by ultrafiltration. *Milchwissenschaft* 38:261.
- Reference deleted in proof.
- El-Samragy, Y. A., C. L. Hansen, and D. J. McMahon. 1992. Production of ultrafiltered skim milk retentate powder. 1. Composition and physical properties. *J. Dairy Sci.* 76:388.
- Ernstom, C. A. 1954. Milk-clotting enzymes and their action. Page 662 in *Fundamentals of Dairy Chemistry*. 2nd ed. B. H. Webb, A. H. Johnson, and J. A. Alford, ed. AVI Publ. Co., Westport, CT.
- German, J. B., T. E. O'Neill, and J. E. Kinsella. 1985. Film forming and foaming behavior of food proteins. *J. Am. Oil Chem. Soc.* 62:1358.
- Hagget, T.O.R. 1976. The whipping, foaming and gelling properties of whey protein concentrates. N.Z.

- J. Dairy Sci. Technol. 11:244.
- 13 Hall, C. W., and T. L. Hedrick. 1966. *Drying Milk and Milk Products*. AVI Publ. Co., Westport, CT.
- 14 Harwalkar, V. R., and M. Kalab. 1985. Microstructure of isoelectric precipitates from  $\beta$ -lactoglobulin solutions heated at various pH levels. *Milchwissenschaft* 40:665.
- 15 Harwalkar, V. R., M. Kalab, and D. B. Emmons. 1977. Gels prepared by adding D-glucose- $\delta$ -lactone to milk at high temperature. *Milchwissenschaft* 32:400.
- 16 Hung, S. C., and J. F. Zayas. 1991. Emulsifying capacity and emulsion stability of milk proteins and corn germ protein flour. *J. Food Sci.* 56:1216.
- 17 Jimenez-Flores, R. 1984. Physical and functional properties of ultrafiltered retentate powders. M.S. Thesis, Cornell Univ., Ithaca, NY.
- 18 Jimenez-Flores, R., and F. V. Kosikowski. 1986. Properties of ultrafiltered skim milk retentate powders. *J. Dairy Sci.* 69:329.
- 19 Kalab, M., P. W. Voisey, and D. B. Emmons. 1971. Heat-induced milk gels. II. Preparation of gels and measurement of firmness. *J. Dairy Sci.* 54:178.
- 20 Kinsella, J. 1971. Chemistry of dairy powders with reference to baking. *Adv. Food Res.* 19:148.
- 21 Kinsella, J. 1988. Page 179 in *Milk Proteins: Nutritional, Clinical, Functional and Technological Aspects*. C. A. Barth and E. Schlimme, ed. Steinkopff Verlag Darmstadt, Germany.
- 22 Kosikowski, F. V. 1979. Low lactose yogurts and milk beverages by ultrafiltration. *J. Dairy Sci.* 62:41.
- 23 Le Graet, Y., and J. L. Maubois. 1979. Fabrication de fromages à pâte fraîche à partir de poudres de retentat ou de prefromage. *Rev. Laitière Française* 373:23.
- 24 Mehta, R. S. 1980. Milk processed at ultra-high-temperature. A review. *J. Food Prot.* 43:212.
- 25 Mistry, V. V., and H. N. Hassan. 1991. Delactosed, high milk protein powder. 2. Physical and functional properties. *J. Dairy Sci.* 74:3716.
- 26 Mistry, V. V., and H. N. Hassan. 1991. Manufacture of nonfat yogurt from a high milk protein powder. *J. Dairy Sci.* 75:947.
- 27 Modler, H. W. 1985. Functional properties of nonfat dairy ingredients—a review. Modification of products containing casein. *J. Dairy Sci.* 68:2195.
- 28 Mohanty, B., D. M. Mulvihill, and P. F. Fox. 1988. Emulsifying and foaming properties of acidic casein and sodium caseinate. *Food Chem.* 28:17.
- 29 Morr, C. V. 1985. Manufacture, functional properties of reference whey protein concentrate. *J. Food Sci.* 50:1406.
- 30 Mulvihill, D. M., and J. E. Kinsella. 1987. Gelation characteristics of whey proteins and  $\beta$ -lactoglobulin. *Food Technol.* 41:102.
- 31 Pearce, K. N., and J. E. Kinsella. 1978. Emulsifying properties of protein: evaluation of a turbidimetric technique. *J. Agric. Food Chem.* 26:716.
- 32 Phillips, L. G., Z. Haque, and J. E. Kinsella. 1987. A method for the measurement of foam formation and its stability. *J. Food Sci.* 52:1074.
- 33 Satterlee, L. D., and B. Free. 1973. Utilization of high protein tissue powders as a binder/extender in meat emulsions. *J. Food Sci.* 38:306.
- 34 Walstra, P., and R. Jenness. 1984. *Dairy Chemistry and Physics*. John Wiley & Sons, Inc., New York, NY.
- 35 Wang, J. C., and J. E. Kinsella. 1976. Functional properties of novel proteins: alfalfa leaf protein. *J. Food Sci.* 41:286.
- 36 Webb, N. B., F. J. Ivey, H. B. Craig, V. A. Jones, and R. J. Monroe. 1970. The measurement of emulsifying capacity by electrical resistance. *J. Food Sci.* 35:501.