Rennet Coagulation of Milk Retentates. 1. Effect of Thermal and Mechanical Stresses Associated with Ultrafiltration

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ABSTRACT

Effects of UF on clotting kinetics, curd hardening, and whey syneresis of raw and pasteurized milk were studied. Milk was subjected to thermal and mechanical stresses similar to those occurring in a UF plant. To isolate the effects of milk recirculation from those due to protein concentration, a UF batch loop system similar to an industrial system was used. Recirculation was obtained returning both retentate and permeate to the feed tank.

Mechanical and thermal stresses associated with UF treatment caused a slight reduction of curd hardness and a decrease in whey separation rate. These effects, however, were less marked than those induced by pasteurization or by an increase in protein concentration, both of which are associated with practical UF of milk.

INTRODUCTION

Clotting kinetics, curd hardening, and whey syneresis are all affected by UF of milk (5, 6, 7, 9, 11). Ultrafiltration changes concentration of milk constituents and its physical properties (especially viscosity). The thermal and mechanical conditions of UF may also negatively affect the functionality of milk constituents such as proteins.

High flow rates, temperature (50°C), and shear stresses associated with recirculation of milk retentates through UF equipment may affect fat globule dispersion and casein micelles aggregation (8). These are critical factors in milk coagulation. There is little experimental evidence on the extent "side effects" of UF on milk properties. The choice of temperature, recirculation rate, and pump type (centrifugal or positive rotating) vary among UF equipment users and manufacturers.

The aim of this study was to investigate the effects of the thermal and mechanical stresses occurring during UF on milk clotting behavior. To discriminate these effects from those due to protein concentration, both retentate and permeate were returned to the feed tank. Milk was recirculated in a UF batch loop under conditions similar to those generally adopted in industrial plants. Recirculation was continued longer than required for concentration. After UF, the milk was evaluated on the basis of rennet coagulation kinetics, curd hardening, and whey syneresis. Both raw and pasteurized milk were tested to determine whether UF thermal and mechanical stresses are significant in comparison with effects of milk pasteurization.

MATERIALS AND METHODS

Samples

Raw and pasteurized whole milk were obtained from a dairy company near Milan, Italy. Milk was produced by different cow breeds and sampled from tanks containing approximately 250,000 L. Pasteurization conditions were 75°C for 20 s. Raw and pasteurized milks were each separated into two batches. Control batches were gently stirred at 50°C for 3 h in a water bath. The others were recirculated in a UF system at 50°C for 3 h. Sodium azide (.01% wt/vol) was added to each sample before any treatment in order to prevent the growth of bacteria.

Equipment

A laboratory-scale UF system, UF 35-2.25 (DDS, RO-Division, Nakskov, Denmark) with

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DDS-GR membranes (cut off 25,000) was used. Both retentate and permeate were returned to the feed tank. The operating conditions were: temperature 50°C, inlet pressure 3.8 atm, outlet pressure 3.2 atm. A flow rate of 6000 L/h in the recirculation loop was maintained by a centrifugal pump. Five additional samples of raw and pasteurized milk, collected from the industry where pasteurization was performed, were also analyzed to estimate the range of natural variance of milk properties.

Analytical Methods

Dry matter, ash, and fat were determined according to AOAC methods 16.032, 16.020, and 16.065, respectively (1). Total calcium was measured by atomic absorption (wavelength 422.7) with a Pye Unicam SP9 spectrometer (Pye Unicam Ltd., Cambridge, England) according to Brule et al. (3). Nitrogen fractions (casein, soluble proteins, proteose peptone, NPN) were determined using a semi-micro-Kjeldahl procedure (13). These were expressed as protein using N to protein conversion factor of 6.38. Nitrogen fractions were evaluated both on milk and on whey collected after evaluation of syneresis.

Evaluation of Coagulation Behavior

Clotting time (CT) was determined using a modified Berridge method (2). Milk (25 ml) was placed in test tubes with .5 ml of rennet solution. The following calf rennet (Chr. Hansen's, Milano, Italy; 1:10,000) concentrations in milk were used: .025, .05, .06, .1, .2, and .4% (wt/vol). After stirring, the test tubes were placed in a 38°C water bath and slowly rotated with an inclination of about 30°. Clotting time was the time to the first visible clots in the sample. Coagulation lines were then obtained by plotting the reciprocal of rennet concentration versus CT and fitting the experimental points to a straight line as designated by Qvist (12).

Evaluation of Syneresis

Curd syneresis was evaluated according to the method of Marshall (10). Coagulum was formed in 100-ml plastic beakers by addition of 1 ml of 2.5% rennet solution (1:10,000) to 50 ml of milk and maintained at 38°C. One hour after rennet addition the curd was cut crosswise and around the sides of the becker with a spatula. After cutting, a plastic grid was placed on curd surface. This restrained the curd while allowing the whey to be poured off. Syneresis was measured by weighing the whey quantity removed at various intervals after cutting.

Rheological Evaluation of Curds

Curd was prepared by adding 1 ml of 5% (vol/vol) rennet solution (1:10,000) to 100 ml of milk. After rennet addition, samples were kept in a thermostatic bath at 38°C until tested. The rheological properties of the curd were evaluated by uniaxial compression tests conducted with an Instron Universal Testing Machine Model 3140 (Instron Ltd. High Wycombe, England). Each test was replicated twice for each sample at a deformation of 10% with a crossbar speed of 200 mm/min. The elastic modulus E (N/m^2) was reported as a function of time elapsed since rennet addition.

RESULTS AND DISCUSSION

The analytical composition of the raw and pasteurized milk samples are given in Tables 1

TABLE 1. Proximate analysis of raw and pasteurized milk.

Milk	pH	Acidity	Total solids	Fat	Ash	Total N[2]	NPN	Calcium
		(°SH[1]/100 ml)			(g/100 g)			(mg/L)
Raw	6.68	6.35	11.04	3.3	.72	3.18	.19	115
Pasteurized	6.71	6.80	11.46	3.3	.72	3.01	.17	126

[1]Soxhlet Henkel degrees.

[2]Nitrogen expressed as N × 6.38.

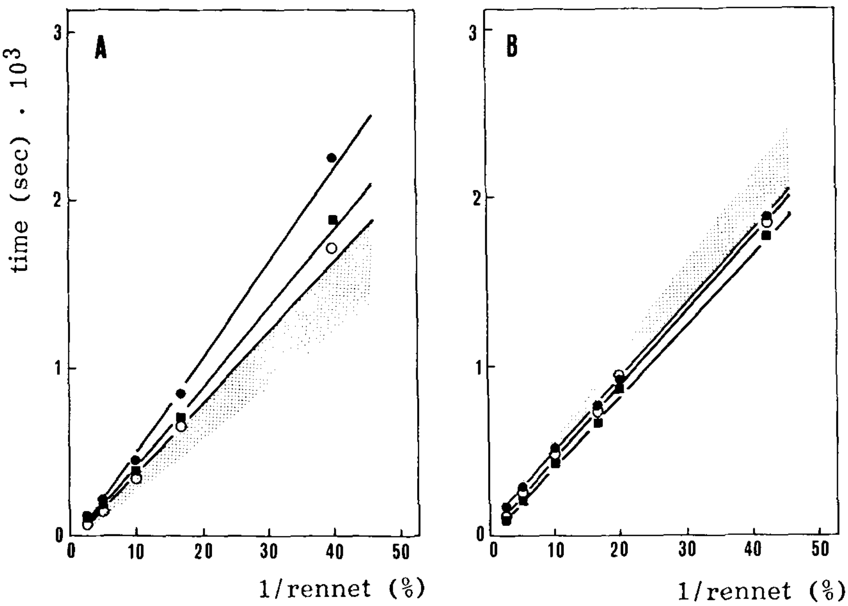

Figure 1. Coagulation lines of raw (A) and pasteurized (B) milk. ○ Untreated; ■ held at 50°C for 3 h; ● recirculated in UF plant at 50°C for 3 h; shaded area indicates range of variation for five samples of raw and pasteurized milk belonging to different batches.

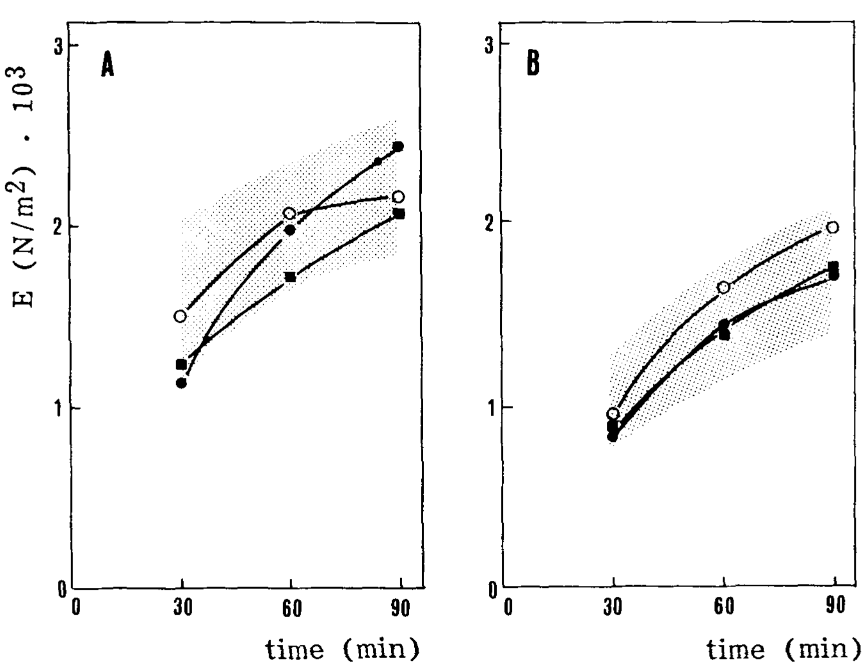

Figure 2. Elastic moduli of raw (A) and pasteurized (B) milk as a function of time since rennet addition. ○ Untreated; ■ held at 50°C for 3 h; ● recirculated in UF plant at 50°C for 3 h; shaded area indicates range of variation for five samples of raw and pasteurized milk belonging to different batches.

TABLE 2. Nitrogen fraction of various treated milk and whey.

Item	Total N[1]		Casein		Soluble protein		Proteose peptone		NPN	
	(g/100 g)	(%)	(g/100 g)	(%)	(g/100 g)	(%)	(g/100 g)	(%)	(g/100 g)	(%)
Raw milk	3.18	100	2.46	77.4	.38	11.9	.15	4.8	.19	5.9
Whey	.98	100	.06	6.1	.46	46.9	.22	22.4	.24	24.6
Raw milk 50°C × 3 h	3.18	100	2.39	75.2	.40	12.6	.20	6.3	.19	5.9
Whey	1.02	100	.03	2.9	.50	49.0	.24	23.6	.25	24.5
Raw milk UF 50°C × 3 h	3.18	100	2.40	75.5	.39	12.2	.21	6.7	.18	5.6
Whey	1.05	100	.05	4.7	.53	50.5	.19	18.1	.28	26.7
Pasteurized milk	3.01	100	2.43	80.7	.25	8.4	.16	5.3	.17	5.6
Whey	.82	100	.08	9.7	.27	32.9	.22	26.8	.25	30.6
Pasteurized milk 50°C × 3 h	3.07	100	2.46	80.3	.24	7.7	.20	6.4	.17	5.6
Whey	.84	100	.10	11.9	.24	28.6	.24	28.6	.26	30.9
Pasteurized milk UF 50°C × 3 h	3.07	100	2.48	80.8	.25	8.1	.16	5.2	.18	5.9
Whey	.89	100	.13	14.6	.31	34.8	.21	23.7	.24	26.9

[1]Nitrogen and protein fractions expressed as N × 6.38.

and 2. Pasteurized milk had a higher percentage of the "casein" fraction than raw milk because of complexing of β-lactoglobulin with κ-casein (14). Consequently, whey obtained from pasteurized milk had a protein content lower than that obtained from raw milk. Recirculation of milk through the UF system caused a slight increase in total protein content of whey obtained from both raw and pasteurized milk.

No other variations in milk chemical composition were observed. The effects of UF recirculation on functionality of the milk constituents were quite evident. In particular, CT of raw milk was increased when milk was tempered at 50°C and, to a greater extent, by UF recirculation (Figure 1A). This effect was not observed when pasteurized milk was used (Figure 1B). This implies that the thermal and mechanical stresses of UF that slow down the casein aggregation process are overshadowed by the effect of prior pasteurization.

The UF recirculation of raw and pasteurized milk at 50°C slightly reduced elastic moduli of their curds (Figure 2). However, these differences were within the range of natural variation of milk. In each case, the elastic moduli of curd obtained from pasteurized milk were lower than those obtained from raw milk.

The kinetics of whey syneresis was the parameter most sensitive to the thermal and mechanical history of the samples. A comparison of the curves in Figure 3, parts A and B, demonstrates that syneresis of curd from pasteurized milk was much slower than curd from raw milk. Both the thermal and mechanical stressed connected with UF further slow the kinetics of whey separation.

CONCLUSION

The conditions under which UF is conducted have measurable, but limited, effects on coagulation and syneresis phenomena. In other experiments (4), type of pump (centrifugal or monopump), membrane system (flat sheets or tubulars) and temperature (15 or 50°C) do not change CT or syneresis.

Any modification in milk properties attributed to UF design or operative conditions is outweighed by the two factors that are an integral part of commercial UF processes: pasteurization and increase in protein concentration.

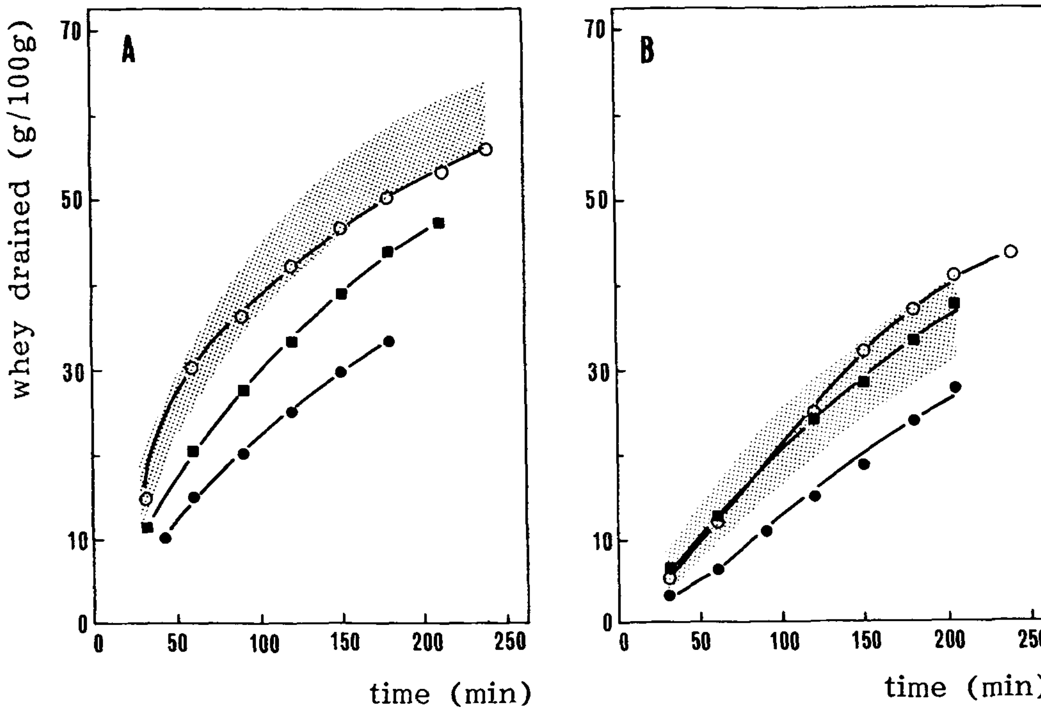

Figure 3. Amount of whey release as a function of time after rennet addition. Curds obtained from raw milk (A) or from pasteurized milk (B). o Untreated milk; ■ milk held at 50°C for 3 h; ● milk recirculated in UF plant at 50°C for 3 h; shaded area indicates range of variation for five samples of raw and pasteurized milk belonging to different batches.

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