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RAPID COMMUNICATION

Physicochemical characteristics and rennet coagulation time of ultrafiltered goat milk

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Goat skim milk was concentrated by ultrafiltration (UF) to volume concentration ratios (VCR) of 2, 3, 4 and 5. Gross composition, titratable acidity, pH, nitrogen distribution, percentage retention and recovery of components and rennet coagulation time (RCT) of skim milk during UF processing were studied. During UF of goat skim milk, all fat, CN, WPN, 19% of NPN, 78.1% of TS, 78.6% of ash and 3.5% of lactose were retained in 5-VCR retentate. Recovery of these components were 14.7, 53, 48, 17 for NPN, TS, ash, lactose and 100% for fat, WPN or CN, respectively. For TN, TS, ash, NPN and lactose, retention was increased by increasing the VCR. The titratable acidity was increased from an initial value of 0.14 to 0.38% in 5-VCR retentate, whereas pH decreased from 6.58 to 6.50. The RCT decreased as the protein concentration of the milk increased, but the precise influence of protein concentration decreased at higher levels of rennet. © 1998 Elsevier Science Ltd. All rights reserved

INTRODUCTION

Application of ultrafiltration (UF) in the dairy industry has been reviewed by several authors (Glover, 1985; Cheryan, 1986; Pal and Cheryan, 1987; El-Gazzar and Marth, 1991; Renner and Abd El-Salam, 1991). The use of UF to concentrate and separate cows' milk constituents is widely recognized and could have far-reaching effects, especially for milk destined for cheesemaking (Moubois and Mocquot, 1975; Zall, 1984; Kosikowski, 1986; Lelievre and Lawrence, 1988), production of low-sodium and low-lactose milk products (Kosikowski, 1979, Kosikowski, 1983; Edelstein *et al.*, 1983), and increased utilization of whey for human food (Renner and Abd El-Salam, 1991). During ultrafiltration of milk, non-protein nitrogen and soluble components, such as lactose, salts and some vitamins, pass through the membrane, whereas milk fat, proteins and colloidal salts are retained (Glover, 1971; Peri *et al.*, 1973; Covacevich and Kosikowski, 1977; Green *et al.*, 1984; Premaratne and Cousin, 1991; Bastian *et al.*, 1991). However, the changes that occur in the physicochemical characteristics of milk during UF have to be considered before retentates are used in the manufacture of various dairy products.

The growing use of UF in the dairy industry, especially in the area of cheesemaking, promises to dramatically change the technology of cheese manufacture (Lelievre and Lawrence, 1988; Renner and Abd El-Salam, 1991). Since the UF retentate has higher dry matter and protein contents, as well as an increased ratio of protein to dry matter compared to native milk, its properties obviously differ from those of the milk from which it was prepared (Mehaia and Cheryan, 1983*a*). In the case of cows' milk, several workers (Garnot and Corre, 1980; Reuter *et al.*, 1981; Mehaia and Cheryan, 1983*a*; Lucisano *et al.*, 1985) reported that the concentration of milk by UF caused a decrease on the rennet coagulation time. On the other hand, other workers (Culioli and Sherman, 1978: Schmutz and Puhan, 1978) reported that the coagulation time increased with increasing protein content. Dalgleish (1980) observed that the coagulation time is relatively unaffected by the concentration of milk by UF. Sharma *et al.* (1993) reported that the coagulation time decreased with an increase in milk concentration when milk pH was unadjusted, but remained unaffected when pH was adjusted (6.8–6.0) by adding lactic acid. Mehaia (1994) reported that the rennet coagulation time of ultrafiltered camel skim milk decreased as the protein concentration increased.

Although data on the physicochemical characteristics and rennet coagulation of ultrafiltered cow milk have

been reported by several workers (Glover, 1971; Peri *et al.*, 1973; Covacevich and Kosikowski, 1977; Green *et al.*, 1984; Renner and Abd El-Salam, 1991; Premaratne and Cousin, 1991; Bastian *et al.*, 1991), there is limited information on ultrafiltered goats' milk.

However, as UF is increasingly being considered and applied in dairy processing, there is a growing need for information on the physicochemical characteristics and rennet coagulation data for different kinds of milk, such as goats' milk. The objective of this study was to evaluate some physicochemical characteristics and rennet coagulation time of ultrafiltered goat milk.

MATERIALS AND METHODS

Preparation of milk for ultrafiltration

Raw goat (Jamunapri) milk was obtained from King Saud University Farm, Buriedah, Saudi Arabia. The milk was warmed to 30–33°C in a water bath and then skimmed, weighed, and pasteurized at 72°C for 20 s. Samples for chemical analyses were taken and refrigerated for subsequent analysis. The pasteurized skim milk was cooled to 50°C before ultrafiltrtion.

Ultrafiltration process

The bench-scale UF system consisted of a feed tank for holding the milk, a Masterflex peristaltic pump (Cole-Parmer Instrment Co., Chicago, IL, USA) for recycling milk, two pressure gauges to monitor inlet and outlet pressures, a hollow fibre UF module with a polysulphone membrane of 30 000 molecular weight cut-off (MWCO) (Model UFP-30-C-4) obtained from A/G Technology, Needham, MA, USA, and a container to collect and measure the permeate. The UF process was started by pumping the milk at 50°C through the membrane module while maintaining inlet and outlet pressures of 137 and 35 k Pa, respectively. Permeate volume was monitored continuously to determine reduction in milk volume to 2, 3, 4 and 5 volume concentration ratios (VCR). At different VCR, retentate and permeate samples were taken and refrigerated for subsequent analysis. As the concentration reached 5 (VCR = 5), the UF system was stopped and the retentate stored at 4°C. After each run, the membrane module was cleaned and sanitized according to the manufacturer's instructions and stored at 5°C. The experiment was repeated three times.

Rennet coagulation time (RCT)

Three levels of rennet were prepared using 1% (w/v) rennet power (Chr. Hansen's Lab. A/S, Copenhagen, Denemark). Each level of rennet was added to 10 ml of milk to give concentrations of 1, 2 and 4 mg 100 ml^{-1} of milk sample. Calcium chloride was added to each sample to give 0.02% (w/v) concentration. The coagulation time of milk samples was measured in triplicate at pH 6.6 and 35°C using a modified version of the Sommer-Matson rennet tester, as described by Mehaia and Cheryan (1983*b*). The pH of milk was adjusted to 6.6 by adding 40% (w/v) lactic acid, while stirring the samples with a magnetic stirrer.

Chemical analysis

Skim milk, retentate and permeate samples were analysed for total solids, fat and ash according to procedures outlined in AOAC (1980). Lactose was determined by difference. Nitrogen was determined by the standard micro-Kjeldahl method (AOAC, 1980). A nitrogen conversion factor of 6.38 was used to calculate protein content. Milk samples were fractionated for total nitrogen (TN) and non-casein nitrogen (NCN) by the method of Rowland (1938) with the following modification. Retentate samples were diluted before analysis with distilled water to VCR = 1. Non-protein nitrogen (NPN) was determined in the supernatants produced by addition of 40 ml 15% trichloroacetic acid to 10 ml milk or diluted retentate, as outlined by Cerbulis and Farrell (1975). Nitrogen fractions were calculated as follows: protein nitrogen (PN) = TN−NPN, casein nitrogen (CN) = TN−NCN, and whey protein nitrogen (WPN) = NCN−NPN. Titratable acidity was determined by titrating 10 g of sample with 0.1N NaOH to pink endpoint using phenolphthalein indicator (AOAC, 1980). The pH of milk samples was measured with an Orion pH meter (Orion Research Inc., Cambridge, MA, USA). Calcium in skim milk, retentate and permeate samples was analyzed by atomic absorption spectrophotometry (Model 1100B, Perkin-Elmer Corp., Analytical Instruments, Norwalk, CT, USA) according to the method reported by IDF (1988). All analyses of skim milk, retentate and permeate samples were performed in duplicate. All chemicals were of reagent grade.

Expression of results

The volume concentration ratio (VCR) was calculated as reported by Cheryan (1986), as follows:

$$\text{VCR} = \frac{\text{Initial volume of milk}}{\text{Concentrate (retentate) volume}} \quad (1)$$

The concentration factor (CF) of a component was calculated as reported by Green *et al.* (1984), as follows:

$$\text{CF} = \frac{\text{Concentration of a component in retentate}}{\text{Concentration of a component in the original milk}} \quad (2)$$

Table 1. Gross composition (mean ± SD)[a] of goats' skim milk during UF processing (g 100 g^{-1})

VCR[b]	Fat	Protein[c]	Lactose	Ash	Total solids
1	0.15 ± 0.05	3.80 ± 0.09	4.20 ± 0.09	0.80 ± 0.04	8.95 ± 0.26
2	0.31 ± 0.07	7.37 ± 0.12	4.02 ± 0.10	0.97 ± 0.04	12.67 ± 0.32
3	0.46 ± 0.10	10.5 ± 0.15	3.72 ± 0.11	1.30 ± 0.09	15.96 ± 0.36
4	0.60 ± 0.10	13.8 ± 0.12	3.70 ± 0.08	1.71 ± 0.08	19.81 ± 0.30
5	0.75 ± 0.10	17.4 ± 0.12	3.63 ± 0.12	1.91 ± 0.10	23.64 ± 0.41

[a]Means of duplicate analyses on each of three runs.
[b]VCR: volume concentration ratio.
[c]Protein: total nitrogen ×6.38.

The percentage retention was calculated according to Bastian *et al.* (1991), as follows:

$$\text{Retention } \% = \left(1 - \frac{(Y_p)/(\%\ \text{water}_p + Y_p)}{(Y_r)/(\%\ \text{water}_r + Y_r)}\right) \times 100 \quad (3)$$

where Y is the percentage of any component in retentate (r) or permeate (p). Because the concentration of components in the final retentate is important for cheese-making, Bastian *et al.* (1991) defined a new term, percentage recovery. This term is similar to percentage retention reported by Glover (1971) and is shown in eqn (4):

$$\text{Recovery } \% = \frac{\text{kg component in retentate}}{\text{kg component in original milk}} \times 100 \quad (4)$$

RESULTS AND DISCUSSION

Gross composition

Concentrations of fat, protein, lactose, ash and total solids during ultrafiltration of goats' skim milk are presented in Table 1. The concentrations (as % of dry matter) of protein, lactose and ash in goat skim milk during UF processing are shown in Fig. 1. Moreover, the changes in the composition of some constituents of skim milk during UF processing are expressed as the increase or decrease of the concentration factor of the individual components as a function of the VCR (Fig. 2). As the VCR of skim milk was increased by 2-, 3-, 4- and 5-fold, the concentrations of total solids, protein, fat and ash increased and the concentration of lactose decreased. The fat content of skim milk increased from an initial value of 0.15 to 0.75 g 100 g^{-1} in the 5-fold retentate, indicating a 5-fold increase in concentration (CF = 5.0). Similarly, protein content increased from 3.80 to 17.4, total solids from 8.95 to 23.64, and ash from 0.80 to 1.91 g 100 g^{-1}. The concentration factor was 4.57, 2.66 and 2.38 for protein, total solids and ash, respectively. The increase of the above components was proportional to the concentration factor of the retentates, indicating the loss of small molecular weight components such as lactose, soluble salts, vitamins and NPN. When the composition of skim milk retentate was evaluated as a function of VCR during UF, there was a great increase in the protein content (as % of dry matter), a corresponding decrease in the lactose concentration and a smaller decrease in the percentage of ash in dry matter (Fig. 1). These observations were comparable with those reported for cows' milk by Ernstrom *et al.* (1980), Green *et al.* (1984), Srilaorkul *et al.* (1989), Premaratne and Cousin (1991) and St-Gelais *et al.* (1992) and for camel skim milk by Mehaia (1996) and for buffalo skim milk by Patel and Mistry (1997). The lactose concentration in goat skim milk was reduced by UF from an initial value of 4.20 to 3.63 g 100 g^{-1}, and the concentration factor

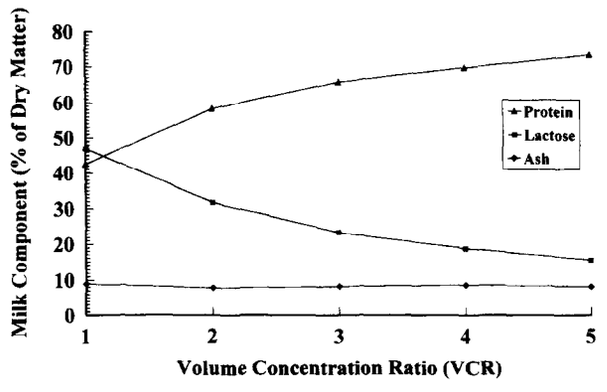

Fig. 1. Concentration (as % of dry matter) of protein, lactose and ash of goat skim milk depending on the volume concentration ratio during ultrafiltration.

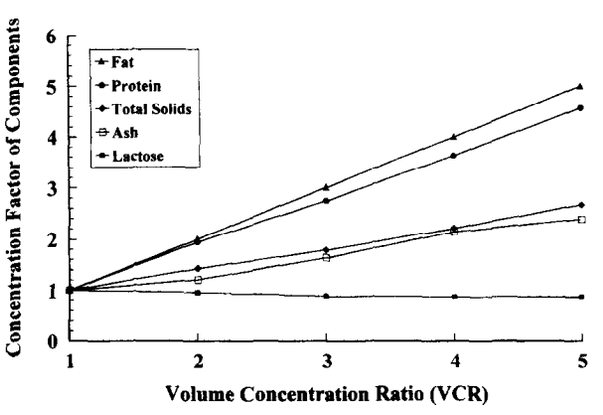

Fig. 2. Concentration factor (CF) of some constituents of goat skim milk as a function of volume concentration ratio.

was reduced from an initial value of 1.0 to 0.86 in the 5-VCR retentate. Because lactose is present in a free state in milk and has a molecular weight much lower than the MWCO of the UF membrane, it permeates freely (Premaratne and Cousin, 1991). The decrease in lactose concentration closely followed the increase in VCR. Similar observations were observed with cows' skim milk (Premaratne and Cousin, 1991; St-Gelais *et al.*, 1992), with camel skim milk (Mehaia, 1996) and with buffalo skim milk (Patel and Mistry, 1997).

Titratable acidity and pH

Typical changes in the titratable acidity and pH of goat skim milk during UF processing are shown in Fig. 3. The titratable acidity was increased from an initial value of 0.14 to 0.38% in the 5-fold retentate, whereas the pH decreased from 6.58 to 6.50. Similar observations were reported for camel skim milk by Mehaia (1994) and for buffalo skim milk by Hofi *et al.* (1982). Johnson (1978) reported that milks with a high acidity are usually high in total solids, and Jenness *et al.* (1978) reported that concentration of milk reduced its pH. Jenness and Patton (1959) reported that the effect of milk concentration on the pH and acidity is due to increase of milk solid not fat and shifts in distribution of calcium and phosphate between dissolved and colloidal states. Brule *et al.* (1974) reported that the concentration of milk by ultrafiltration increased the colloidal Ca and P proportionately to the amount of protein. They also stated that the amount of soluble Ca, and consequently the amount of colloidal Ca, was chiefly dependent on physicochemical characteristics of the aqueous phase of milk. Milk proteins and insoluble salts of calcium and phosphates exert a buffering effect. As these components are concentrated by UF, the buffer capacity of retentate is increased (Brule *et al.*, 1974; Covacevich and Kosikowski, 1979; Mistry and Kosikowski, 1984; Srilaorkul *et al.*, 1989).

Nitrogen distribution

Changes in the nitrogen distribution in goat skim milk during UF processing are presented in Table 2. The concentrations of PN, CN and WPN were increased from an initial value of 535, 444 and 91 mg $100\,g^{-1}$ to 2685, 2230 and 455 mg $100\,g^{-1}$ in the 5-fold retentate, respectively, indicating a 5-fold increase in concentration. However, the NPN concentration of goats' skim milk was almost constant during UF processing, whereas the concentration factor decreased from an initial 1 to 0.73 in the 5-VCR retentate.

Table 2. Nitrogen distribution[a] of goats' skim milk during UF processing (mean ± SD, mg $100\,g^{-1}$)[b]

VCR[c]	TN	PN	NPN	CN	WPN
1	595 ± 21	535 ± 20	60 ± 8	444 ± 12	91 ± 8
2	1156 ± 26	1098 ± 21	58 ± 10	911 ± 15	187 ± 9
3	1643 ± 19	1585 ± 18	58 ± 8	1324 ± 17	261 ± 8
4	2165 ± 28	2105 ± 24	60 ± 9	1750 ± 18	355 ± 9
5	2743 ± 27	2685 ± 22	58 ± 8	2230 ± 16	455 ± 8

[a]TN: total nitrogen; PN: protein nitrogen; NPN: non-protein nitrogen; CN: casein nitrogen; WPN: whey protein nitrogen.
[b]Means of duplicate analyses on each of three runs.
[c]VCR: volume concentration ratio.

During UF of milk, great changes also occur in the individual nitrogen fraction as a proportion of total nitrogen. The proportion of casein as well as whey protein increase with elevated concentration factors, due to the corresponding decreases of all the other nitrogen fractions (Renner and Abd El-Salam, 1991). Figure 4 shows typical changes in nitrogen fractions (as % of TN) of goats' skim milk during UF processing. Both casein and whey proteins increased from 75 and 15.2% of total nitrogen in skim milk to 81.2 and 16.6% in 5-VCR retentate, respectively. NPN content decreased from 10 to 2.1%. A similar observation was reported for cows' whole milk by Green *et al.* (1984) and for camel skim milk by Mehaia (1996).

Retention and recovery of milk components

The percent retention and recovery of total solids, fat, lactose, ash and protein in goat skim milk during UF are presented in Table 3. Figure 5 shows the percentage retention and recovery of nirogen fractions in goat skim milk concentrated by UF to 5-VCR. Retention and

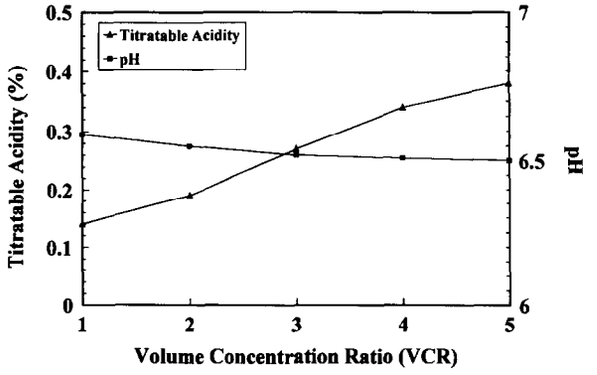

Fig. 3. Typical changes in the titratable acidity and pH of goat skim milk during ultrafiltration processing.

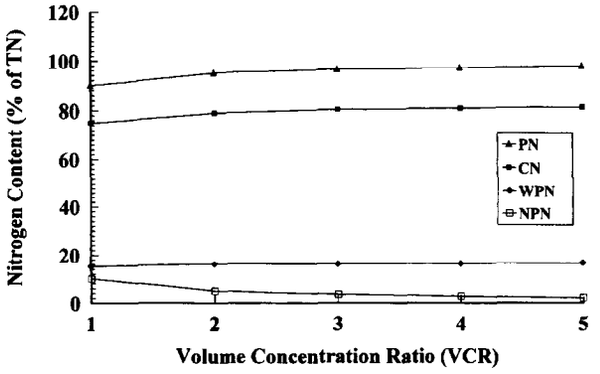

Fig. 4. Typical changes in nitrogen fractions (as % of TN) of goat skim milk during ultrafiltration processing.

recovery of fat and protein nitrogen were 100% in all retentates. The percentage retention of total solids, ash and total nitrogen were increased from 58.2, 39.8 and 96.9% in 2-VCR retentate to 78.1, 78.6 and 97.9% in 5-VCR retentate. The corresponding figures for percentage recovery were 71 to 53, 60 to 48 and 97 to 92%, respectively. Similar observations were reported for cows' milk (Glover, 1971; Bundgaard *et al.*, 1972; Green *et al.*, 1984; Fischbach-Greene and Potter, 1986) for camel skim milk (Mehaia, 1996) and for buffalo skim milk (Patel and Mistry, 1997). The percent retention of lactose was increased form 0.3 in 2-VCR retentate to 3.5% in 5-VCR retentate. This is lower than values reported for UF of cows' skim milk using a different equation, which does not rely on concentration factor, to calculate percent retention (Peri *et al.*, 1973; Covacevich and Kosikowski, 1977), but it agrees with Green *et al.* (1984) and Bastian *et al.* (1991) who used an equation similar to that used here (Bastian's equation). The recovery of lactose was reduced from 48% in the 2-VCR retentate to 17% in the 5-VCR retentate. Reported values for lactose recovery for 5-fold retentate cows' milk were 12% for whole milk (Bastian *et al.*, 1991), 22% (Pompei *et al.*, 1973) or 16% (Premaratne and Cousin, 1991) for skim milk and 17.3% for camel skim milk (Mehaia, 1996).

During UF, retention of TN increased from 96.9% in 2-VCR retentate to 97.9% in the 5-VCR retentate, whereas the recovery decreased from 97 to 92%. The corresponding figures for NPN were 10 to 19 for percentage retention and 40 to 14.7 for percentage recovery. Similar observations were reported for whole cows milk by Bastian *et al.* (1991). Glover (1971) and Pompei *et al.* (1973) reported 96.4 and 89% TN recovery (calculated from data using eqn (4)) for 1.6- and 5-fold retentate of cow skim milk, respectively. Casein and whey protein nitrogen were completely retained in the concentrates, i.e., retention was 100% (Fig. 5). The low molecular weight components comprising the NPN fraction were not concentrated at all, and their retention and recovery in 5-VCR retentate were 19 and 14.7%, respectively. Pompei *et al.* (1973) reported that the retention of casein, whey protein and NPN were 100, 98 and 59%, respectively, for 5-fold retentate of cows' skim milk (calculated from their data using eqn (3). Mehaia (1996) reported 100% retention and 100% recovery of casein, whey protein and 18% retention and 6.71% recovery of non-protein nitrogen for 5-VCR retentate of camel skim milk calculated using the same equations (eqns (3) and (4)).

Table 3. Levels of retention and recovery (%) of goats' skim milk components during UF process[a]

Component[b]	VCR[c] 2	3	4	5
Total solids				
Retention	58.2	66.2	73.1	78.1
Recovery	71.0	59.0	55.0	53.0
Fat				
Retention	100	100	100	100
Recovery	103	102	100	100
Lactose				
Retention	0.3	0.4	0.8	3.5
Recovery	48.0	29.0	22.0	17.0
Ash				
Retention	39.8	62.0	74.8	78.6
Recovery	60.0	54.0	53.0	48.0
TN				
Retention	96.9	95.5	97.0	97.0
Recovery	97.0	92.0	91.0	92.0

[a]Means of duplicate analyses on each of three runs (calculated from mean values in Table 1).
[b]TN: total nitrogen.
[c]VCR: volume concentration ratio.

Barbano *et al.* (1988), Bastian *et al.* (1991), Pompei *et al.* (1973) and Peri *et al.* (1973) showed that less than 1% of bovine whey proteins passed through 10 000 and 20 000 MWCO membranes using Dorr-Oliver plate and frame and Abcor spiral-wound membrane models, while Premaratne and Cousin (1991) reported 99% retention and 100% recovery of bovine total proteins using a hollow fibre membrane with 30 000 MWCO. Passage of protein through a UF membrane pore could be dependent on several factors such as: (a) molecular weight, charge, hydrodynamic size and shape, and hydrophobic or hydrophilic character of molecule; (b) type of membrane materials; (c) membrane configuration; (d) presence of other solutes; and (e) absorption of solutes by the membrane (Cheryan, 1986).

Rennet coagulation time (RCT)

Coagulation time is an empirical measurement of coagulation which is easier to determine than the three parameters on which it depends: (1) the enzymatic rate; (2) the aggregation rate and (3) the degree of proteolysis at which the aggregation starts (Garnot, 1988).

The effect of volume concentration ratio and rennet concentration on rennet coagulation time of goats' skim milk during UF processing, at pH 6.6 and 35°C, are shown in Fig. 6. The RCT decreased as the VCR (protein concentration) increased. RCT decreased from 200 to 130 s as the protein concentration increased from 3.80

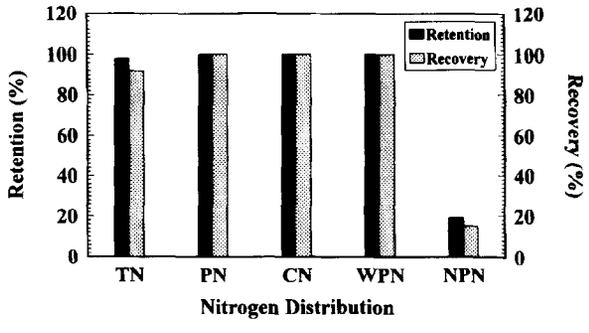

Fig. 5. Percentage retention and recovery of nitrogen fractions in goat skim milk concentrated by ultrafiltration to 5-VCR.

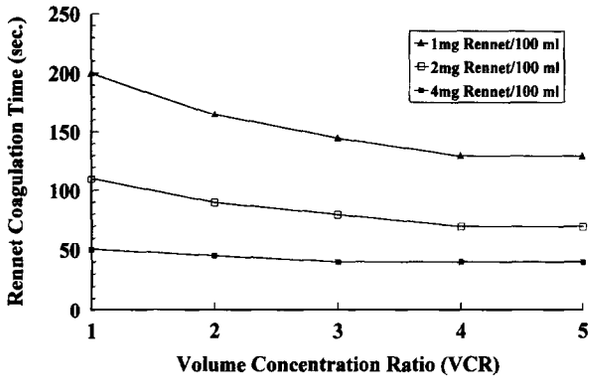

Fig. 6. The effect of volume concentration ratio and rennet concentration on rennet coagulation time of goat skim milk during UF processing, at pH 6.6 and 35°C.

to 17.4 g $100\,g^{-1}$, using 1 mg rennet $100\,ml^{-1}$ sample. However, the precise influence of protein concentration decreased at higher levels of rennet. These effects were presumably due to: (1) an increase in the number of effective collisions as a result of the decrease in the volume of the aqueous phase; (2) an increase in calcium concentration leading to more secondary phase interactions (Mehaia and Cheryan, 1983*b*; Garnot, 1988); and (3) an increase in the ratio of protein to total solids in the UF milk (Table 4). Similar observations were reported for cows' milk by some workers, eg. Reuter *et al.* (1981), Mehaia and Cheryan (1983*a*), Lucisano *et al.* (1985) and Pahkala *et al.* (1985) and for camel skim milk by Mehaia (1996). The conclusions of Dalgleish (1980) are even contradictory. He observed that the coagulation time is relatively unaffected by the concentration of milk by UF. On the other hand, other workers (Culioli and Sherman, 1978; Schmutz and Puhan, 1978) reported that the coagulation time increased with protein content and decreased with rennet concentration.

CONCLUSIONS

From the foregoing results it could be concluded that the concentrations of fat and protein in goats' skim milk increased proportionally with the VCR used for UF. In 5-VCR retentate, the CF was 5, 4.57, 2.66, 2.38 and 0.86 for fat, protein, TS, ash and lactose, respectively. All fat, CN, WPN, 19% of NPN, 78.1% of TS. 78.6% of ash and 3.5% of lactose were retained during UF of goats' skim milk. The transfer of lactose through the membrane was similar to that of water. During UF of goats' skim milk, retention of TN, TS and ash increased by increasing VCR. This means that permeate to retentate ratios of these constituents did not remain constant during UF processes. The titratable acidity of ultrafiltered goat skim milk increased during UF, whereas pH slightly decreased, indicating that UF milk has a high buffer capacity. The effect of milk concentration on the pH and acidity is due to increase of milk solid–not–fat and shifts in distribution of calcium and phosphate between dissolved and colloidal states. Rennet coagulation time of UF-concentrated goat skim milk decreased as the protein concentration increased, but the precise influence of the latter decreased as the rennet concentration increased.

Such data have relevance to questions of standards of characteristics and nutritional quality of ultrafiltered goat milk products.

Table 4. Protein to total solids ratio and calcium concentration of goats' skim milk during UF processing

VCR[a]	Protein: total solids	Calcium concentration (mg $100\,g^{-1}$)
1	0.43	115
2	0.58	218
3	0.66	310
4	0.70	398
5	0.73	516

[a]Volume concentration ratio.

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