Microfiltration, Ultrafiltration, and Centrifugation Separation and Sterilization Processes for Improving Milk and Cheese Quality

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ABSTRACT

Separation and sterilization processes involving centrifugation, ultrafiltration, and microfiltration are reviewed as to characteristics, tentative performance, and potential advantages. These processes require one or more separating means to remove skim milk or permeate from whole milk, followed by heating the resulting creams or retentates to 120°C and then blending the fat-free material with the fat component. The blend is thereafter homogenized as required, pasteurized, cooled, and used as fluid milk or for manufacture of cheese and other dairy products.

Trials on milk blends produced by separation and sterilization principles using centrifugation, ultrafiltration, or microfiltration show increased shelf-life at chill temperatures for all blends. Such whole milk retained good quality compared with quality of controls for many days, the number being influenced directly by storage at 3.3 to 8.0°C. Cheddar and Emmental cheese of high quality were produced by the separation and sterilization processes.

(Key words: separation and sterilization, cheese and milk quality, microfiltration)

INTRODUCTION

Separation and sterilization of milk was developed and applied to fluid milk processing and cheese making a decade ago or more.

During 1980, Schilhabel (13) obtained a German patent for a process that involved pumping raw milk into a vertical holding vat and separating it by gravity through a step called stacking. This created a skim milk and separate layers of fat-rich cream. The cream was pumped through a disc centrifuge to attain a richer product and heated to 150°C or bactofugated. Resulting cream and skim milk, both very low in bacteria, were blended, pasteurized, cooled, and utilized as fluid milk or manufactured into Emmental cheese. The fluid milk maintained its quality for 20 d at 12°C.

In the same year, Kosikowski and Mistry (7) described research on subjecting market milk and cheese milk to separation and sterilization. Raw whole milk was converted into heavy cream and skim milk by passage through a standard mechanical milk separator or centrifuge. Portions of the resulting cream were heated at 120°C for different times and then blended with the skim milk. The resulting whole milk blend was heated to 61.7°C for 30 min for fluid milk or heat-treated to 61°C for 30 s for Cheddar cheese milk. Similar experiments were conducted in the same period using ultrafiltration to separate raw milk into a high fat portion (retentate) and an aqueous portion (permeate), the details of which were originally presented in a United States patent application (3).

Glimenius et al. (2) in 1979 obtained a United States patent describing a method wherein whole milk was separated by microfiltration into cream and skim milk. Separation was accomplished by cross flow of milk at high speed over a fine filter with a pore size of .2 to 10 μ. The patent, however, did not deal with sterilization of cream and recombination with the microfiltered skim milk. Malmberg and Holm (8, 9) first reported pilot plant experience by their organization on the application of a microfiltration separation and sterilization concept followed in 1988 by another by Vincens and Tabard (15).

Our objective here is to review some tentative results, salient features, and potential ad-

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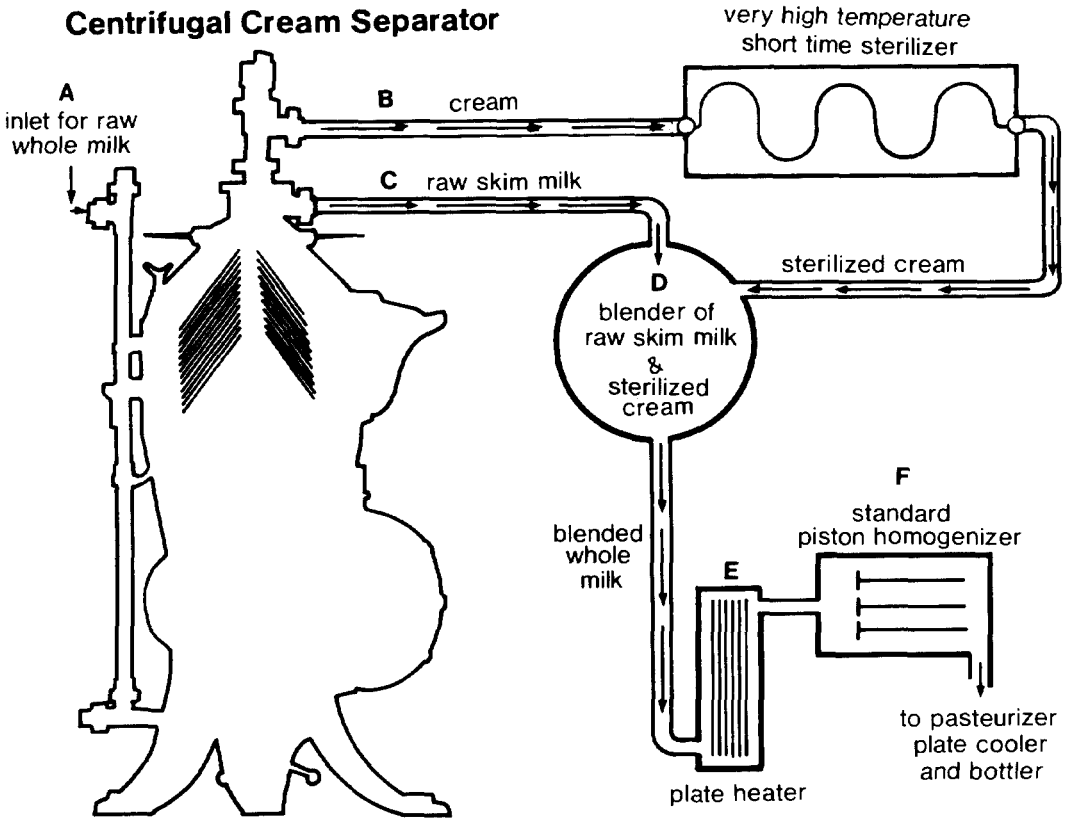

Figure 1. A graphic view (not to scale) of the separation and sterilization process utilizing mechanical separation and sterilization at 120°C.

vantages of separation and sterilization processes employing centrifugation, ultrafiltration, and microfiltration.

EARLY CORNELL SEPARATION AND STERILIZATION EXPERIMENTS

Using Centrifugation

Raw milk from a commingled source was mechanically separated at approximately 5°C into heavy cream and skim milk at the Cornell University Dairy Plant. The cream was filled into 6-oz evaporated milk cans, then sealed, and in the absence of a UHT unit, these cans were heated in a reel retort to 120°C for intervals from 3 to 10 min. Thereafter, the creams were cooled to room temperature and blended with the raw skim milk to give a product having fat levels comparable to those of whole milk. Such separated and sterilized blends were homogenized, heated to 61.7°C for 30 min, cooled, and stored in sterile Erlenmeyer flasks, covered with a plastic seal, at 3.3 or 7.2°C for shelf-life trials. Unhomogenized milk blends treated similarly were used for Cheddar cheese making except that the blends were either postheated to 61°C for 30 s or not postheated. A graphic illustration envisioning a small plant operation is shown (Figure 1), and previously unpublished results on composition and quality are listed in Tables 1 to 8.

Flavor and body-texture of resulting fluid milks and Cheddar cheeses were scored where 10 = outstanding quality and 0 = unacceptable. Flavor intensity of cheeses was scored where 5 = strong intensity and 0 = lack of flavor.

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TABLE 1. Quality of mechanically separated, raw skim milk and sterilized cream blends used as fluid milk and held at 3.3°C (Trial 1).

		Flavor quality of milk blend held			
		14 d		38 d	
Lot	Treatment	Score[1]	Comments	Score[1]	Comments
1	Whole milk homogenized and heated to 61.7°C for 30 min (control)	9.0	Excellent	0	Unacceptable, putrid
2	Raw skim milk and raw cream blended, homogenized, and heated to 61.7°C for 30 min (control)	9.0	Excellent	0	Unacceptable
3	Cream autoclaved at 120°C for 8 min and blended with raw skim milk; homogenized and heated to 61.7°C for 30 min	9.0	Excellent	9	Excellent

[1]Based on a flavor score range of 10 to 0 where 10 = outstanding flavor and 0 = unacceptable as scored by two experienced milk judges.

Cheddar cheese was made according to directions outlined for pasteurized milk by Kosikowski (5) using small 14-kg capacity vats resting in insulated metal containers. Chemical analyses of milk, whey, and cheese were conducted using methods described by the same source (5).

Results

A milk blend formed from mechanically separated raw skim milk and sterilized (120°C for 8 min) cream, which was subsequently postheated (61°C for 30 s), was evaluated for flavor quality after storage at 3.3 and 7.2°C. The separated and sterilized milk blends held at 3.3°C exhibited excellent flavor after 38 d of storage. Control samples demonstrated a putrid flavor and were judged unpalatable (Table 1). A separated and sterilized blend, containing cream heated to 120°C for 5 min, displayed good flavor quality after 15 d at 7.2°C, while controls were unacceptable after 10 d of storage (Table 2). Additionally, three trials at 7.2°C using separated and sterilized blends containing creams heated to 120°C for 3, 6, and 10 min exhibited similar good flavor at 15 d.

A separated and sterilized (120°C for 5 min) blend, postheated to 61.7°C for 30 min and supplemented with 1.5 ppm copper sulfate to induce oxidized flavor, showed no off-flavor following 10 d at 7.2°C (Table 3). For control samples, strong oxidized off-flavors occurred within the same time span.

The previous experiments were conducted on a fixed time schedule; hence, the maximum

TABLE 2. Quality of mechanically separated, raw skim milk and sterilized cream blends used as fluid milk and held at 7.2°C (Trial 2).

		Flavor quality of milk blend held			
		10 d		15 d	
Lot	Treatment	Score[1]	Comments	Score[1]	Comments
1	Whole milk homogenized and heated to 61.7°C for 30 min (control)	0	Putrid	0	Putrid
2	Raw skim milk and raw cream blended, homogenized, and heated to 61.7°C for 30 min (control)	2.0	Slightly oxidized	0	Oxidized
3	Cream autoclaved at 120°C for 5 min and blended with raw skim milk homogenized and heated to 61.7°C for 30 min	8.5	Very good, slightly cooked	8.5	Very good, slightly cooked

[1]Based on a flavor score range of 10 to 0 where 10 = outstanding flavor and 0 = unacceptable as scored by two experienced milk judges.

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TABLE 3. Oxidized quality of mechanically separated, raw skim milk and sterilized cream, copper-exposed blends used as fluid milk (Trial 3).

Lot	Treatment	Milk blends held 10 d at 7.2°C		
		$CuSo_4$ Addition	Flavor score[1]	Comments
		(ppm)		
1	Whole milk, homogenized and heated to 61.7°C for 30 min (control)	1.5	0	Oxidized, lacks freshness
2	Raw skim milk and raw cream blended, homogenized, and heated to 61.7°C for 30 min (control)	1.5	2.0	Oxidized
3	Cream autoclaved at 120°C for 5 min and blended with raw skim milk; homogenized and heated to 61.7°C for 30 min	1.5	8.0	Very good, slightly cooked

[1]Based on a flavor score range of 10 to 0 where 10 = outstanding flavor and 0 = unacceptable as scored by two experienced milk judges.

number of days that good or satisfactory flavor quality could be retained was not determined.

Cheddar cheeses were made from raw skim milk and sterilized (120°C for 5 or 10 min) cream blends. These displayed excellent flavor of cheese to medium intensity and good body and texture after 2 and 4 mo at 10°C (Table 7). No flavor defects were observed and body-texture defects were limited to slight curdiness and fragility (Table 7). Cheeses and wheys from these trials displayed normal, or near normal, compositions (Tables 4, 5, 6, and 8).

Using Ultrafiltration

Several exploratory trials were carried out on blends of whole milk UF retentates and permeates. The whole milk UF retentates were concentrated to approximately 5:1 total protein at 54°C in a tubular Abcor UF-22s unit (Koch Inc., Kansas City, MO) with membrane pore size of 20,000 daltons. For fluid milk quality studies, the UF retentate was heated to 120°C for 5 min and blended with the permeate to a fat content consistent with the control milk. This blend was thereafter postheated to 61.7°C for 30 min.

Results

A UF permeate, sterilized (120°C for 5 min) retentate blend that was postheated to 61.7°C for 30 min and then cooled and stored at 4.5°C exhibited good to excellent flavor after 22 and 38 d. The controls showed poor flavor over the same period. Some cooked flavor apparent in the experimental samples at 0 and 22 d had disappeared after 38 d.

MICROFILTRATION OF MILK

Microfiltration, or fine filtration, is a separation process requiring filters with pore size of .1 to 10 μ. It is used widely in the brewing and wine industries to cold-sterilize beer and wine prior to packaging. Apparently little had been

TABLE 4. Composition (%) of mechanically separated, raw skim milk and sterilized cream blends used for Cheddar cheese making.

Trial	Heat treatment of cream	Total solids	Total protein	Fat	Ash	Lactose
1	120°C for 5 min	13.2	3.2	4.5	.7	4.8
2	120°C for 10 min	13.3	3.2	4.7	.7	4.8
		Postheated to 61°C for 30 s				
3	120°C for 5 min	12.7	3.2	4.1	.7	4.7

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TABLE 5. Composition (%) of Cheddar cheese made from mechanically separated, raw skim milk and sterilized cream blends.

Trial	Total solids	Total protein	Fat	Ash	Lactose	NaCl
1 and 2[1]	67.8	24.5	37.5	3.2	2.4	1.6
3[2]	60.4	24.3	30.0	3.5	2.6	2.0

[1]Mixture of equal weights of curds from 120°C for 5 min and 120°C for 10 min sterilized cream and raw skim milk blends.

[2]Curds from 120°C for 5 min sterilized cream and raw skim milk blend, postheated at 61°C for 30 s.

published on the application of microfiltration to milk before Glimenius et al. (2) separated whole milk into skim milk and cream by this method. Recent technical developments involving the sterilization of cream and recombination with microfiltered skim milk and its application to fluid milk processing and cheese making have been carried out by the Alfa-Laval Engineering Group at Lund, Sweden (8, 9) and by Maubois' Dairy Technology Group at Rennes, France [(11); personal communication].

Essentially, this process, as the others, is used to produce low bacteria milk with substantially reduced protease and lipase content and, consequently, improved product quality and enhanced food safety. To make microfiltration of milk industrially possible, new engineering technology involving type of membrane, flow patterns, and maintenance of proper pumping pressures was necessary. Specifications were met in part by a cross flow of milk on ceramic membranes, by maintaining an optimum pressure differential between microfiltration retentate and permeate, by increasing pressure at cycle end with a supplementary pump, and by permitting only a portion of the milk access across the membrane surface. As a result, fouling of membranes was minimal.

Bactocatch, the term used to describe this new Alfa Laval process, was initially tested for its effectiveness in improving whole milk quality at a 12,000 l/h dairy unit in Sweden (Figure 2). In the Swedish study, whole milk was separated by microfiltration and the resulting cream was heated by UHT at 115 to 120°C for 3 s. Then the skim milk, with about 99.6% of the bacteria removed, was blended with the sterilized cream, and the blend was homogenized, pasteurized, chilled, and packaged. More recently, rather than microfiltering whole milk, the process has been modified so that the whole milk is first separated mechanically and the resulting skim milk is microfiltered and then blended with the heated cream.

Malmberg and Holm (8, 9) reported that the flavor quality of fresh milk mixtures subjected to Bactocatch technology was maintained for 16 to 21 d at 8°C. By contrast, control pasteurized milk retained its peak quality for only 6 to 8 d at 8°C. The flavor of the microfiltered milk blends was without obvious cooked overtones. Caution was expressed, however, about possible microbial contamination between the cooling and packaging stages.

Results obtained by Maubois and his group [(11); personal communication] show the great potential of Bactocatch principles for improving quality of many dairy products. This was demonstrated with a Bactocatch pilot plant

TABLE 6. Composition (%) of whey from Cheddar cheese made from mechanically separated skim milk and sterilized cream blends.

Trial	Heat treatment of cream	Total solids	Total protein	Fat	Ash	Lactose
1	120°C for 5 min	7.0	.80	.34	.58	5.2
2	120°C for 10 min	7.1	.80	.35	.59	5.3
		Postheated to 61°C for 30 s				
3	120°C for 5 min	6.9	.82	.32	.56	5.2

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TABLE 7. Flavor and body quality or ripened Cheddar cheese made from mechanically, separated, raw skim milk and sterilized cream blends.

		Cheese held 60 d at 10°C				
Trial	Heat treatment of cream	Flavor score	Comments	Body score	Body comments	Flavor intensity[1]
1	120°C for 5 min	9.0	Excellent	8.0	Slightly curdy Slightly fragile	2.0
2	120°C for 10 min	9.0	Excellent	8.0	Slightly fragile (weak)	1.5
		Postheated for 61°C for 30 s				
3	120°C for 5 min	9.0	Excellent	8.0	Slightly curdy	2.5

[1]Based on flavor score range of 10 to 0 where 10 = outstanding and 0 = unacceptable. Flavor intensity range based on score of 5 to 0 where 5 = strong intensity and 0 = lacks flavor as judged by an experienced cheese judge.

(Figure 3) equipped with 1.4 m^2 of 1.4-μ pore size Membralox membranes (multichannel tubular conception with 4-mm inner diameter). These membranes obtained a 1000 l/h flux for 10 h on raw skim milk. Also, 99.9% of the contaminant bacteria, including *Clostridium tyrobutyricum* spores, were retained in the microfiltered retentate (5% of the total starting volume). The blend of 14.5 parts UHT-treated cream plus microfiltered retentate and 85.5 parts of raw skim milk was then transformed into high quality Emmental cheese with the addition of propionic starters.

The high effectiveness of the microfiltration separation and sterilization process to reduce bacterial levels in milk has been most recently confirmed in 1989 by Olesen and Jensen (12) of the Danish Dairy Research Institute. They report a 99.99% reduction in total bacteria numbers of whole milk and an average 99.98% reduction in *Bacillus cereus* spores.

"Bactocatch skim milk" was also used to prepare phosphocaseinate according to the microfiltration process described by Fauquant et al. (1). The resulting powder (89% protein) contained less than .2% β-lactoglobulin or about ten times lower than commercial caseinates.

Discussion

A description of separation and sterilization processes for improving the quality of milk and cheese is presented here along with some results. The processes include separation of milk by gravity plus disc centrifugation, direct mechanical separation or centrifugation, ultrafiltration, and microfiltration. Gravity separation is

TABLE 8. Soluble protein, free volatile fatty acids (FVFA), and pH of ripened Cheddar cheese made from mechanically separated, raw skim milk and sterilized cream blends.

		Cheese					
		Held 2 mo at 10°C			Held 4 mo at 10°C		
Trial	Heat treatment of creams	Soluble protein (%)	FVFA[1]	pH	Soluble protein (%)	FVFA[1]	pH
1	120°C for 5 min	2.3	14.0	4.8	4.3	15.8	4.8
2	120°C for 10 min	3.3	12.0	4.9	4.2	16.0	4.9
		Cheese from sterilized cream-raw skim milk postheated for 61°C for 30 s					
3	120°C for 5 min	4.1	8.0	5.0	NA[2]	NA[2]	NA[2]

[1]Milliliters n/10 KOH per 100 g.

[2]Not analyzed.

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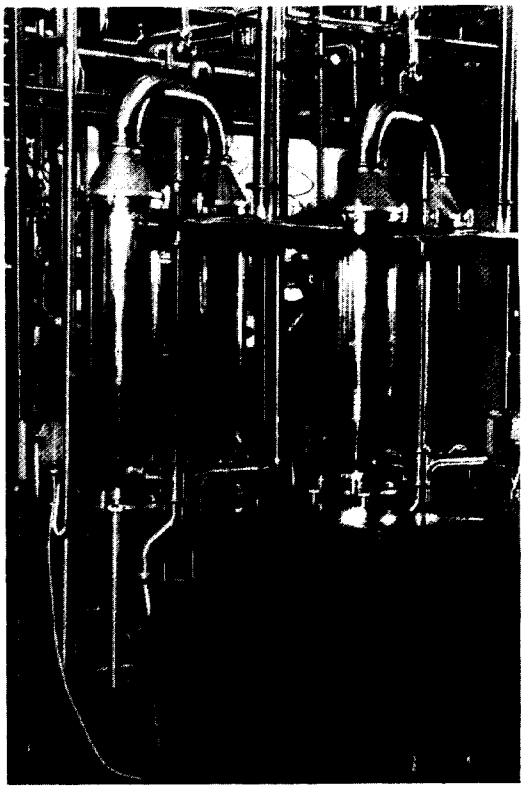

Figure 2. Industrial microfiltration unit (Bactocatch).

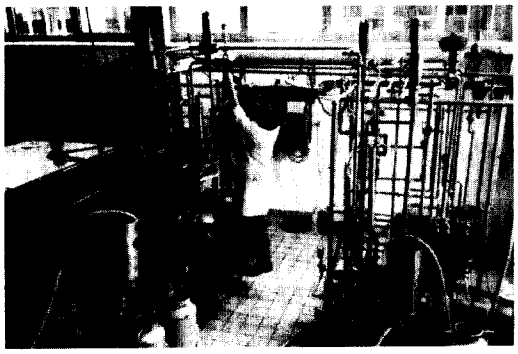

Figure 3. Microfiltration pilot plant in laboratory of J. L. Maubois.

only briefly mentioned, because it is not typically used in practice. Of the remaining three processes, the microfiltration separation and sterilization process has been tested under commercial dairy plant conditions and appears most advanced and potentially most promising. Reportedly, several dairies outside the United States are employing the Bactocatch process in their plants, and at least 40 pilot plants have been sold.

The term "sterilization", as it appears in separation and sterilization, is used here only advisedly. Although microfiltration and ultrafiltration can produce permeates basically free of bacteria, the present very high heat treatments used for cream and retentates, with the exception of 150°C applied by Schilhabel (13), were not fully equivalent to standard heat sterilization. In practice, however, all fresh milks and milk products treated by separation and sterilization procedures would normally be pasteurized, chilled, and distributed cold, and any heat-treated milk cheeses would be held for 60 d before distribution. Also, it is possible that creams and retentates if actually heat-sterilized (150°C for 2.5 s) in the process will not suffer serious loss of flavor quality, thus confirming the findings of Schilhabel (13).

The processes have one common denominator; all appear to increase the shelf-life of fluid milk by many days. The mere physical act of separation in any of the procedures concentrates bacteria and enzymes in the creams or retentates where they can be destroyed or inactivated by heat. Bacteria, and possibly somatic cells, do not pass through the ultrafiltration or microfiltration membranes, leading initially to sterile permeates or skim milks. Centrifugation or mechanical separation reduces bacteria and enzymes greatly in skim milk, too. In part, their concentration in creams or retentates may be explained by their surface adsorption on fat globules. We have observed, for example, approximately nine times more alkaline phosphatase in cream compared with skim milk from raw milk that has been mechanically separated (6).

The marked improvement in milk keeping quality by the separation and sterilization processes discussed here suggests a need for more in-depth research. This could include studying the behavior of low to high bacteria count milks, comparing effectiveness of mechanical

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versus membrane separation and sterilization, assessing change in milk and cheese properties, and analyzing relative costs. Future mechanical separation research to improve milk and cheese quality should consider the inclusion of Bactotherm treatment in the experimental design. Bactotherm, stemming from bactofuges developed by Simonart (14), and a patent on sterilization-recycling (4, 5) is an early separation and sterilization system, which removes bacteria continuously from milk or skim milk by centrifugal force. The small amount of bacteria-rich bactofugate resulting (3%) is UHT treated and recycled back to the nonsterilized cheese milk. There is an approximate 98% reduction in the spores of butyric acid-fermenting bacteria by the Bactotherm process without any concomitant disruption of the physical properties in resulting cheeses.

Ultrafiltration separation and sterilization trials for fluid milk were initiated by us to note whether whole milk retentates could be sterilized, blended with unheated permeate, and then postheated to make a good quality product. Limited results show that recycling the permeate, theoretically free of bacteria, to the sterilized retentate with postheating improved the flavor and keeping quality in the resulting fluid milk blend and potentially increased safety. In future ultrafiltration separation and sterilization studies, not only milk but also cheeses and other dairy products should be included, as there are indications that such research may lead to interesting results.

It is well known that milk loses its ability to be coagulated by rennet when heated above 100°C without calcium addition or pH adjustment. The UHT milk normally does not form a rennet gel, and consequently, could not be used for cheese making. In 1973, Maubois et al. (10) noted a surprising property of ultrafiltered UHT milk, i.e., its ability to form a rennet coagulum suitable for cheese making. The UF retentates containing twice the normal amount of protein than milk were easily transformed into cheese, regardless of whether application of UHT treatment was before of after ultrafiltration. Advantage should be taken of this phenomenon to study the ultrafiltration separation and sterilization process utilizing highly heated or sterilized retentate blends for cheese making.

A number of advantages may accrue should separation and sterilization processes for treating milk prove successful. They include:

1. Extended keeping quality of milk. The high rate of destruction and inactivation of spoilage bacteria (particularly psychrotrophs with their associated proteases and lipases) by exposure of a small portion of the milk as cream or retentate to high heat should extend the keeping quality of milk blends substantially without creating excess cooked flavor.
2. Increased safety of milk and milk products. Important pathogenic and food poisoning bacteria, including the spore former *Bacillus cereus*, may be controlled more effectively.
3. Reduction or elimination of oxidized off-flavor. Oxidized flavor of milk appears to be eliminated without concomitant excessive cooked flavor through cream sterilization, perhaps because sulfhydryl groups inhibitory to autoxidation processes of milk fat are formed.
4. Improvement in quality and safety of cheese. Destruction of food poisoning and spoilage organisms, including more effective control over anaerobic gas formers, such as *Clostridium tyrobutyricum*, without jeopardizing normal development of cheese appears possible.
5. Improved cheese yield. High heat applied to creams or retentates denatures considerable lactalbumin and lactoglobulin, thus enabling these proteins to associate with the curd and thereby increase yield.

SUMMARY

The potential of separation and sterilization processes appears promising for future new milk processing technologies and for marked improvement in milk and milk product quality and safety. Employing proper sterilization and separation principles may result in a marked transformation of how milk and its products will be treated throughout the world.

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