

Comparison of the Physicochemical Properties of Freeze-Concentrated versus Evaporated Milk

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Abstract This study was designed to compare various quality characteristics, such as nutrient composition and physicochemical and sensory properties of freeze-concentrated milk made by a newly developed continuous multi-stage process with those of evaporated milk. The freeze concentration process reduced the water content up to 73%. Most of the physicochemical properties of evaporated milk were different from raw milk; however the freeze-concentrated milk showed little difference from the raw milk. The thiobarbituric acid value and free fatty acid concentrations were significantly greater in the evaporated milk than in the freeze-concentrated milk. Several effects on sensory characteristics, such as off-taste, were significantly stronger in the evaporated milk. Overall, this study indicates that the newly developed freeze concentration technique results in improved physicochemical and sensory properties, and has little effect on most nutrient levels when compared with the evaporation process. Further research is necessary to further elucidate the chemical and sensory properties of freeze-concentrated milk.

Keywords: milk, concentrated milk, freeze-concentrated milk, evaporated milk

Introduction

Concentrated milk products occupy less space, weigh less, and have a longer shelf-life than fresh milk. Hence, they save storage and packaging space, cost less to transport, and serve as a reserve in times of short supply. Concentrated sources of milk solids are required in numerous foods to avoid the dilution that occurs with less concentrated forms of milk (1).

In dairy foods, concentrated milk has been widely used as an intermediate material or final product for consumers. To obtain a concentrate, the water content in milk must be reduced by 75%, therefore a process to eliminate water is necessary (2). Among the three most common methods for eliminating water, evaporation, reverse osmosis, and freeze concentration (3), evaporation, a thermal process, is most frequently used for the concentration of milk in the dairy industry. However, this method can have undesirable effects such as discoloration, heat-coagulation, and a burnt flavor, on the final product. In addition, when milk is heated at temperatures above 70°C, the whey protein is denatured and will interact with other milk proteins (4).

The most common problems associated with evaporation are insufficient thermal stability, excessive thickening and gelation, fat separation, sediment formation, and color and taste alterations. Insufficient thermal stability of condensed milk is demonstrated during the sterilization of proteins when product coagulation occurs. Insufficient thermal stability can be caused by milk composition and properties (salt composition, protein content, pH, etc.), and by inadequate heat treatment, the degree of concentration, and sterilization. Careful preheating and test sterilization measures and stabilizing agents may correct the thermal

instability. (5)

A rather new concentration technique is freeze concentration (6), which is being studied as a non-thermal processing technology to preserve milk quality. Several attempts have been made to apply this technique to milk and dairy products, however the amount of product loss and the costs associated with the process were too high. Therefore, a less expensive method for removing ice crystals with minimal product loss is necessary (6).

The application of freeze concentration to the dairy industry has been demonstrated in the past (6), however, only limited commercial success has been obtained due to low solute concentrations (10-17%) in the final product (7). In the present study, melt-refreeze recrystallization was applied to increase the solute concentration using artificial temperature fluctuation. Due to the low temperature of operation, no heat-induced changes occur resulting in high quality products for many liquid foods. In addition, though high efficiency wash columns are used for ice-concentrate separation, the loss of volatile aromas and flavors is minimized. Freeze concentration has been particularly successful in the concentration of citrus juices, and is also useful for the production coffee and tea extracts, beer and wine (6). Despite these potential advantages, freeze concentration has found only limited application in the concentration of fluid foods mainly due to 1) high capital costs, 2) losses of solids, particularly at higher concentrations, and 3) a lack of understanding ice crystallization, which is necessary to optimize the process (6).

It has been claimed that reconstituted skim milk, previously concentrated by freeze concentration, has a smoother and creamier product texture than the original skim milk. No scientific evidence for this claim has been put forth, however physical changes in protein structure may cause some chemical and organoleptic changes and this should be evaluated (8). There are relatively few studies address-

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ing freeze-concentrated milk with limited technologies, therefore the present study was carried out to investigate changes in nutrient composition, and chemical and sensory properties of freeze-concentrated milk by the newly developed process of multi-stage freeze concentration.

Materials and Methods

Preparation of freeze concentrated milk After purchasing LTLT (low temperature long time, 63°C, 30 min) pasteurized whole milk (Pasteur Milk Co., Hoesungseong, Korea) having initial total solids of 13% from a local market, the milk was pre-cooled at 4°C for 12 hr. Exactly 1,860 mL was loaded into the concentrator for each freeze concentration procedure. Each freeze concentration process was done in duplicate.

Freeze concentration was carried out in a multi-stage freeze concentrator (9). The initial structure of the concentrator can be explained as follows. Ice crystals formed on the inner wall of the stainless vessel by circulating coolant from the cryostat (FP-80; Julabo, Seelbach, Germany). A teflon scraper continually rotated at 50-60 rpm in the center of the concentration vessel to remove ice crystals while maintaining a minimum distance (15 mm) from the inner wall. Displaced ice crystals were collected in the central portion of the chamber and recrystallized. The coolant temperature was varied in a regular pattern to accelerate the recrystallization of ice, a process defined as heat and cold shock in this study. Supercooling was observed to a sample temperature of -4°C in the beginning of freezing process. Initial freezing occurred at a sample temperature of -4°C coupled with nucleation and crystallization, followed by an increase of sample temperature to -0.3°C due to latent heat during the crystallization process. After nucleation, the coolant temperature was increased to -2°C as a heat shock step and maintained constant during the recrystallization process. The sample temperature was -0.3°C in the early stages and slowly decreased to -1.8°C at the end of recrystallization. The temperature range was intended to let the sample temperature be near the freezing point in the middle of the recrystallization course. Temperature profiles of coolant and milk were collected with a K-type thermocouple and data logger (MV-100; Yokogawa, Tokyo, Japan). After each recrystallization process, ice crystals and milk concentrates were separated by filtration involving a stainless steel net (200 mesh) and a vacuum pump.

Preparation of evaporated milk The evaporated milk was prepared using a vacuum evaporator (R-205, Büchi, Bern, Switzerland) such that the solute concentration was equivalent to that of the freeze concentrated milk. During evaporation, the vapor temperature was maintained at 70°C under a vacuum pressure of 300 Torr to prevent major changes in various milk components.

Analysis of chemical composition All milk samples were analyzed for moisture, fat, protein, and ash content using the methods of the AOAC (10). The lactose content in milk was determined by the method of Kwak and Jeon (11). Sample (10 mL) was poured into a 25 mL volumetric flask and 15 mL of 2-propanol was added followed by

thorough mixing. The mixture was allowed to stand at room temperature for 20 min and then centrifuged at 275×g for 10 min. The supernatant was filtered through Whatman No. 540 paper and Sep-Pak C₁₈ column for HPLC determination (Waters Corporation, Polumouth, MA, USA). Lactose analysis was performed by using a Cosmosil packed column 10_{NH2} (4.6 mm i.d. × 25 cm), and HPLC. Acetonitrile:water (3:1) was used as the mobile phase and propelled at 2 mL/min. A refractive index detector was used to measure the sample and the injector volume was 20 µL. A standard curve was constructed by injecting glucose and galactose standards to yield a linear curve. All measurements were done in triplicate.

pH and titratable acidity (TA) The pH and TA (determined by titration to pH 8.3) of the milk samples were measured at room temperature using a pH meter (Sartorius, Gottingen, Germany). The TA was determined after mixing 9 mL milk samples with 18 mL distilled water and titrating with 0.1 N NaOH using 0.5% phenolphthalein to indicate the end point of titration with a faint pink color (12).

Analysis of oxidation products by the thiobarbituric acid (TBA) test Oxidation products were analyzed spectrophotometrically using the TBA test (13). The TBA reagent was prepared immediately before use by mixing equal volumes of freshly prepared 0.025 M TBA which was neutralized with NaOH and 2 M H₃PO₄/2 M citric acid. TBA test reactions were begun by pipetting 5.0 mL milk samples into glass centrifuge tubes and mixing thoroughly with 2.5 mL of TBA reagent. The mixture was heated immediately in a boiling water bath for exactly 10 min and cooled on ice. Ten mL of cyclohexanone and 1 mL of 4 M ammonium sulfate were added and centrifuged at 2,490×g for 5 min at room temperature. The orange-red cyclohexanone supernatant was decanted and its absorbance at 532 nm was measured spectrophotometrically with a 1-cm light path. All measurements were done in triplicate.

Short-chain free fatty acid (FFA) analysis Samples (1 mL) were removed periodically from the raw milk, and extracted with diethylether and hexane for 2 hr and passed through a 10 mm i.d. glass column containing neutral alumina as described by Ikins *et al.* (14). A Hewlett-Packard Model 5880A GC (Palo Alto, CA, USA) equipped with a flame ionization detector was used. The preparation of short-chain FFA was achieved using a 15 m × 0.53 mm i.d. Nukol fused-silica capillary column (Supelco Inc., Bellefonte, PA, USA). The GC was operated with helium carrier gas at 2 mL/min, hydrogen gas at 37 mL/min, and air at 300 mL/min. The column oven was programmed for an initial holding temperature of 110°C for 1 min followed by an increase to 180°C at 5°C/min for 10 min and holding at this temperature for 20 min. The temperatures of both injector and detector were 250°C. All quantitative analyses were done by relating the peak area of each individual FFA to the peak area of tridecanoic acid as an internal standard. Each FFA was identified by the retention time of the standard.

Free amino acid analysis To determine the FAA content,

5 g of milk was mixed with 5 mL of distilled water followed by the addition of 500 mg of sulfosalicylic acid. The mixture was stored at 4°C for 1 hr and centrifuged at 1,300×g for 15 min. The supernatant was filtered through a 0.45 µm filter and pre-treated as described by Lindorth and Mopper (15). FAA determination by HPLC was carried out using the modified method of Hodgins *et al.* (16). The flow rate was 2 mL/min and two mobile phases were used: solvent A was 0.05 M sodium acetate (pH 6.3), and solvent B was methanol:THF (90:10, v/v). The linear gradient of solvent B was programmed as follows: the initial level was 20%, followed by an increase to 40% for 6 min, 42% for 9 min, 50% for 3 min and finally to 70% for 12 min. FAAs were analyzed on an ODS-µ-Bondapak C column (3.9 mm × 30 mm) using an HPLC equipped with a RI detector. All quantitative analyses were performed by relating peak areas of individual FAAs to those of external standard amino acids (Wako, Osaka, Japan). All samples were analyzed in triplicate.

Vitamin analysis For the analysis of water soluble vitamins, 0.5 mL of milk was placed in a 50 mL volumetric flask, mixed with mobile phase and sonicated for 20 min. The mixed solution was centrifuged at 452×g for 20 min, filtered through MILLEX-HV13 (0.45 µm, Millipore, Ireland) analyzed by HPLC equipped with a RI detector using a Spadex RSpak DE-413L column (4.6 mm × 250 mm). The flow rate was 0.5 mL/min and two mobile phases were used: solvent A was 0.005 M PIC B₆, and solvent B was a mixture of 0.4 mL triethylamine in 15 mL methanol:acetic acid (1:1, v/v). The linear gradient of solvent B was programmed as follows: the initial level was 20%, followed by an increase to 40% for 6 min, 42% for 9 min, 50% for 3 min, and finally to 70% for 12 min. All quantitative analyses were performed by relating peak areas of individual FAAs to those of external standard amino acids (Wako). All samples were analyzed in triplicate.

To determine fat soluble vitamins, 1.0 mL of milk was placed in a screwed-capped glass tube followed by the addition of 1 mL of 10% pyrogalllic acid/ethanol mixture and 9 mL of 1 N KOH solution. and the mixture was then sonicated for 30 min. After cooling to room temperature, fat-soluble vitamins were extracted with 30 mL ether and 30 mL distilled water. The process was repeated three times. The ether layers were transferred to a round-bottom flask and dried under vacuum. The extract was re-dissolved in 10 mL isopropanol, filtered through 0.45 µm MILLEX-HV13 and analyzed via HPLC equipped with a RI detector and a Shiseido Nanospace SI-1 column (4.6 mm × 250 mm). The flow rate was 1.0 mL/min and two mobile phases were used: solvent A was 0.005 M PIC B₆, and solvent B was a mixture of 0.4 mL triethylamine in 15 mL methanol:acetic acid (1:1, v/v). The linear gradient of solvent B was programmed as follows: the initial level was 20% followed by an increase to 40% for 6 min, 42% for 9 min, 50% for 3 min, and finally to 70% for 12 min. All samples were analyzed in triplicate.

Color analysis Color values were compared between the freeze concentrated and evaporated milk samples using a colorimeter (CR210; Minolta, Tokyo, Japan) after

calibrating its original value with a standard plate ($X=97.83$, $Y=81.58$, $Z=91.51$). Measured L -, a -, and b -values were used as indicators of lightness, redness, and yellowness, respectively. The ΔE value representing total color difference was calculated as follows.

$$\Delta E \text{ value} = \sqrt{(L-L')^2 + (a-a')^2 + (b-b')^2}$$

L , a , b : color values of control milk. L' , a' , b' : color values of concentrated milk.

Viscosity analysis The viscosity of 50 mL milk samples was measured at 5°C using a viscometer (VISCO STAR-L; J.P. Selecta S.A., Spain) with a single spindle at 200 rpm. All samples were measured in triplicate.

Statistical analysis All data were analyzed using the Statistical Analysis System (17), and the significance of each average was determined at $\alpha=0.05$ using the least significant difference (LSD) test.

Results and Discussion

Chemical composition of freeze concentrated versus evaporated milk To find out whether there were differences in the chemical compositions of evaporated versus freeze-concentrated milk, the chemical composition of each was measured as described in the materials and methods. As shown in Table 1, no differences in chemical composition were found between the two different milks. Total solid contents were 27.1 and 27.2% in the evaporated and the freeze-concentrated milks, respectively. The raw milk contained 12.4% total solids.

The moisture content of both freeze concentrated and evaporated milk was 72.9%, and the fat content was 8.5–8.7%. The 14.8% decrease of water content in both milks resulted in twice the concentration of most milk components relative to the control milk. The levels of all components were consistent with the standard composition of concentrated milk (18).

Changes in pH and titratable acidity (TA) Figure 1 shows a comparison of the pH values of control, freeze-concentrated and evaporated milks. The raw milk had a pH value of 6.56, reflective of a fresh state. Both the freeze-concentrated and the evaporated milks showed a significantly decreased pH value as 6.13 and 6.06, respectively ($p<0.05$) compared to the raw milk.

Figure 2 shows the TA (%) values for the raw milk

Table 1. Chemical compositions of milks produced by different concentration processes ¹⁾ (Unit: %)

Treatment of milk	Moisture	Protein	Fat	Lactose	Ash	Total solid
Raw ²⁾	87.65 ^a	3.15 ^c	3.60 ^c	4.80 ^c	0.80 ^c	12.35 ^c
Evaporated	72.89 ^c	7.78 ^a	8.65 ^a	9.10 ^a	1.72 ^a	27.11 ^a
Freeze-concentrated	72.85 ^c	7.81 ^a	8.50 ^a	9.08 ^a	1.75 ^a	27.15 ^a

¹⁾Means within each column having the same letter are not significantly different ($p>0.05$).

²⁾LTLT treated milk.

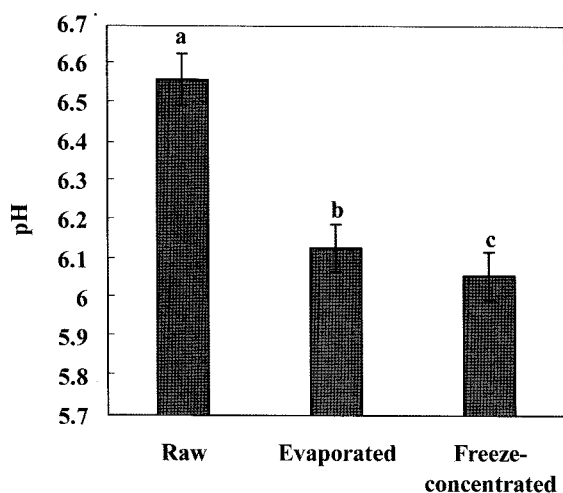


Fig. 1. Changes of pH in milk produced by different concentration processes.

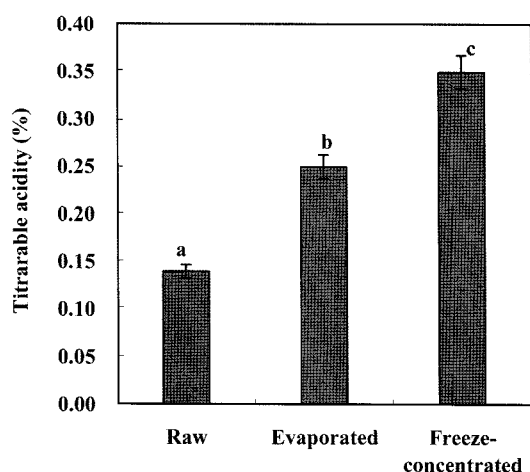


Fig. 2. Changes of titratable acidity (TA) in milk produced by different concentration processes.

(0.14), evaporated milk (0.25), and freeze-concentrated milk (0.35). As expected, the TA of the raw milk was markedly lower. The increased acidity in the evaporated milk was reported in the work of Walstra *et al.* (19). It is generally accepted that due to evaporation, the non-fat solid and organic acid contents increase the TA value. Among these factors, we postulated that organic acid content could account for the difference in TA between the freeze-concentrated and evaporated milks. It is possible that some organic acids in milk may be increased by the evaporation process through high temperature (more than 70°C for several min), while remaining stable throughout freeze-concentration process.

This lowering of pH (<6.2) causes increased coagulation and chemical crosslink formation resulting in increased colloidal phosphate and lactose isomerism with lactulose and organic acids due to heat treatment. In the evaporation of milk, the pH decreased and this would alter the charge of proteins therefore facilitating association reactions (20). Also, greater levels of β -lactoglobulin and α -lactalbumin association with casein micelles have been observed in

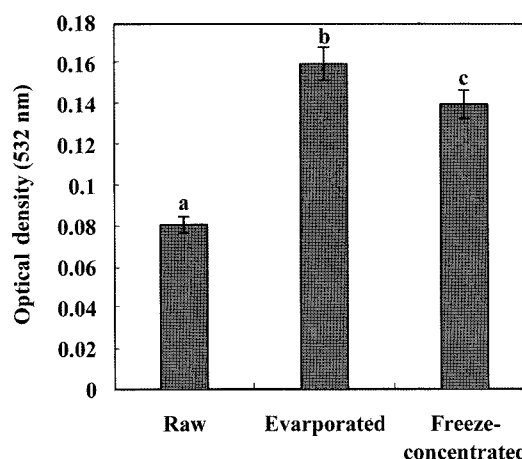


Fig. 3. Changes of thiobarbituric acid values (TBA) in milk produced by different concentration processes.

heated milks adjusted to a more acidic pH (21-23). Interestingly, pH changes in the freeze-concentrated milk were not remarkable relative to that of the evaporated milk.

Oxidation as determined by the TBA test Heat treatment associated with evaporated milk production is known to cause fat oxidation, resulting in an unpleasant odor and flavor. The potential of off-flavor and taste due to oxidation is a significant problem with the evaporation process which needs to be overcome. Therefore, we examined the degree of oxidation in concentrated milk as determined by the TBA test as shown in Fig. 3.

TBA absorbance was significantly higher in both the concentrated milks (the evaporated and the freeze-concentrated) than in the raw milk. Between the evaporated and the freeze-concentrated milks, the freeze-concentrated milk showed a significantly lower TBA value than the evaporated milk (0.14 vs. 0.16, respectively).

It is known that the velocity of milk fat autocatalytic oxidation increases the more concentrated the milk (24), which explains the increased TBA values for the concentrated milks in this study. Oxidative changes of milk fat may happen during storage of concentrated and dried dairy milk products containing fat. These changes are attributed to the presence of a high content of unsaturated fatty acids in milk fat. The oxidation of milk fat occurs in the presence of oxygen and is catalyzed by light and metal ions such as copper and iron. Based on the above information, to protect the fat from oxidation, packaging the concentrated milk in a partial vacuum is important and can be achieved by the partial replacement of oxygen with an inert gas such as nitrogen (25).

Short-chain free fatty acid (FFA) analysis The concentration of short-chain FFAs in the raw and experimental milks is shown in Table 2. The amounts of individual short-chain FFAs were significantly higher in both the freeze-concentrated and the evaporated milks, compared with the raw milk. When the freeze-concentrated milk was compared with the evaporated milk, all individual FFAs were significantly higher in the evaporated milk sample

Table 2. Concentration of short-chain free fatty acids (FFA) in milks produced by different concentration processes¹⁾

Treatment of milk	Short-chain FFAs concentration (Unit: ppm)				
	C ₄	C ₆	C ₈	C ₁₀	Total
Raw ²⁾	3.3 ^c	1.2 ^b	0.7 ^a	3.3 ^c	8.5 ^c
Evaporated	9.6 ^a	1.9 ^a	1.0 ^a	6.8 ^a	19.3 ^a
Freeze-concentrated	8.2 ^b	1.2 ^b	0.8 ^a	5.4 ^b	15.6 ^b

¹⁾Means within each column with the same letter are not significantly different ($p>0.05$).

²⁾LTLT treated milk.

than in the freeze-concentrated milk except for C₈ fatty acids ($p<0.05$).

Hydrolytic changes of milk fat in this product are rare since lipase, which catalyzes lipolysis (i.e., the release of free fatty acids), is activated by heat treatment during processing. Therefore, a greater amount of FFAs in evaporated milk was expected since heat treatment is necessary for the increase in lipase activity (25).

Free amino acid analysis The concentration of free amino acids is shown in Table 3. In both concentrated milks, the amounts of most individual amino acids increased significantly compared to the raw milk. However, there was no difference in the amounts of most amino acids between the evaporated and the freeze-concentrated samples except for

Table 3. Concentration of free amino acids in milks produced by different concentration processes¹⁾

Free amino acid	Treatment of milk (Unit: $\mu\text{mol/mL}$)		
	Raw ²⁾	Evaporated	Freeze-concentrated
Asp	17.1 ^c	46.8 ^a	48.3 ^a
Glu	76.2 ^c	142 ^a	145 ^a
Ser	18.8 ^c	50.4 ^a	48.5 ^a
Asn	3.2 ^c	12.5 ^a	11.2 ^a
Thr	16.6 ^c	39.3 ^a	34.5 ^a
Ala	10.1 ^c	22.4 ^a	21.9 ^a
Arg	25.5 ^b	32.9 ^a	33.3 ^a
Tyr	67.2 ^c	139 ^a	143 ^a
Val	51.1 ^c	124 ^a	128 ^a
Met	11.9 ^b	10.3 ^b	19.2 ^a
Ile	28.8 ^b	34.4 ^a	37.4 ^a
Leu	16.1 ^b	21.9 ^a	24.2 ^a
Phe	28.2 ^a	29.9 ^a	29.1 ^a
Trp	6.4 ^b	16.8 ^a	17.3 ^a
Lys	18.2 ^b	15.7 ^b	23.7 ^a
Total	395.4 ^c	749.3 ^a	764.6 ^a

¹⁾Means within each row with the same letter are not significantly different ($p>0.05$).

²⁾LTLT treated milk.

methionine and lysine. The freeze-concentrated milk resulted in less amount of methionine and lysine ($p<0.05$). It is known that in the evaporated milk, carbonyl-amino reactions between lactose and the amino groups of lysine are induced (5), and methionine also reacts to produce hydrogen sulfide and mercaptans. The decreased amount of lysine and methionine could be caused by these reactions in heat-treated milk products.

A high total solid concentration has a negative effect on the stability of milk proteins, especially if the system is exposed to high temperatures. If the milk has increased acidity as well, the proteins may completely destabilize and coagulate during sterilization (24). The higher amount of individual amino acids in concentrated milk may result from both increased protein concentration and increased acidity due to the heat treatment of evaporated milk.

Vitamin analysis The concentrations of most water-soluble vitamins were significantly decreased in both the evaporated and the freeze-concentrated milks compared with the control milk. When the two experimental samples were compared, L-ascorbic acid, niacin, and riboflavin in the evaporated milk were markedly lower than in the freeze-concentrated milk (Table 4).

As expected, all fat-soluble vitamins were significantly lower in the evaporated milk than in the raw milk. However, the concentrations of retinol and cholecalciferol in the freeze-concentrated milk were not significantly lower than in the raw milk (Table 5). Based on both water- and fat-soluble vitamin concentrations, most of vitamins may be affected by the heat treatment associated with the evaporated milk. Therefore, these results suggest that the stability of vitamins is higher in the freeze-concentrated milk than in the evaporated milk.

The nutritive value of dairy products is affected by the processing technology used. The modern technology used

Table 4. Concentration of water-soluble vitamins in milks produced by different concentration processes¹⁾

Water-soluble vitamin	Raw ²⁾	Evaporated	Freeze-concentrated
L-Ascorbic acid	3.87 ^a	1.20 ^c	2.23 ^b
Niacin	1.28 ^a	0.17 ^c	1.15 ^b
Thiamin	0.31 ^a	0.14 ^c	0.16 ^c
Riboflavin	1.28 ^a	0.21 ^c	1.22 ^b
Pyridoxine	0.08 ^a	0.00 ^b	0.02 ^b

¹⁾Means within each row with the same letter are not significantly different ($p>0.05$).

²⁾LTLT treated milk.

Table 5. Concentration of fat-soluble vitamins in milks produced by different concentration processes¹⁾

Fat-soluble vitamin	Raw ²⁾	Evaporated	Freeze-concentrated
Retinol	0.54 ^a	0.48 ^b	0.49 ^{ab}
Tocopherol	0.52 ^a	0.15 ^d	0.38 ^b
Cholecalciferol	0.03 ^a	0.02 ^b	0.03 ^a

¹⁾Means within each row with the same letter are not significantly different ($p>0.05$).

²⁾LTLT treated milk.

Table 6. Color changes in milks produced by different concentration processes¹⁾

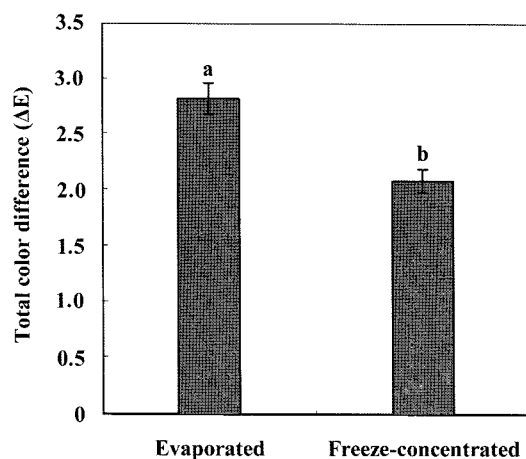
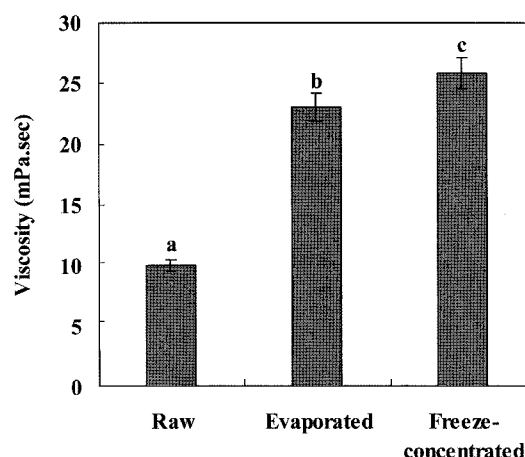
Treatment of milk	L-value	a-value	b-value
Raw ²⁾	92.67 ^a	-3.12 ^b	8.95 ^c
Evaporated	92.97 ^a	-3.96 ^a	11.62 ^a
Freeze-concentrated	91.50 ^b	-3.21 ^b	10.65 ^b

¹⁾Means within each column with the same letter are not significantly different ($p>0.05$).

²⁾LTLT treated milk.

in processing concentrated and dried products (vacuum evaporation, spray drying, multi-stage drying) is known to produce minimal undesirable changes in the milk components. For the most part, all milk components after evaporation and drying by such methods are concentrated without any negative effects. Still, lysine and lactose undergo minor changes due to Maillard reactions, but this has no major impact on the nutritional value of the product if the product is not allowed to absorb water.

Color analysis Table 6 compares the color values of freeze-concentrated and evaporated milks. Discoloration of milk was observed in all concentrated samples and it was significant. The L-value decreased significantly in the freeze-concentrated milk relative to the raw and the evaporated milks. The a- and b-values for the evaporated milk were significantly higher compared with those of other milks ($p<0.05$). Higher ΔE values (total color difference) indicate greater discoloration. A higher ΔE value of 2.82 was observed in the evaporated milk, whereas the freeze-concentrated milk showed less discoloration with a low ΔE value as 2.10 (Fig. 4). This phenomenon is considered to be due to lactose caramelization and Maillard's reaction due to thermal treatment in the evaporation process (26). This lactose denaturation in milk causes browning and a reaction product with a bitter, unpleasant, and burned taste in insufficiently controlled thermal processing (5). Previous reports have shown that consumers have the highest preference for fluid milks with the visual properties of whole milk (27). From our experimental results, the

**Fig. 4.** Changes of color in milk produced by different concentration processes.**Fig. 5.** Changes of viscosity in milk produced by different concentration processes.

process of freeze-concentration induced less discoloration of milk, which can preserve the milk's original appearance. Therefore, the freeze-concentration may have some advantages for the color of milk relative to the conventional evaporation process of concentration.

Viscosity analysis Figure 5 indicates changes in the viscosity of freeze-concentrated and evaporated milks. The viscosity value for the control was 10 mPa·sec. Both the freeze-concentrated and the evaporated milks showed significantly increased values of 23.0 and 25.8 mPa·sec, respectively. However, the difference between the freeze-concentrated and the evaporated milks was minimal. This result is in accordance with a similar report (6) which suggested that the elevated viscosity may be explained by the relatively long residence time of the evaporation or freeze-concentration process.

The concentration of milk is known to increase viscosity (19) and this phenomenon was apparent in our research using a rotational viscometer. Similar numerical results were found in a study by Chang and Hartel (28) involving the viscosity of concentrated skim milk. Increased viscosity may result in reduced flow rates, higher pressure drops, and decreased turbulence, such as evaporation, reverse osmosis, and ultra filtration, thus the extent of

Table 7. Comparison of sensory characteristics in milks produced by different concentration processes¹⁾

Sensory description	Treatment of milk	
	Evaporated	Freeze-concentrated
Cooked flavor	6.2 ^a	4.2 ^b
Oxidized flavor	4.4 ^a	4.3 ^a
Fat-taste	5.7 ^a	5.6 ^a
Oxidized taste	5.1 ^a	4.7 ^b
Off-taste	5.2 ^a	4.6 ^b

¹⁾Means within each row by with same letter are not significantly different ($p>0.05$).

The scale of sensory scores: 1=very slight, 2=slight, 3=slight-moderate, 4=moderate, 5=moderate-strong, 6=strong, and 7=very strong.

concentration may well be limited by the viscosity (29). The role of viscosity in the freeze concentration process in the relation to flow behavior needs to be investigated further.

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