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Effect of Freeze Concentration Process on the Physicochemical Properties of Milk

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### **Abstract**

Physicochemical properties were compared between freeze concentrated and vacuum evaporated milk through colour, brix, viscosity, freezing point and pH measurement. Brix and viscosity in each concentrated milk significantly increased due to solute concentration(P<0.05), and there was not much difference between freeze concentrated and evaporative one. Brix results were numerically modeled with the logarithmic regression: Y=-33.460+18.4513 · ln(X), R<sup>2</sup>=0.9798 and this model was fairly fit to predict the solute concentration in the middle of freeze concentration process. Freezing point significantly decreased according to concentration increment(P<0.05) and there was not the significant difference between freeze concentrated and evaporated one. Whereas, in colour and pH value, there were some differences between freeze concentrated and evaporative milk. Vacuum evaporated milk expressed higher discoloration comparing to freeze concentrated one. In pH values, evaporated milk showed the significantly decreased results comparing to freeze concentrated sample, whereas the pH value of freeze concentrated sample expressed the similar value to the reference milk.

### Introduction

In the dairy industry, the concentrated milk have been widely used as intermediate materials or final products for consumers. To obtain a concentrate, the water content in milk must be reduced by 70 %, and so there should be some operation to eliminate water(speer et al., 1998). Generally, there are three basic methods in the concentration process that can be used: the removal of water as vapor by evaporation, removal as a liquid(water) by reverse osmosis and removal as a solid(ice) by freeze concentration(van Mil & Bouman, 1990). In these methods, the evaporation is being mainly used for the concentrated milk in the dairy industry, but there are

some problems like discoloration, heat-coagulation and burnt flavor in the final products. Therefore, nowadays, the freeze concentration are being studied as non-thermal processing technology in the dairy industry. This freeze concentration process have several unique advantages: low chemical deterioration, reduced enzymatic activity, and no loss of volatile aroma components owing to the absence of liquid-vapor interfaces in the closed system as non-thermal processing(van Mil & Bouman, 1990). However, there have been few researches about the physicochemical characteristics about freeze concentrated milk up to now. Consequently, this research was conducted to provide the basic qualitative aspects of freeze concentrated milk through the comparison of freeze concentrated and evaporative milk.

### Materials and Methods

Sample preparation

Freeze concentrated milk was obtained from the multi-stage freeze concentrator described in the previous our research(Park et al., 2005). Evaporative sample was made by vacuum evaporator(R-205, Buechi, Swiss). During evaporation process, the vapour temperature maintained 70°C to prevent the major change of milk component under the vacuum state of 300 torr. Each freeze concentrated and evaporated sample of same total solids content was prepared to compare the physicochemical properties. All samples were precooled at 4°C for 12 hr for a consistency before the physicochemical analysis.

Physicochemical analysis

Colour, Brix, viscosity, freezing point and pH value were compared between freeze concentrated and vacuum evaporated milk in the same solute concentration.

### Results and Discussion

Colour value

Changes in colour due to each concentration process are presented in Table 1. Discoloration of milk was observed in all concentrated samples and significant in higher concentrate. Especially, evaporated milk expressed more discoloration comparing to that of freeze concentrated one in the same total solids content. This phenomena is considered to originate in the consequence of lactose caramelization and Maillard's reaction due to thermal treatment in the evaporation process. By the report of Caric(1994), this lactose denaturation in milk causes browning and a reaction product with bitter, unpleasant and burned taste in insufficiently controlled thermal processing.

Table 1. Changes in colour value of freeze concentrated and evaporative milk

Colour value	Re	TS, 17%		TS, 27%	
		FC	Eva	FC	Eva
L	92.67±0.121°	92.45±0.035 <sup>d</sup>	93.28±0.083 <sup>a</sup>	91.45±0.082 <sup>e</sup>	92.97±0.101 <sup>b</sup>
a	-3.12±0.044 <sup>a</sup>	-3.10±0.016 <sup>a</sup>	-3.31±0.036°	-3.19±0.015 <sup>b</sup>	-3.96±0.017 <sup>d</sup>
b	8.95±0.020 <sup>e</sup>	9.60±0.049 <sup>d</sup>	10.04±0.155 <sup>c</sup>	10.63±0.075 <sup>b</sup>	11.62±0.095 <sup>a</sup>

Different superscript in same line indicate the significance difference among the experimental results

$$\Delta E = \sqrt{(L - L')^2 + (a - a')^2 + (b - b')^2}$$
 (eq. 1)

L, a, b: each colour value of reference milk, L', a', b': each colour value of concentrated milk

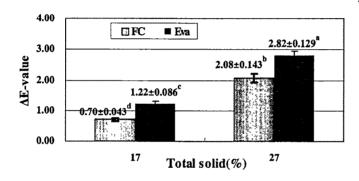


Fig 1. Total colour difference ( $\triangle E$ ) of each freeze concentrated and evaporated milk.

Total colour difference ( $\Delta E$ , Fig 1) was calculated to compare the overall changes in each colour value (L, a and b value) with eq. 1. High  $\Delta E$ -value means more colour changes in the product comparing to lower one. The highest value was obtained as 2.82 in the evaporated milk with 27% of total solids, whereas freeze concentrated milk expressed the significantly low  $\Delta E$ -value comparing to evaporated ones. Consequently, the freeze concentration process induced lesser discoloration of milk which can preserve the similar appearance to non-thermal treated fresh milk. Therefore, the freeze concentration is recommended to have some advantages in colour appearance of milk than conventionally thermal concentration processing.

# Brix

Fig 2 shows the evolution of brix in each concentration process. Brix significantly increased according to the increment of total solids(P<0.05) and there was no difference between freeze concentrated and evaporated milk excepting the 27 % of total solids. Brix results in freeze concentration process were numerically modeled as eq. 2 representing high R<sup>2</sup> of determination.

In our research, this numerical model was usefully utilized for predicting the total solids(%) before measuring the real total solids content in product through infrared vacuum drying. By the previous our research(Park et al., 2005), the numerical correlation between brix and total solids in the freeze concentrated milk was considered as fit for the prediction of concentration progress in industrial progress.

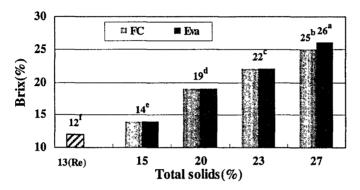


Fig 2. Brix profiles due to total solids in freeze concentrated and evaporative milk.

Y=-33.460+18.4513·
$$\ln(X)$$
, R<sup>2</sup>=0.9798  
X: Brix(%), Y: Total solids(%) (eq. 2)

### Viscosity

Viscosity significantly increased according to solute concentration in each freeze concentrated and evaporative milk represented in Fig 3(P<0.05). Viscosity results were linearly modeled with eq. 3.

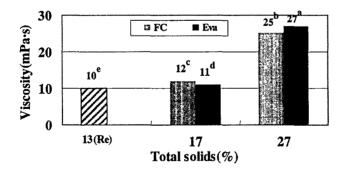


Fig 3. Changes in viscosity due to solute concentration.

$$Y = 1.115 \cdot X - 5.5256$$
 (R<sup>2</sup> = 0.9753)  
X: Total solids(%), Y: Viscosity(mPa·s) (eq. 3)

However, it was difficult to recognize the reliable difference between freeze concentrated and evaporative milk. Concentration of milk causes the increment of viscosity and Newtonian fluid(Walstra etal., 1999) and this phenomena was found in our research with a rotational viscometer. Similar results were found in the research of Chang & Hartel(1997) that meant the increment of viscosity in concentrated skim milk. Viscosity is important factor in milk affecting the consumers' appeal. In addition, the flow behaviour with the proper viscosity is essential in freeze concentration process and so further researches are required.

## Changes in freezing point(FP)

Evolution of FP due to concentration is described in Fig 4. Freezing point of each concentrated milk significantly decreased and the results of freeze concentration were numerically modeled with eq. 4. Similar results were obtained in the research of Chen et al(1996) that reported the FP in whole milk due to concentration. This FP in concentrated milk should be adequately considered in the multi-stage freeze concentration to let the sample have the similar temperature to its' freezing point in the recrystallisation process.

$$Y = -0.0096 - 0.0304 \cdot X - 0.008 \cdot X^2$$
 (R<sup>2</sup> = 0.9753)  
X: Total solids(%), Y: Freezing point(°C) (eq. 4)

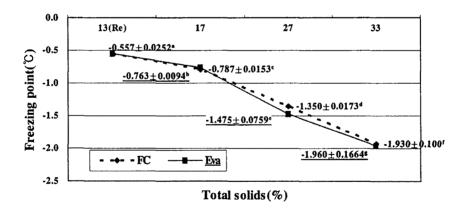


Fig 4. Evolution of freezing point due to solute concentration,

#### Changes in pH

Fig 5 describes the changes in pH due to solute concentration. Evaporated milk showed the significantly decreased pH value(P<0.05), whereas there was not much difference between the reference and the freeze concentrated milk. The increased acidity in the evaporated milk originates in the postulation of the work from Walstra et al., (1999) whereas the lowering of

pH(<6.2) causes the coagulation and chemical cross links in the form of increased colloidal phosphate and lactose isomerism with lactulose and organic acids due to heat treatment. However, in the freeze concentrated milk, pH changes were not significant. Therefore, in our research, it is considered that the freeze concentration process can provide the similar chemical properties in concentrated milk similar to fresh milk in the aspects of pH value. Further researches are required to elucidate the chemical properties of freeze concentrated milk associated with pH value.

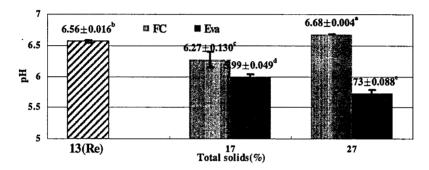


Fig 5. Evolution of FPD due to solute concentration.

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