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Effect of the recrystallization of ice on the freeze concentration process of milk in the lab-scale operation

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Abstract

This study was carried out to develop the efficient freeze concentration process of milk through controlling the recrystallisation phenomena of ice. Freeze-concentration was progressed with multi-stage freeze concentrator and there was artificial temperature control to induce recrystallisation phenomena. In each stage of freeze concentration process, the regular recrystallisation time was fixed as 1, 2, 4 and 8 hr to compare the solute increment, yield, brix and ice-crystal size among experimental conditions. Higher concentration as total solids was observed due to the elapse of recrystallisation time, and the maximum total solids in final products: 32.67% was obtained at the ripening time of 8 hr in two-stage process. This result was excessively high concentration comparing to the existing researches and presented the possibilities of milk freeze concentration in the dairy industry. The results of brix and ice-crystal size showed the direct correlation with the recrystallisation time that meant the increased processing time showed the increment of brix and ice-crystal size. Obtained results were numerically modelled to predict the progress of concentration in the industrial process and all of them had fairly high R2 of determination. Therefore, we regarded that these numerical models could be utilized for the development of efficient technology in industrial freeze concentration process.

Introduction

In the dairy industry, the concentration process has been widely used to extend the shelf life, accommodate the transportation and make the processed dairy products like condensed milk and powered milk. However, this concentration could cause the major physicochemical, structural and nutritional modifications to the milk if it is progressed by thermal processing like evaporation(Velez-Ruiz and Barbosa-Canovas, 1998). Therefore, nowadays, non-thermal processing technology is required to preserve the quality aspects of concentrated milk in the dairy industry, and so freeze concentration of milk is being studied as one of them which separates water as crystallized ice without thermal treatment. However, its' application has been limited by low solute concentration in final product: 10-17 % (Zhang and Hartel, 1996). In addition, freeze concentration process requires high capital costs and so it is the main disadvantage of its' industrial utilization(van Mil&Bouman, 1990). Nevertheless, there has been few researches about the freeze concentration process of milk including the solute concentration and yield in final products which is important factor in productivity and economical efficiency. Consequently, this study was conducted to examine the influence of ice-recrystallisation on the freeze concentration process of milk to develop the efficient processing method.

Materials and Methods

LTLT(low temperature long time pasteurized, 63℃, 30 min) milk having initial total solids of 13% was pre-cooled at 4℃ for 12 hours before experiment. Freeze concentration was progressed in the multi-stage freeze concentrator described in Fig 1. Ice crystals were continually formed on inner wall of the stainless steel vessel by circulating coolant and removed with the rotating scrapper(50-60 r.p.m.). Removed ice crystals were recrystallised in the central part of concentrator for constant time(1, 2, 4 and 8 hr) in each experimental condition. Coolant temperature was varied with regular procedure to accelerate the recrystallisation of ice crystals and this operation was defined as a heat and cold shock in our study. After each recrystallisation process, ice crystals and milk concentrate were separated by filtration process coupled with stainless steel net(200mesh) and vacuum pump. In this experiment, the multi-stage process(one and two stage) was conducted to increase the concentration. Degree of concentration was determined as total solids in final products by infrared vacuum-drying. yield, brix and ice crystal size were measured to predict the proper processing methods in freeze concentration and their results were statistically evaluated with Nalimov Data Analysis Program(simplified t-test) and Duncan's Multiple Range test for the comparison of significance difference in experimental conditions.

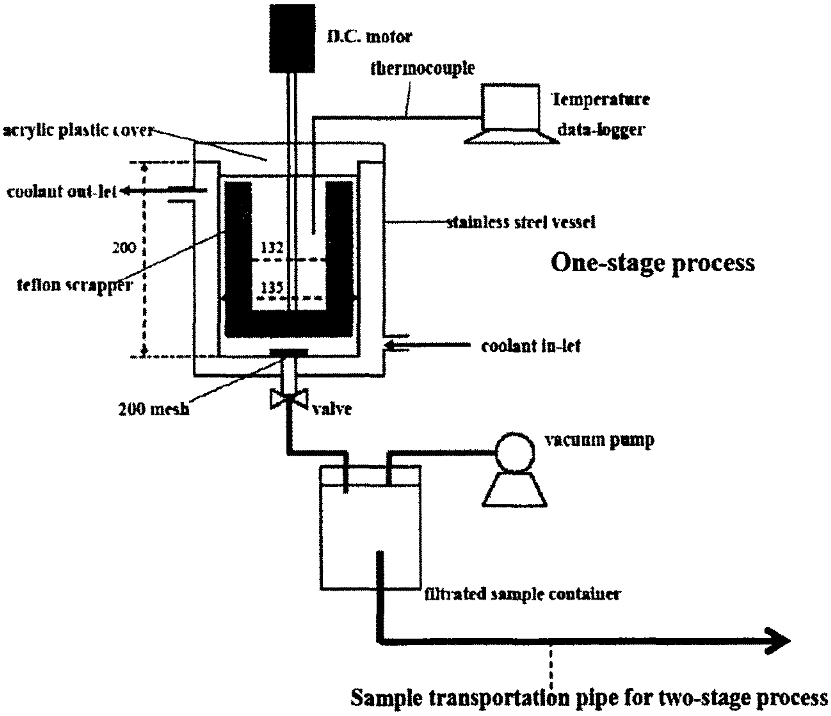

Fig 1. Schematic diagram of multi-stage freeze concentrator.

Results and Discussion

Total solids and yield

Fig. 2 and 3 represents the influence of recrystallisation time on total solids and yield in the one and two stage freeze concentrated products, respectively. The solute concentration as total solids in both stages linearly increased due to the duration of recrystallisation time and their results were numerically modelled as linear regressions(eq. 1 and 2) with comparatively high R^2 of determination.

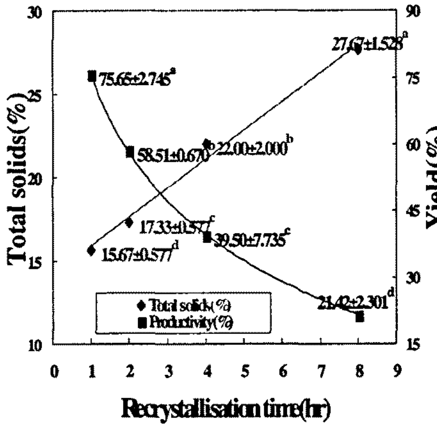

Fig 2. Profiles of total solids and yield due to recrystallisation time in one-stage process.

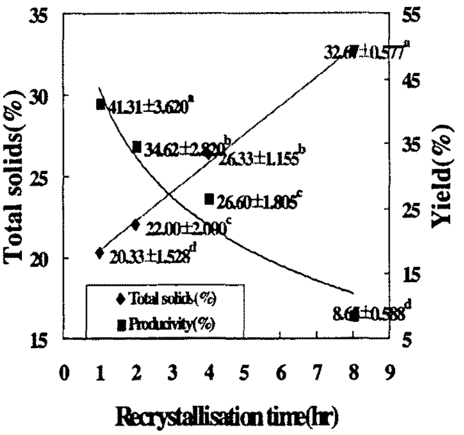

Fig 3. Profiles of total solids and yield due to recrystallisation time in two-stage process.

$T_1=1.728 \cdot X+14.188$, $R^2=0.9875$ (eq. 1) $T_2=1.775 \cdot X+18.677$, $R^2=0.9954$ (eq. 2)

T_1 : Total solids in one-stage process(%), T_2 : Total solids in two-stage process(%), X: Ripening time(hr)

$P_1=76.03-26.21 \cdot \ln X$, $R^2=0.9996$ (eq. 3) $P_2=43.70-15.29 \cdot \ln X$. $R^2=0.9407$ (eq. 4)

P_1 : Productivity in one-stage process(%) T_2 : Productivity in two-stage process(%), X: Ripening time (hr)

Similar results of total solids were reported in the research of Wideham and Cochet(2003) that showed the concentration increment in saccharose solution due to processing time. Whereas, the yield significantly decreased according to recrystallisation time(P<0.05), and so it showed the negative correlation with total solids profile(eq. 3 and 4). Decreased yield had its' origin in excessive ice-slurries in the concentration vessel and there should be appropriate methods to maintain the proper amount of ice-slurries during freeze concentration process. In our experiment, it was possible to increase the solute concentration in milk to the maximum level of 32.67% comparing to initial total solids of 13% in reference milk. However, further researches are required to raise the yield in high solute concentration from the viewpoint of economical efficiency.

Brix

Changes in brix during one and two-stage recrystallisation process are presented in Fig 4 and 5, respectively. They significantly increased according to recrystallisation time(P<0.05) and had the similarities to the profiles of total solids representing logarithmic regressions of eq. 5 and 6. These equations were considered as fairly fit model for the prediction of solute increment with the high R2 of determination during freeze-concentration. Especially, in this experiment, brix measurement was determined as comparatively convenient method just using refractometer and made it possible to estimate the progress of concentration in industrial freeze-concentration process.

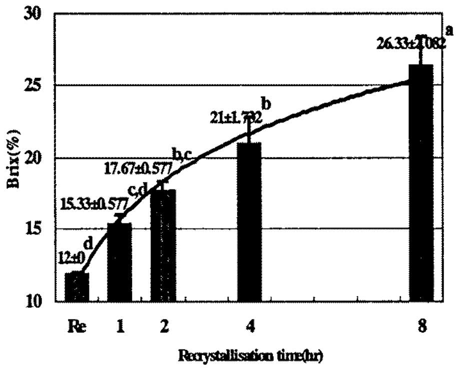

Fig 4. Changes in brix of concentrated milk during during one-stage recrystallisation process.

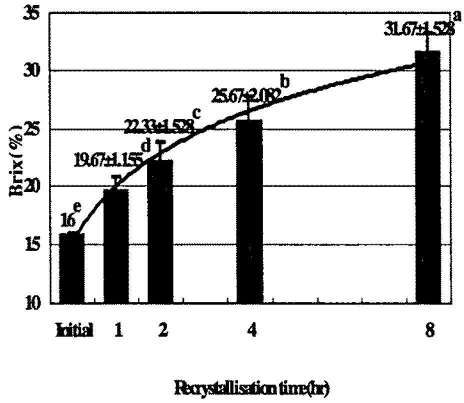

Fig 5. Changes in brix of concentrated milk two-stage recrystallisation process.

$$B_1=14.63+5.24\cdot \ln X,\ R^2=0.9665 \quad (eq.5) \qquad B_2=18.93+5.68\cdot \ln X,\ R^2=0.9629 \quad (eq.6)$$

B_1: Brix in one-stage process(%), B_2: Brix in two-stage process(%), X: Ripening time

Ice-crystal size

In the recrystallisation process, ice-crystal size significantly increased due to the elapse of processing time(Fig 6 and 7, $P<0.05$) and its' evolution was numerically modeled with logarithmic regression of eq. 7 and 8. In the growth rate, more rapid growth of ice-crystal was observed in the beginning of recrystallisation process and these results were in accordance with the research of Hartel and Espinel(1993) that reported ice-crystals initially grew rapidly in the early stage of freeze concentration process. In our research, there was the direct correlation between total solids and ice-crystal size that meant the increment of ice-crystal size resulted in higher concentration. Heat and cold shock in our experiment was regarded as the major effects on ice-crystal size increment with artificially induced recrystallisation. In addition, the caution was taken to maintain the low freezing rate for the large ice-crystal sizes from the research of Woinet, Andrieu and Min(1998) that meant the low freezing rates lead to large ice-crystal size. Therefore, it was considered that this temperature control in heat and cold shock operation could promote the separation of ice-slurry and solid components which can increase the efficiency of freeze-concentration process. The proper temperature variation near the freezing point of sample was considered as important factor which can accelerate the solute concentration during freeze concentration process. Regarding this artificial recrystallisation with heat and cold shock operation, further researches are required to increase the efficiency in the freeze concentration process.

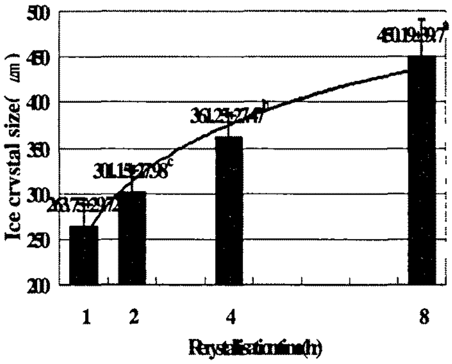

Fig 6. Evolution of ice-crystal size due to one-stage recrystallisation time.

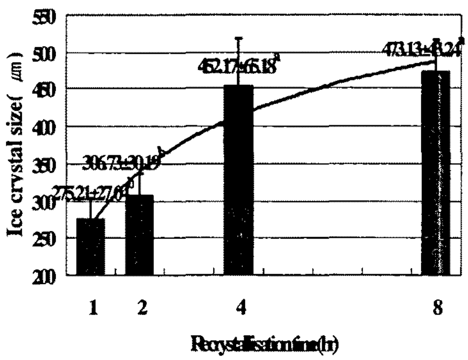

Fig 7. Evolution of ice-crystal size due to one-stage recrystallisation time.

$C_1=251.17+89.36\cdot \ln X,\ R^2=0.9664$ (eq.7) $C_2=265.93+106.64\cdot \ln X,\ R^2=0.9049$ (eq.8)

C_1: Ice-crystal size in one-stage process(μm), C_2: Ice-crystal size in two-stage process(μm), X: Ripening time(hr)

Acknowledgement

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