Behaviour of Ice Crystal Growth in a Vertical Finned Cylindrical Freeze Concentrator

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Abstract. Behaviours of ice crystal growth at two different operating parameters namely coolant temperature and circulation time were investigated for progressive freeze concentration (PFC) of glucose solution through a vertical finned crystallizer (VFC). Two determinant parameters which are ice production rate (m_u), and water recovery (W_R) were used to illustrate the behaviours of ice crystal growth in this study. From the result, higher ice production rate (m_u) and water recovery (W_R) were achieved at lower coolant temperature. On the other hand, longer circulation time resulted in lower ice production rate (m_u), but at the same time increased the water recovery (W_R). The maximum ice production rate (m_u) and water recovery (W_R) attained through this study were 1.522 $gm^{-2}s^{-1}$ and 51.131 %, respectively.

Introduction

Freeze concentration (FC) is a technique used to concentrate liquids or solutions by freezing water into pure ice and subsequently separating the part of frozen water from the concentrated liquid. The greatest attraction of FC is it could preserve the thermally sensitive components in the concentrate due to the low process temperatures involved, as well as low energy requirement (0.33kJ/g water) as compared to the conventional concentration method by evaporation [1].

There are two basic types of freeze concentration which are suspension freeze concentration (SFC) and progressive freeze concentration (PFC) [2]. In SFC, small size ice crystals are produced in the suspension of mother solution while PFC continuously produces ice crystal layer by layer on a cooled surface until it forms a single and large block of ice. Hence, separation of ice crystal from the concentrated solution becomes much easier in PFC than SFC [1]. As PFC offers a simpler separation step, it has been suggested in recent studies to associate the future application of FC more with the progresses in the configuration of PFC system than SFC system.

In PFC process, it is a great importance to predict the behaviour of crystal growth as it could facilitate future planning of the process which involves the determination of suitable operating conditions and equipment size to meet the product demands. Theoretically, the formation of ice as an aqueous solution brought into contact with a cooled surface is initiated with ice nucleation. Once the ice embryos appear, the cooled solid surface becomes a favourable place for ice crystallization since the fusion heat can be given directly to the solid surface through conduction. During this stage, the separated ice embryos rapidly spread along the cooled surface to form thin films of ice until it entirely covers the surface. Next, the thin ice film starts to grow thicker and continuously until it forms a visible ice layer. After certain period of time, the increasing thickness of the ice layer will promote higher resistance for heat transfer and the ice growth starts to decline afterwards.

The principal aim of this paper is to study the changes in the ice crystal growth in relation to concentration of glucose solution through PFC at different coolant temperature and circulation time. In this particular study, a new PFC crystallizer named vertical finned crystallizer (VFC) was used to provide space for ice crystallization. Two determinant parameters which are ice production rate, m_u ($gm^{-2}s^{-1}$), and water recovery, W_R (%) were used to characterize the behaviour of ice crystal growth.

Materials and Methods

Materials. Glucose solutions at a concentration of 8 %Brix were used as the target solution to be concentrated. On the other hand, 50% (v/v) of ethylene glycol-water solution was used as a coolant to supply cooling energy to the system. Both glucose powder (D(+)-Glucose) (QRec, Malaysia) and ethylene glycol (QRec, Malaysia) were purchased from local suppliers.

Experimental Setup. Fig. 1 shows the experimental setup of the PFC process. The setup comprises of the new crystallizer (VFC), a refrigerated waterbath (Scientz, China), a feed tank, a peristaltic pump (Cole-Palmer, Canada), silicone tubes (Masterflex, USA) and a data acquisition tool, Picolog (Picolog USB TC-08, United Kingdom) connected to a computer for temperature profiling. The VFC is made of stainless steel with 30 cm height and 8 cm diameter. Four vertical fins of 30 cm height, 2 cm length and 1.5 cm width are installed inside the VFC to provide larger surface contact for heat transfer between the coolant and the solution, where the fins increase the surface area by 63.7%.

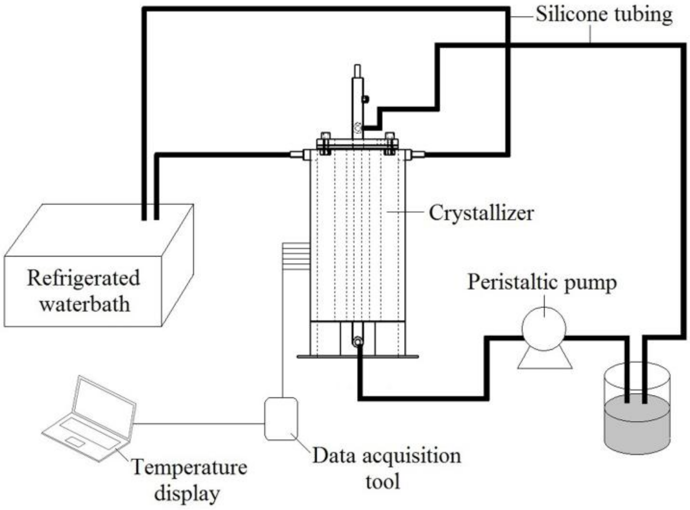

Fig. 1. Experimental setup for the new PFC system

Experimental Procedure. Glucose solution was initially prepared at the desired concentration (% Brix). Two portions of glucose solution with similar concentration were next prepared, where the first portion was kept in a freezer to maintain its temperature near the freezing point of water, while the second portion was frozen into solid form (ice cubes). On the other hand, the temperature of coolant in the waterbath was set to the desired temperature before it was circulated within the cooling jacket. Prior to be fed into the crystallizer, the cooled glucose solution was mixed with glucose solution ice cubes in the feed tank to maintain the low temperature. The glucose solution was then fed into the crystallizer by using the peristaltic pump at 2800 ml/min through the silicone tube. When the crystallizer and the silicone tube were filled with the solution, the feeding process was stopped by closing a valve located at the feed tank. At this time, the glucose solution was left in circulation within the crystallizer and the silicone tube for crystallization to occur for a period of time. At certain designated time, the circulation was stopped and the concentrated glucose solution in the crystallizer was collected. For analysis purpose, volume of the collected concentrate and ice formed were measured before collecting the samples of both products. Then, the collected samples were analysed for their glucose concentration by using a refractometer (MA871, Romania, Europe). The experimental procedure was repeated at different coolant temperature and circulation time.

Data Analysis.

Ice Production Rate (m_u). In PFC, ice production rate portrays the ability of the process to concentrate the targeted solution through water removal. In this study, the ice production rate, m_u was determined using the following equation:

$$m_u = M_i / [(2\pi rh + 8xh)t] \tag{1}$$

where m_u is the ice production rate per unit surface area ($gm^{-2}s^{-1}$), M_i is the mass of ice produced in grams, x is the length of vertical fins in meter, r and h are the radius height of the VFC is meter while t is the freezing time of each run of experiment in seconds.

Water Recovery (W_R). As water is being removed through freezing in PFC of glucose solution, it is also important to calculate the percentage of water recovered through the process to represent the degree of crystal growth. The water recovery (%) can be calculated from Eq. (3) below:

$$W_R = (W_i/W_o) \times 100 \tag{2}$$

where W_o is the mass of water in the initial solution while W_i is the mass of water in the ice crystal formed.

Results and Discussion

According to Radhakrishnan and Balakrishnan [3], the basic characteristic of PFC process is normally described by the rate of crystal growth, in which this rate is mainly determined by heat and mass transfer at solid-liquid interface. Fig. 2 shows the trends of ice production rate (m_u) and water recovery (W_R) at different coolant temperature and circulation time.

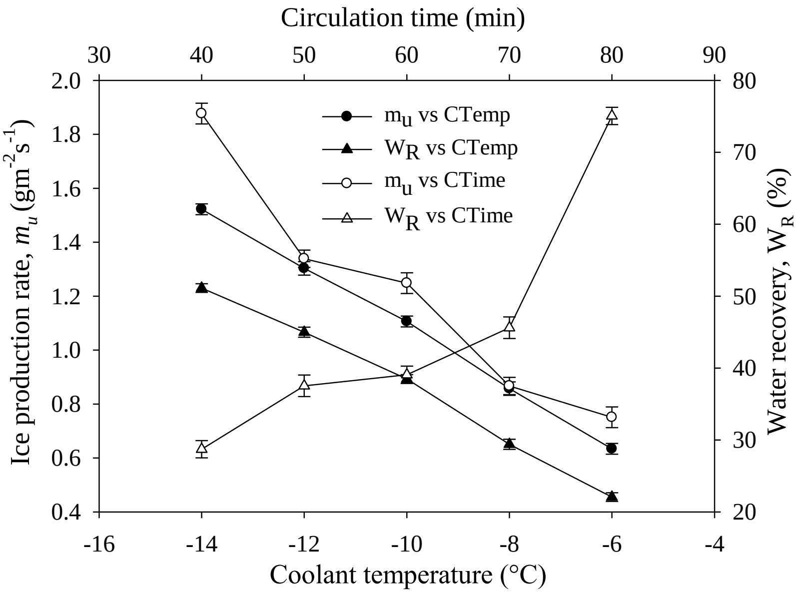

Fig. 2. Ice production rate (m_u) and water recovery (W_R) as a function of coolant temperature (°C) and circulation time (min)

In PFC, low temperature of coolant plays an important role not only in providing initial supercooling for the ice nucleation, but also in maintaining lower surface temperature for heat transfer. From the figure, it can be observed that lower coolant temperature resulted in higher ice production rate and water recovery, as high as 1.522 $gm^{-2}s^{-1}$ and 51.131 % respectively. The highest recorded value of ice production rate (m_u) in this analysis was found a little higher than the maximum values recorded from the previous studies done by Hernández et al. [4]. The value thus indicates that the VFC is competent to be compared with the existing PFC crystallizers in terms of ice production. As stated by Ramos et al. [5], the big difference between the wall temperature and the solution temperature caused by the lowered coolant temperature promotes the rate of crystal growth. This phenomenon clearly explains the increase in the ice production rate (m_u) and water recovery (W_R) as the coolant temperature decreases. In the perspective of concentration process, crystal growth rate which is higher than the solute outward movement is unfavourable since it could trap the solute into the ice crystal formed. Hence, it is important to determine the suitable cooling temperature for PFC process, where the demand of ice production and ice purity comply with each other.

In PFC process, circulation time also plays an important role in providing an efficient separation process. As already shown in Fig. 2, the ice production rate (m_u) decreases from 0.581 to 0.405 gm^{-2}s^{-1} over time. On the other hand, longer circulation time resulted in higher water recovery (W_R) as high as 32.354 %. Generally, longer circulation time could provide higher concentration efficiency to the PFC process. By providing longer circulation time, the ice layer grows thicker, leaving the unfrozen solution in a state approaching the saturation level. This condition also explains the increase in water recovery (W_R). In addition, the thickness of ice produced is also one of the resistances to heat transfer, in which higher thickness of ice at longer circulation time resulted in higher heat transfer resistance [6]. Hence, lower rate of ice production is obtained at higher circulation time. Due to the low mass diffusion coefficient of solution and high thermal conductivity of ice, the rate of mass transfer was found to be slower than the rate of heat transfer. Hence, longer and adequate freezing time is needed to enhance the concentration rate as well as to remove water from the original solution.

Summary

In PFC, it is important to understand the kinetics of the impurity or solute incorporation in the crystal formed during the crystallization, as well as to observe the relationship between ice crystal growth rate and the impurity concentration in the ice crystal. Since freezing temperature and concentration profiles near the solid-liquid interface control the supercooling condition, it can also be concluded that the heat and mass transfer rates at the solid-liquid interface basically influence the crystal growth rate. The ice production rate (m_u) and water recovery (W_R) which illustrate the crystal growth rate in this study were found to be dependent on the coolant temperature and circulation time. The maximum ice production rate (m_u) and water recovery (W_R) attained through this study were 1.522 gm^{-2}s^{-1} and 51.131 %, respectively.

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