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The Effect of Coolant Temperature and Stirrer Speed for Concentration of Sugarcane via Progressive Freeze Concentration Process

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Progressive freeze concentration system engaging an anti-supercooling holes crystalliser was used to concentrate sugarcane juice in order to increase its sugar content and achieve highly concentrated sugarcane juice without initial supercooling or ice lining process. The other function of this process is to retain the aromatic compound, quality and avoid operating pressure compared to using another separation process like evaporation and reverse osmosis. The process was conducted with a specific process condition including coolant temperature, stirrer speed, constant initial concentration and process time. The parameter of coolant temperature and stirrer speed was in the range of -6 to -14 °C and 150 to 350 rpm, meanwhile the constant process time was set at 180 min and constant initial concentration at 12 - 13 % Brix. The effects of coolant temperature at stirrer speed were evaluated and observed based on the value of effective partition constant, K-value, and concentration efficiency of the sugarcane juice. This system achieved its best performance at intermediate coolant temperature which is -8 °C and stirrer speed of 300 rpm, referring to the best K value and concentration efficiency with the values of 0.438 and 51.15 %. The heat transfer analysis was done by determination of overall heat transfer coefficient, where the highest overall heat transfer coefficient, U_o was found at coolant temperature of -8 °C and stirrer speed of 350 rpm, implying that the smallest thickness of ice was formed on the wall of the crystalliser at this condition, which favours the heat transfer in the process.

1. Introduction

Many innovative techniques have been developed for food processing, but technically the process that are commercially available for the concentration of liquid foods include evaporation, freeze concentration, reverse osmosis, and ultrafiltration. Evaporation is considered to be the most economical and most widely used method of concentration. It involves the removal of water from a non-volatile component by turning the water into vapor through the application of heat and/or vacuum. It is not suitable for food products with very delicate flavours (Sánchez et al., 2011). Reverse osmosis process consumes high energy for the attainment of the osmotic pressure and requires high maintenance to overcome membrane clogging and high cost for membrane replacement (Randall and Nathoo, 2015). Freeze concentration has the highest potential for the concentration of aroma-rich liquid foods, including fruit juices, coffee, tea, and selected alcoholic beverages due to the low temperature used (Miyawaki et al., 2016).

The term freeze concentration is regularly utilised conversely with the term freeze crystallisation. In fact, freeze concentration is a particular type of freeze crystallisation in which the crystallised substance is physically removed, and a more concentrated solution will be left behind (Jusoh, 2010). According to Miyawaki et al. (2016), by using freeze concentration, it can be related to possible energy savings, as it requires less energy compared to evaporation, and it also has lower capital costs and requires less maintenance because it operates at low temperatures where corrosion is not significant. Among all the existing methods for food concentration, freeze concentration, is the most advantageous technique to obtain high quality products without appreciable loss in taste, aroma, colour, or nutritive value (Otero et al., 2012). Freeze concentration also can be divided

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into two process methods which are suspension freeze concentration (SFC) and progressive freeze concentration (PFC).

Suspension freeze concentration is well known in industrial processes and it has been used for more than a decade in food industry. The SFC method consists of two main units which are the crystalliser and the washing unit and the principle of SFC is to have the ice crystal nucleation followed by the second phase of growing the ice crystal from the solution (Sánchez et al., 2010). The process is complicated because it involves many unit operation, long operation time and high cost. PFC is more practical and efficient to be applied due to less operation time and it is easier to handle. PFC produces ice crystal layer by layer on the cooled surface depending on the temperature and the operation time of the process until it forms a large single block of ice (Miyawaki et al., 2012). According to appropriate process condition, the formation of ice crystal would reflect the rate of growth and nucleation to produce pure ice. The feature of ice crystal is in the form of a single block, it would be easier to separate the ice crystal from the solution and thus resulting in low operation and capital cost (Jusoh, 2010). Despite its simplicity and less cost, the productivity of PFC is comparatively lower than SFC. The development on the improvement of the design of PFC is still on-going to find the most suitable design to increase its productivity.

This present paper aims to study and evaluate the effect of coolant temperature and rotation speed of a new design of crystalliser towards the process efficiency and heat transfer in the PFC of sugarcane juice. More specifically, a rotating crystalliser with anti-supercooling holes has been invented to overcome initial supercooling. The function of holes is to provide room for the process of nucleation and crystallisation of pure water molecules. In this crystalliser, the water molecules are cooled down to below freezing point earlier in the holes than the average bulk solution, which becomes the seeds for further ice crystallisation. The chance for ice nucleation is higher with lower opportunity for the contaminant to be trapped in the ice, resulted from the higher freezing point of the pure water molecules compared to the solution that contains foreign solute molecule (Hamid et al., 2015b). The advantage of this feature is that the operation becomes easier because the ice lining process can be neglected. The initial supercooling can also be avoided, thus preventing operation at super low temperature to accommodate ice nucleation. This will improve its productivity and reduce operation cost.

2. Materials and Methods

2.1 Materials and Equipment

Sugarcane juice was purchased from a store at Taman Universiti which was collected from the same harvest situated in Johor. About 1.5 L sugarcane juice was used throughout the experiment as raw material and 50 vol% of ethylene glycol and water mixture was used as coolant in the refrigerated water bath. Ethylene glycol is commonly applied to transfer heat in very low temperature processes. There are three main parts in the laboratory setup for the PFC (Figure 1) which are the newly designed crystalliser called Anti-Supercooling Holes Crystalliser (ASHC), refrigerated waterbath and a motorised rotator, while refractometer in unit % Brix was used for measurement of the concentration of the solution.

2.2 Experimental Procedure

The experiment started with the sugarcane juice first kept in the refrigerator at 2 °C or 3 °C as the initial temperature of the target solution should be near the freezing temperature of water. The cooled sugarcane juice with constant initial concentration of 12 - 13 % Brix was fed directly into the crystalliser then immersed in the water bath at the desired cooling temperature during operation. Motor rotator was engaged to rotate the crystalliser and to make the solution mixed well in the range of 150 to 350 rpm. Thermocouples were attached to the crystalliser to measure the temperature (-6 to -14 °C) which were connected to a computer for the data to be displayed. A set of baffles was introduced to improve circulation in the crystalliser as can be seen in Figure 1b. In order for the crystallisation process to take place, the filled crystalliser was immersed into the cooling bath at the desired temperature and rotated for 180 min of operation time. After 180 min, the rotation was stopped and the crystalliser was taken out from the water bath to be thawed. The concentrated solution was drained out completely and a sample of the ice layer produced was collected. The thickness of the ice layer formed was measured using Vernier caliper and a sample of ice produced was taken out for further analysis. Lastly, in order to determine the concentration of sugar in the concentrate and also in the ice, % Brix refractometer was used.

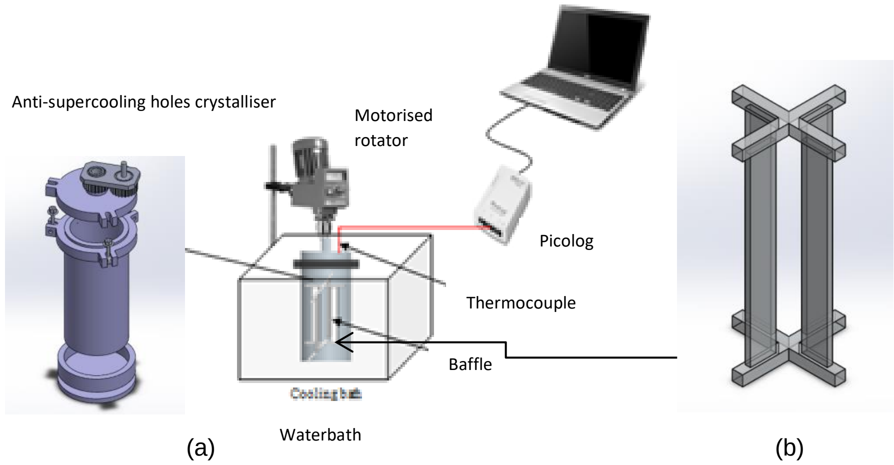

Figure 1: (a) Schematic diagram for experimental setup (b) Illustration of baffle

2.3 Calculation for efficiency analysis

Products from the experiment which are the concentrated sugarcane juice and ice layer that was formed as a layer on the inner wall of the anti-supercooling holes crystalliser were both collected to analyse the efficiency of PFC method. Effective partition constant, K evaluates the efficiency of the system which is related to the quality of the ice produced. The calculation of K-value between the ice and the concentrated liquid is formulated as follows Eq(1) (Bae et al., 1994):

$$K= C_S/C_L \tag{1}$$

where C_L is defined as the concentration of the concentrated solution of glucose in sugarcane juice and C_s is the concentration of the ice sample. As the equation is integrated, another equation is produced based on the concentration and volume of target solution and concentrated sample in order to find the value of K. The equation is stated as Eq(2) follows:

$$(1-K) \log (V_L/V_o) = \log (C_o/C_L) \tag{2}$$

The value V_o and C_o are the volume and the solute concentration at the beginning in the solution phase. V_L and C_L are the volume and solute concentrated after the process done. Sugar increment is also one of the important determinant parameters to be determined other than the quality of ice produced (Jusoh et.al, 2014). The increment of sugar concentration was determined in percentage and calculated using Eq(3).

$$Eff(s)= (C_L– C_o) /C_o \times 100 \% \tag{3}$$

where C_L and C_o are the concentration of sugar in the concentrate and concentration of the initial solution.

3. Result and Discussions

3.1 Effect of Coolant Temperature

The experiment was conducted to study the effect of coolant temperature on the sugarcane juice concentration efficiency (% Brix) and effective partition constant, K. Since coolant temperature strongly influences the formation of ice crystal, the range of temperature used has been investigated. It is crucial to observe every single change of the colour and concentration of the product. The range of coolant temperature which is the manipulated variable, was varied from -14 °C to -6 °C, while other parameters were kept constant with rotator speed at 205 rpm and rotation time of 180 min.

From Figure 2(a), it can be seen that the value of K increased as the temperature was decreased. Higher K-value represents low efficiency for the system. Coolant temperature of -8 °C is considered as the optimum condition for this separation process due to its low value of effective partition constant, K which is 0.438 portraying higher efficiency of the system with up to 51.15 % Brix increment that is quite satisfactory for this concentration process limit. Lower value of K implies that higher purity of ice has been produced, where more soluble solutes remain in the concentrate.

It can be observed that when the coolant temperature was too low, which is from -8 °C to -14 °C, the value of K started to increase, thereby leading to low concentration efficiency of the system. As stated by Amran and

Jusoh (2013), coolant temperature has a great impact on the ice growth rate, where the ice growth rate increases together with the increase in the difference between the entering solution and the surface temperature. The entering temperature is referring to the temperature of the solution which is sugarcane juice inside the crystalliser, while the surface temperature is referring to the temperature of the coolant. As a result, more solute will be trapped in the ice, which reduces the purity of the ice obtained. Higher growth rate gives rise in the speed of the moving solids in solution, as it becomes too fast and will block the solute outward movement, resulting in the promotion of solute inclusion in the ice crystals (Chen et al*.,* 1998). There is a possibility where the sugarcane juice would also freeze when the coolant temperature is too low caused by the faster freezing rate which can lead to higher impurities to be trapped in the ice crystal, reducing the purity of the ice produced. This phenomenon is undesirable in this system as it will increase the value of K, hence lowering the efficiency of the system.

3.2 Effect of Rotation Speed

Rotation speed represents the flow rate of the solution and the studied range which is 150 to 350 rpm, chosen based on the capacity of the motor that has been used in this setup to rotate the crystalliser at the desired speeds. While the rotator speed was being manipulated, other parameters were kept constant with a coolant temperature of -10 °C and operation time of 180 min.

The purpose of introducing the solution movement is mainly to provide a uniform distribution of flow, therefore the accumulation of solute near the liquid-ice interface can be reduced (Hamid, 2015a). Figure 2(b) illustrates that higher flowrate resulted in a lower K and higher concentration efficiency. As can be seen from the graph, the lowest value of K and higher percentage of concentration efficiency can be observed at rotator speed of 300 rpm. This is also supported by Miyawaki et al. (2005) where they have found that higher flowrate will lower down the advance rate of the ice front, thus high purity of ice will be produced. This finding is also consistent with the study conducted by Okawa et al. (2009), suggesting that higher flow rate promotes slower solidification rate, resulting in less concentration captured in ice. Further analysis of data revealed that the presence of shear force due to high circulation flowrate also will affect the concentration efficiency, where the shear force of the fluid flow is capable of carrying the solute in the solution (Jusoh, 2010). The occurrence of high shear force will bring away the sugar that is entrapped between the dendritic structure of ice layer and will remain in the concentrated liquid, resulting in a high concentration efficiency as achieved when the rotator speed is 300 rpm. However, at the highest rotator speed which is at 350 rpm, high value of K was observed. The possible reason for this finding is that when the rotator speed applied is too high, it might have a potential to erode the ice layer formed on the crystalliser wall, thus reducing the solution concentration in the liquid phase due to the increment of water component in the solution after the erosion (Hamid, 2015b). From the finding, the optimum condition that has been observed in producing low value of K (0.439) was at rotator speed of 300 rpm. At this speed, the system is capable to give high concentration efficiency of up to 59.02 % Brix.

3.3 Heat Transfer Analysis

In order to carry out a heat transfer analysis in this study, the overall heat transfer coefficient, U_o was calculated for both manipulated operating parameters which are coolant temperature and rotator speed. The thickness of ice generated at each operating condition was collected. The overall heat transfer coefficient, U_o can be measured by thermal resistance using Eq(4) and logarithmic mean area, A_m using Eq(5).

$$R = \frac{1}{U_0 A_m} = \frac{1}{A_i h_g} + \frac{x}{K_i A_m} + \frac{1}{A_o h_o} \tag{4}$$

where, A_o is the outside surface areas, m^2; Ai is the inside surface areas of tube, m^2; h_g is the heat transfer coefficient for glucose solution, $W/m^2.°C$; h_o is the heat transfer coefficients for ethylene glycol 50 %, $W/m^2.°C$; k_i is the thermal conductivity of ice, W/m.°C; *x* is the thickness of ice layer, m.

$$A_m = 2\pi L \frac{x}{\ln(\frac{r}{r-x})} \tag{5}$$

where, r is the radius of crystalliser, m; L = the total length of the crystalliser, m; x is the thickness of medium wall, m. Generally, the trends for rotator speed and coolant temperature against the overall heat transfer coefficient can be described by referring to the ice thickness, in which the ice thickness will reflect the resistance for the heat transfer, thereby will affect the efficiency of the overall heat transfer coefficient in the system. Figure 3 shows that U_o gradually increased, resulting from the increase in the coolant temperature. The possible reason that can explain this is the small temperature difference between the solution and coolant, promoting a small thickness of ice formed on the inner wall of the crystalliser that will lead to low resistance for the heat transfer, hence increasing the overall heat transfer coefficient respectively. The result from the analysis shows that the highest U_o (0.39074 $W/m^2.K$) can be observed at coolant temperature of -6 °C therefore it can be assumed that the heat from the solution to the coolant through the wall of the crystalliser on the acquired surface can be transferred effectively in this condition. The temperature difference between the solution and coolant will increase as the coolant temperature is further decreased, thus will promote faster freezing rate, causing all the solution to become a solid, leaving no passage for the solution to be circulated (Hamid, 2015a). Figure 3 also displays that as the rotator speed was increased, the value of the overall heat transfer coefficient also increased. The highest value of U_o (0.3989 W/m^2.K) can be observed at the highest circulation flow rate which is at 350 rpm. This finding is also in agreement with Wakisaka et al. (2001), who suggested that increasing the circulation flow rate of solutions promotes heat transfer with ice crystals from its tips, hence improving the planar ice growth from the wall by keeping soluble solids away from the ice-liquid interface. In addition, the presence of shear force when higher circulation flowrate is applied will enhance the fluid flow to remove the solute in the solution, which will be brought away from the surface of the stagnant solid ice layer, resulting in heat transfer to occur more effectively. The residence time of the solution to be in contact with the cooling surface also will be reduced, therefore heat can be transferred from the solution to the ice layer in a shorter time, resulting in a better heat transfer efficiency with a high value of the overall heat transfer coefficient, U_o.

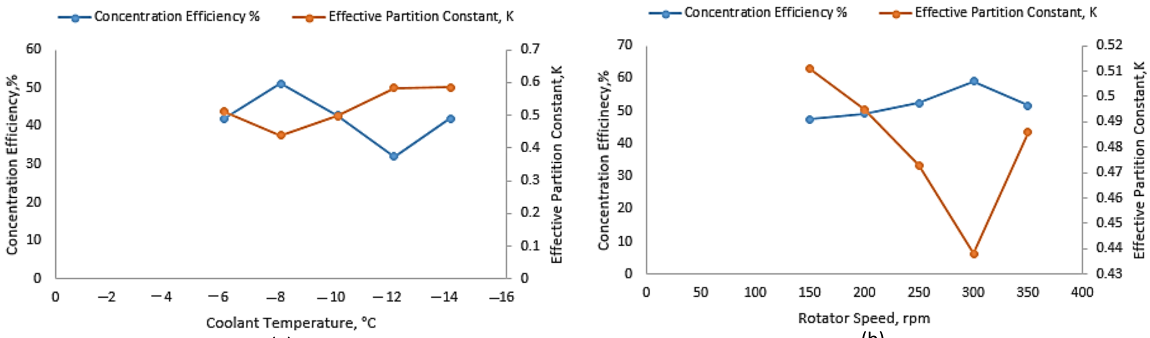

Figure 2: (*a) Effect coolant temperature on K-Value and concentration efficiency (b) Effect of rotator speed on K-value and concentration efficiency*

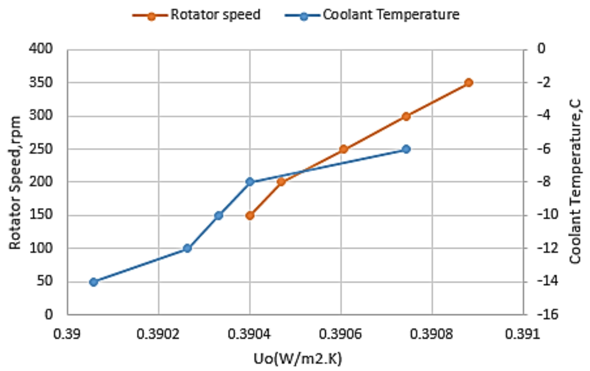

Figure 3: Effect of operating parameters on the overall heat transfer coefficient Uo

4. Conclusion

From this study, it has been proven that progressive freeze concentration is relevant to be applied in the food processing industry to concentrate the sugarcane juice as it can produce a concentrate with higher sugar content. In general, lower coolant temperature and higher rotator speed resulted in a lower K value and higher efficiency in term of sugar increment, thus promoting a better efficiency for the system. In this study, coolant temperature of -8 °C and rotator speed at 300 rpm have been chosen as the optimum condition for the process based on the lowest K value and highest concentration efficiency. The analysis on the heat transfer activity in progressive freeze concentration by the determination of overall heat transfer coefficient, U_o by using thermal resistance equation has been successfully achieved in this study. From the analysis, it has been proven that the thickness of ice is a chief factor that affects heat performance in the process. The process will encounter a high resistance as the thickness of ice increase, therefore will reduce the efficiency of heat transfer between coolant and solution and lowering the rate of ice formation, resulting in lower value of overall heat transfer coefficient, U_o. The smallest ice thickness was observed at coolant temperature of -6 °C and rotator speed of 350 rpm proving that heat transfer had occurred at its maximum in this condition.

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