Jurnal Teknologi

Full paper

Enhancement of Sugar Content through Progressive Freeze Concentration: Effect of Initial Concentration

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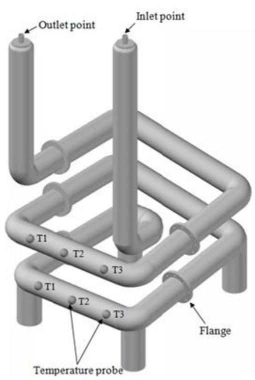
Article history

Received :4 October 2013
Received in revised form :
3 April 2014
Accepted :8 May 2014

Graphical abstract



Stainless steel crystallizer

Abstract

A new concentration technique is required to eliminate portion of water from coconut water (CW) and reduce the cost of storage, handling and shipping. As Progressive Freeze Concentration (PFC) could retain the nutritional compounds, it was applied to concentrate CW and enhance its sugar content. In PFC system, only a single block of ice is formed as a layer on the cooled surface. A coil stainless steel crystallizer was used as FC unit to investigate the enhancement of sugar content in CW. The effect of initial concentration of CW was then investigated on the performance of the PFC system through the Effective Partition Constant (K) value and increment of sugar content. It was found that lower initial concentration yielded lower K, which is favourable and high sugar content. The best K achieved was of 0.3101 and the highest sugar content increment acquired was 53% with the range of initial concentration investigated (3 to 5%Brix).

Keywords: Freeze concentration; coconut water; ice crystal; effective partition constant; progressive freeze concentration

1.0 INTRODUCTION

One of the important issues in health care comes in the form of a new beverage that can sustain healthy nutrition and energy booster. Due to this issue, the natural water found within the shell of a green coconut has recently gained attention as the new isotonic drink. From previous study, coconut water stores high nutritional content which includes potassium, antioxidants and mineral[1]. These nutritious components help to replenish the body electrolytes lost through sweat[2]. Traditionally, most coconut water (CW) is consumed directly from the fruit because the cavity of the coconut can sterilize the CW to ensure its freshness. However, as consumers come from many countries around the world, the coconut fruits have to be transported to thousands miles away with only twenty percent of the transported fruits is the wanted water and eighty percent is the coconut fibers. This factor contributes to the high sale price of CW due to the high costs of storage, handling and shipping. This problem leads to the search for new or improved concentration techniques which can retain the freshness, nutrition and unique aroma of the CW as well as to eliminate portion of the water from the aqueous solution and henceforth ease the handling of CW by reducing its volume and weight.

At present, there are three methods available for concentration of fruit juice which gained attention for commercial application. These three methods are evaporation, reverse osmosis (RO) and freeze concentration (FC)[3]. Every process has its specific limitation of attainable concentration. Among those three, the highest juice concentration up to 65 ºBrix can be achieved through evaporation concentration technique. The intermediate concentration is achieved by using FC which is limited at 50 ºBrix and the lowest is via RO which limited at 22 to 30 ºBrix due to fouling phenomena. Evaporation is considered to be the simplest and widely used method, but it is not suitable to be engaged when the fruit juice to be concentrated is heat sensitive due to loss of their volatiles and aromas compound when heated[4]. Besides, it does consume high energy which about 4 $kWh.m^{-3}$ for evaporators[5]. RO is also not a favorable method for juice concentration although the energy consumed for this method is the lowest. This is because in most cases, clogging of the membrane can easily occur which will reduce the yield[6] and it involves high cost to attain the osmotic pressure required. The latest method introduced for fruit juice concentration is FC. It is a process where water molecules is frozen out and leave behind the most concentrated solution. As this method does not involve any heating, most volatiles and aromas compound will stay in the concentrate produced[7]. This feature makes FC a better option for fruit juice concentration because the aroma is one of the most important factors to make it marketable. Moreover, the ice lattice produced is in small dimensions, thus the inclusion of solutes like sugar is almost impossible; once again making it a highly effective concentration process for juice. Basically, there are two types of FC methods available, which are conventional Suspension Freeze Concentration (SFC) and the newer Progressive Freeze Concentration (PFC)[8]. SFC is a process where the small sizes of ice crystals are formed in a suspension of the mother liquor. The limited size of the ice formed in this process makes its separation from the mother solution is difficult to be handled and need a very complicated system to enlarge and to obtain uniform size of ice crystals formed.

As an alternative for SFC, a different concept of FC has been introduced, called PFC. PFC involves formation of a large single ice crystal that grows layer by layer from the bulk solution on the cooled surface. The separation of the ice from the bulk solution is much easier in this system as only a single ice crystal is formed[9]. PFC has been applied to concentrate fruit juices such as tomato juice[10], raspberry[11], orange juice[12], apple and pear juices[13-14]. In this present paper, the principal aim is to study the process of PFC of CW to enhance its sugar content. For this purpose, the effect of initial concentration on the variables that can define the efficiency of this type of FC was examined. Effective Partition Constant (K) value and percent increment of sugar concentration were analyzed.

2.0 EXPERIMENTAL

2.1 Materials

CW at concentrations ranging from 3 to 5% Brix was used as raw material. Fresh green coconut was obtained from a local plantation in Johor, Malaysia. It was then perforated to obtain its water and then filtered before being used as raw material. Distilled water was used in making seed ice crystals in the crystallizer, whereas a 50% (v/v) Ethylene Glycol- distilled water mixture was used as coolant.

2.2 Equipment

Figure 1 shows the stainless steel crystallizer fabricated as the equipment used. Practically, crystallizer is an important part in PFC system, where it provides a surface area where ice crystal for ice crystal formation and attachment. In this study, the crystallizer was designed with thickness of 0.8 mm and internal diameter of 2.54 cm. It has three stages and also equipped with six flanges to enable the crystallizer to be easily opened. Hence, the ice layer produced in each experiment can be visualized. For each stage, nine temperature probes (thermocouples type K) was engaged.

2.3 Experimental Procedure

The experimental set-up for this system is as shown in Figure 2. First, distilled water was fed into the system by a peristaltic pump for the formation of uniform 0.1 mm seed ice lining on the surface of the crystallizer. This step is necessary to avoid supercooling which can promote contamination of the first ice formed. The peristaltic pump was used because of its capability to fluidize the solution with minimal heat generation, which can avoid the reduction of cooling effects during the freezing process. It is also used to control the circulation flow rate of the solution. It was circulated using a pair of silicon tube where each end can be connected to each other. Then, the full crystallizer was immersed in a water bath at -12ºC. After 5 minutes of circulation, the distilled water was flushed out and the crystallizer was filled again with CW solution to start the PFC process. Before being introduced into the crystallizer, the CW was precooled close at 2ºC to avoid the seed ice crystal from melting.

The filled crystallizer was once again immersed in water bath, and the solution was started to circulate at the desired circulation flow rate of 2800 ml/min and circulation time of 14 minutes. Then, at the end of the circulation time, the process was stopped and the crystallizer was taken out from the water bath for purpose of solution flushing and ice thawing. The concentrate in the silicone tube was flushed using the pump to be collected. The flanges were unassembled and the whole volume of concentrate was collected in order to determine the sugar concentration using Brix refractometer. Then, the thickness of the ice layer formed was measured at each flange point and a sample of ice produced was taken for sugar concentration measurement. The experiment was repeated three times for each value of initial concentration.

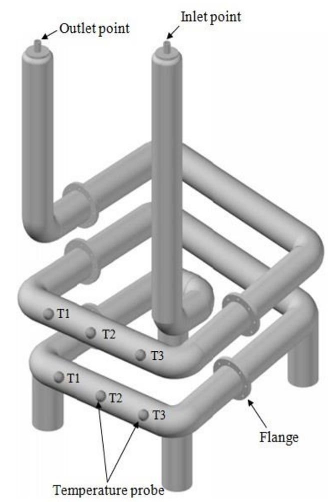


Figure 1 Stainless steel crystallizer

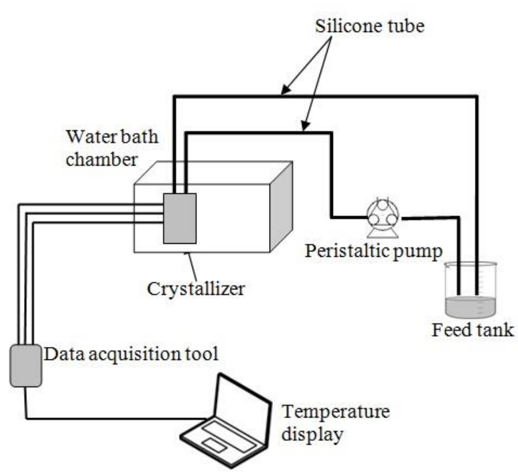


Figure 2 Schematic drawing for experimental set-up

3.0 RESULTS AND DISCUSSION

In PFC process, the ice crystals are formed as a layer on the inner wall of the stainless steel crystallizer. Figure 3 shows the ice layer formed when the flange is opened at the end of the experiments. The quality of the ice produced is associated with the K value which was determined by using equation (1). The smaller the value of K the higher the purity of ice obtained and vice versa. In this equation, the initial volume and initial solute concentration of solution are represented by V_0 and C_0 respectively, whereas, the final volume and final solute concentration of the solution are represented by V_L and C_L respectively.

$$(1\text{-}K)\log(V_L/V_0) = \log(C_0/C_L) \quad (1)$$

Other than the quality of the ice produced, sugar increment is also one of the important determinant parameters to be determined in this study in order to know the system efficiency. Thus, the increment of sugar concentration was determined in percentage and calculated using equation (2).

$$\text{Sugar increment} = (C_f - C_i)/C_i \times 100\% \quad (2)$$

where, C_f and C_i respectively are the concentration of sugar in the concentrate and concentration of the initial solution.

3.1 Effect of Initial Concentration

The experiment was started with varying the initial concentration from 3.0 to 5.0% Brix, while the other parameters were kept constant at flowrate of 2800 ml/min, operation time of 14 minutes and coolant temperature of -12ºC. Refractive index (RI) analysis was performed at the end of each experiment to indicate percent sugar (% Brix) in the collected concentrated solution.

The lowest K value was obtained at the lowest value of initial concentration and it continued to increase in parallel with the increasing concentration of the initial solution. This can be observed from Figure 4 which shows that the small amount of solute present in the solution occur at low initial concentration. Hence, the probability of solute to be trapped by the ice growth is low, which is opposite to high initial concentration.

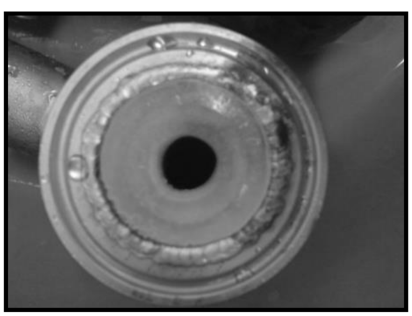


Figure 3 A close-up of the ice layer formed

This finding can be best described by the concentration polarization model suggested by Miyawakiet *et al.*[15], which reveals that the highest concentration of solute is at the ice-liquid interface. This phenomenon can be best described by the tendency of the solute to accumulate at the interface after being rejected from the ice crystal lattice which is very small in dimension. Thus, the inclusion by the solute is impossible during its growth process. As a result, the solute concentration in the liquid surrounding the ice front will be low. This situation leads to the formation of solute concentration gradient and could rise to a modification in the solid-liquid equilibrium temperatures as shown in Figure 5. Low initial concentration produces low solute concentration at the interface. Therefore, it will reduce the concentration gradient. The equilibrium temperature increase as the concentration gradient decreased. Therefore, a region of increasing supercooling which is called constitutional supercooling, would not be generated during the occurrence of solid-liquid phase and thus avoid the occurrence of the constitutional supercooling. As the supercooling does not occur, there would be no formation of dendritic ice crystals which normally trap the solutes between its structures[16]. Hence, the amount of solute in the ice layer formed becomes lower, resulting in lower K value. Ultimately, it can be concluded that the efficiency of this system is affected by the initial amount of sugar in the solution to be concentrated through constitutional supercooling.

Meanwhile, Figure 6 shows the result obtained for the effect of initial concentration towards percentage of sugar increment. From the graph, it is shown that the lowest initial solution concentration of 3.0% Brix is increased to 4.7% Brix at the end of the process, gives 53 percent increment of sugar concentration. On the other hand, for the highest concentration of 5.0% Brix, there is only a 22 percent increment of sugar concentration. This indicates that in the concentration of fruit juice, PFC could benefit juices with low solute content to yield juices with higher concentration for further commercial use.

This observation could be explained by the occurrence of mass transfer diffusion in the boundary layer next to the ice-liquid interface during phase change in FC system. Basically in this phenomenon, the solute molecule will move from high concentration region to regions having low concentration. At low initial concentration, the sugar at the interface would tend to move into the liquid phase which is less saturated than the ice phase throughout the process, thus yielding high concentration of sugar in the concentrate produced. However, for high initial concentration, the liquid phase itself is saturated compared to the ice phase. This phenomenon leads to the tendency of sugar molecules to move into the solid phase from the beginning of the process up to the end, resulting in low sugar content in the concentrate but higher in the ice formed. Hence, it is proved that the purity of ice will decrease at high initial concentration. High circulation flow rate and rotation would be needed for this condition.

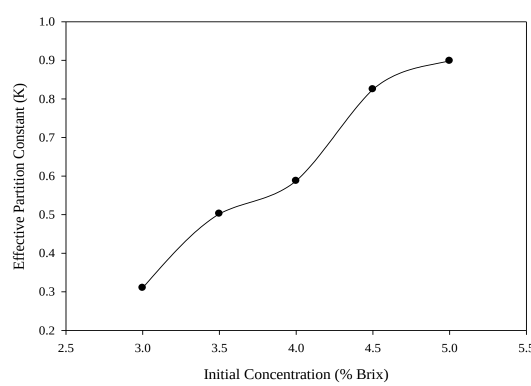


Figure 4 Effect of circulation flowrate on percent of sugar increment and K

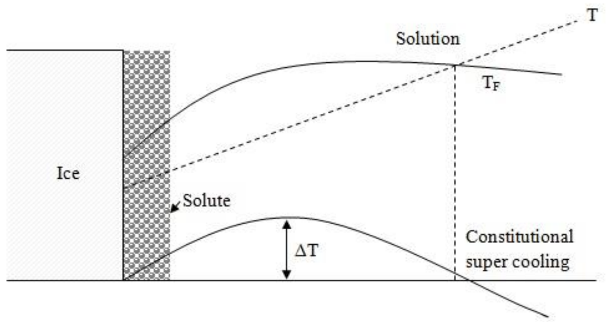


Figure 5 Constitutional supercooling during freezing process of solutions[17]

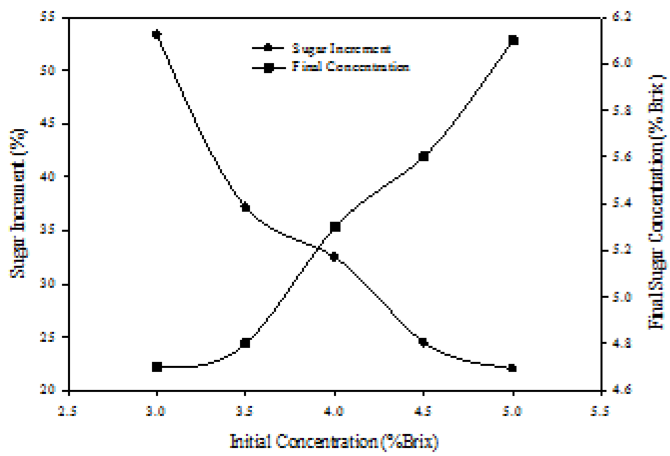


Figure 6 Effect of initial concentration on increment of sugar concentration

■4.0 CONCLUSION

This study has proven that the progressive freeze concentration system is suitable to be used for juice concentration at a very dilute solution, which is commonly faced in food processing. This is based on the capability of PFC to produce concentrates with high sugar content. However, this study should be further pursued by using coconut water from different stage of maturation. This is because sugar content in coconut water differs for each maturation stage. High flow rate of 4000 ml/min, 17 minutes of operation at -15°C are suggested to achieve low K and high sugar increment for high initial concentration of sugar.

Acknowledgement

The support by the Universiti Teknologi Malaysia for the financial support GUP grant (04H46) is gratefully acknowledged.

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