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Progressive Freeze Concentration of Coconut Water

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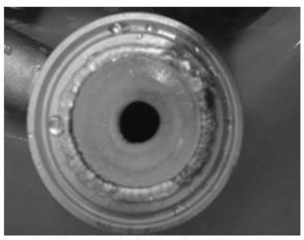
Graphical abstract



A close-up of the ice layer formed

Abstract

In order to provide convenience for consumers around the world, it is highly beneficial if the nutritious coconut water (CW) could be concentrated and just easily be added with water for later consumption. A new concentration technique is required to eliminate some parts of the water from the CW while retaining its nutritious and unique aroma. As Progressive Freeze Concentration (PFC) could retain the nutritional compounds, it was applied to concentrate CW and reduce its volume. 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 low initial concentration and intermediate coolant temperature yielded lower K value and high increment of sugar content. The best K achieved was 0.3101 and the highest increment of sugar content was 53% at initial concentration of 3 %Brix and coolant temperature of 12°C.

Keywords: Freeze concentration; progressive freeze concentration; initial concentration; coolant temperature; coconut water

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. This is due to its high nutrition content, as studies found high content of potassium, antioxidants and mineral [1]. These nutritious components help to replenish the body electrolytes lost through sweat [2]. Traditionally, most 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. As CW consumers come from many parts of the world that are not suitable for coconut tree to grow, it is highly beneficial if the CW could be concentrated, packaged and only be added with water for later consumption. This 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 reduce 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 their specific limitation of concentration attainable. 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 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 and it involves high cost to attain the osmotic pressure required [6].

The latest method introduced for fruit juice concentration is FC. It is a process where water molecules are 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], which makes it 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 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 system 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 principal aim of this research is to study the effect of initial concentration on the variables that can define the efficiency of this type of FC. 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 take its water and then filtered before being used as raw material. Distilled water was used in making ice seed crystals in the crystallizer whereas a 50% (v/v) Ethylene Glycol solution mixed with 50% of distilled water was used as coolant.

2.2 Equipment

Figure 1 shows the stainless steel crystallizer fabricated as the equipment used. The purpose of this crystallizer is to provide a surface area where ice crystal will form and attach to. It was designed with thickness of 0.8 mm and 1 inch of internal diameter. The crystallizer has three stages and also equipped with six flanges to enable the crystallizer to be split into two. Hence, the ice layer produced in each experiment can be visualized. Nine temperature probes (thermocouples type K) was engaged in each stage to determine the temperature profiling of the solution, crystallizer wall and coolant which was displayed by Picolog recorder software through a connected computer.

2.3 Experimental Setup

The experimental set-up for this system is as shown in Figure 2. The crystallizer was immersed in a water bath at the desired cooling temperature. The crystallizer was connected to a peristaltic pump using a pair of silicon tube. The purpose of the silicon tube is to circulate solution inside the crystallizer and the solution from feed solution tank for a designated period of time. The peristaltic pump is 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 flowrate of the solution.

2.4 Experimental Procedure

Distilled water was fed into the system by a peristaltic pump. It was circulated using a pair of silicon tube where each end can be connected to each other. This is the step for the formation of 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. Then, the full crystallizer was immersed in a water bath at -12 ºC. After 5 minutes of circulation, the distilled water was flushed out and the crystallizer was filled again with CW solution to start PFC. Before being introduced into the crystallizer, the CW was diluted with distilled water until its concentration achieved 3.0 %Brix. Then it was precooled close to the water freezing temperature by immersing some of the ice cubes into the feed solution tank to avoid the seed ice crystal from melting. The ice cubes also was makes from distilled water and the solution temperature was kept at 2 ºC.

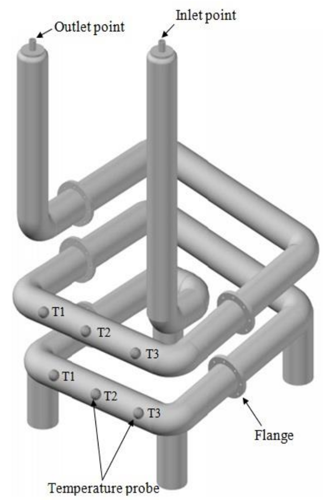


Figure 1 Stainless steel crystallizer

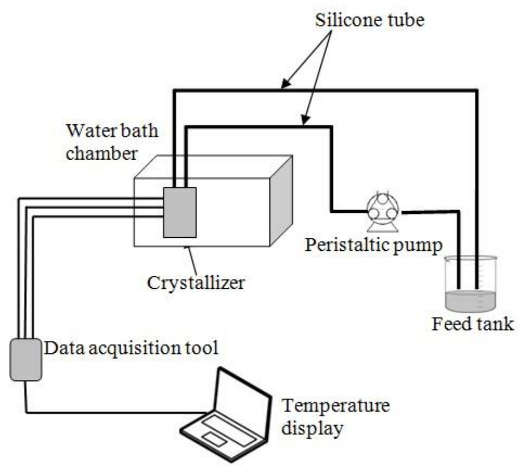


Figure 2 Schematic drawing for experimental set-up

The filled crystallizer was once again immersed in a precooled water bath at -12 ºC, and the solution was started to circulate at the desired circulation flowrate of 2800 ml/min and the circulation time of 14 minutes. Then, after 14 minutes, the circulation was stopped and the crystallizer was taken out from the water bath to be thawed and flushed. The pump was flushed to collect the concentrate in the silicone tube. The flanges were unassembled and the whole volume of the concentrate was collected. Then, the thickness of the ice layer formed was measured at each flange point and a sample of ice produced was taken. Lastly, in order to determine the concentration of sugar in the concentrate and also in the ice, Brix refractometer was used. The experiment was repeated for each value of circulation flowrate and circulation time as shown in Table 1.

Table 1 Value of varied and constant variables

Variables	Range	Constant			
		Flowrate (ml/min)	**Time (min)**	**Initial Conc. (%Brix)**	**Coolant Temp. (°C)**
Initial Conc. (%Brix)	3.0 3.5 4.0 4.5 5.0	2800	14	-	-12
Coolant Temp. (°C)	-10 -12 -14 -16 -18	2800	14	3.0	-

3.0 RESULTS AND DISCUSSION

In this process, the ice crystals are formed as a layer on the inner wall of the stainless steel crystallizer. Figure 3 show the ice layer formed when the flange is opened at the end of the experiments. Throughout the experiment, the ice thickness was varied with the different operating conditions used.

The K value which was related to the quality of the ice produced was determined by using equation 1. 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.

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

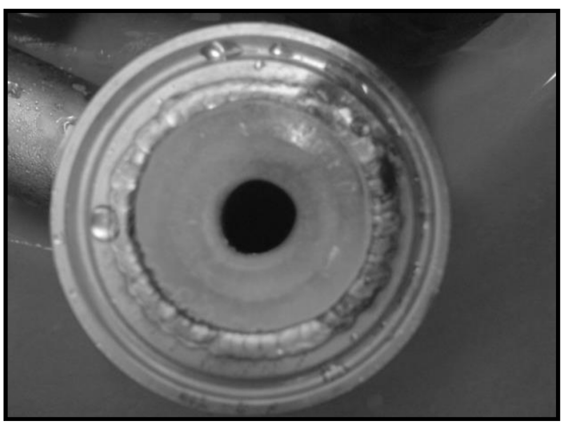


Figure 3 A close-up of the ice layer formed

Instead of the quality of ice, sugar increment is also one of the important determinant parameters in this study to determine the system efficiency. The increment of sugar concentration in this study was determined in percentage and calculated using equation (2).

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

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 investigation of the effect of initial concentration on the efficiency of this system was done by ranging the concentration from 3.0 to 5.0 %Brix while the other parameters was kept constant at 2800 ml/min, 14 minutes and -12 ºC respectively for circulation flowrate, circulation time and coolant temperature.

From Figure 4, it can be observed that the lowest K value is obtained at the lowest value of initial solution concentration and it has continued to increase in parallel with the increasing concentration of the initial solution. Low ice contamination can occur at low initial concentration due to the small amount of solute present in the solution as compared to solvent or water content. Hence, the probability of being trapped by the ice growth is low. It is on the contrary for high initial concentration.

It can be best described by using the concentration polarization model suggested by Miyawaki *et al.* [15]. This model reveals that the highest concentration of solute is at the ice-liquid interface as they accumulate at the interface after being rejected from the ice crystal lattice which is very small in dimension, making 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 solute concentration gradient and could rise to a modification in the solid-liquid equilibrium temperatures as shown in Figure 5. This equilibrium temperature decreases as the concentration gradient increase. Therefore, a region of increasing supercooling can be generated during the occurrence of solid-liquid phase and is called as constitutional supercooling.

Low initial concentration produces low solute concentration at the interface. Therefore, it will reduce the concentration gradient 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], causing the amount of solute in the ice layer formed lower resulting in lower K value. Ultimately, it can be concluded that efficiency of this system is affected by the initial amount of sugar in the solution to be concentrated through constitutional supercooling.

System efficiencies in terms of percentage sugar increment was also analyzed for each concentration studied to investigate any effects towards it and the result is plotted in Figure 6. 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, which 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, the liquid phase itself is saturated compared to the ice phase as the concentration of sugar in the initial solution is increased. This 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.

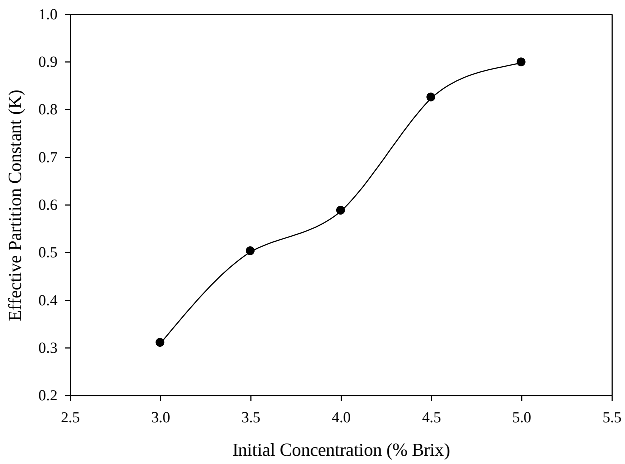


Figure 4 Effect of circulation flowrate on percent of sugar increment and K

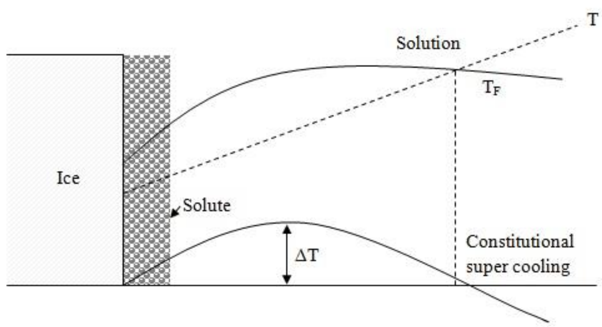


Figure 5 Constitutional supercooling during freezing process of solutions [17]

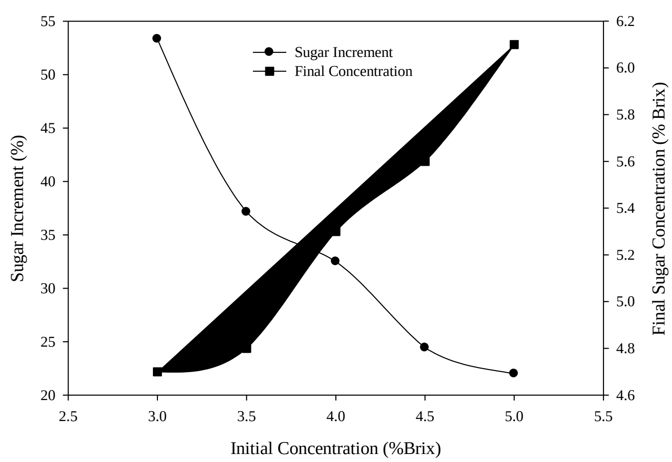


Figure 6 Effect of initial concentration on increment of sugar concentration

3.2 Effect of Coolant Temperature

Figure 7 shows a plotted graph according to equation 1 to obtain K values at various coolant temperatures in the range of -8 ºC to -16 ºC and at constant circulation flowrate, circulation time and initial solution concentration as detailed in Table 1.

It can be seen from the graph that the value of K increases with the reduction of coolant temperature. However, there is a decrease in K value from -8 °C to -10 °C. This is because at -8 ºC, the ice layer produced is fragile and dendritic in structure where the sugar would easily be trapped, thus resulting higher value of K. It is seen that the K value is satisfactorily low starting at temperature of -10 ºC until -12 ºC. Further reduction of the temperature will rapidly increase the K value as shown at coolant temperature of -14 °C until -16 ºC. This means that the efficiency of the system would decrease when the coolant temperature is too low. Therefore, it can be deduced that the suitable coolant temperature to be applied for high efficiency PFC system with this coil crystallizer is between -10 ºC and -12 ºC.

The effect of coolant temperature in terms of enhancement of sugar content is shown in Figure 8. From the plotted graph, it is evident that there are increments in sugar concentration with decreasing coolant temperature until -12 ºC. However, when the temperature reduction was continued up to -16 ºC, the sugar concentration has reduced. Thus, again this shows that the efficiency of the system is unsatisfactory if the coolant temperature used is too low. Based on high increment of sugar concentration and satisfactory low K value that was attained, -12 ºC is considered as the most suitable coolant temperature in producing concentrated solution with high concentration of sugar. These two findings could be explained based on the phenomenon of ice crystal growth on the cooled surface. As described previously, throughout this PFC process, ice layer is formed on the inner surface of the crystallizer wall. According to Miyawaki *et al*., the rate of the ice formation is theoretically controlled by the coolant temperature [18].

Coolant temperature that is too low will lead to the high rate of ice growth per unit time. Besides that, the ice growth rate increases as the temperature difference between the entering solutions and the cold wall is high. The low coolant temperature will cause the temperature of the wall to be very low. This will increase the temperature difference and thus increase the ice growth rate. As a result of this high growth rate, the tendency of the solute being trapped in the outward movement of the ice front is higher and thus giving high K value. When the sugar concentration in ice phase is high, it would surely result in lower sugar content in the concentrate produced. This can be evident by the two graphs plotted in Figure 7 which shows that the lower the coolant temperature the higher the value of K and at once resulted in reduction in sugar concentration in the concentrate.

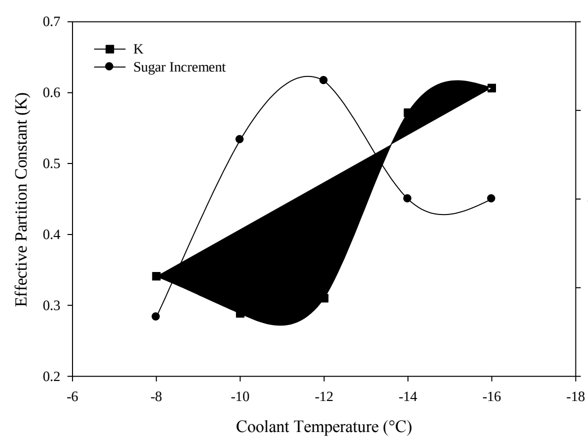


Figure 7 Effect of coolant temperature on K

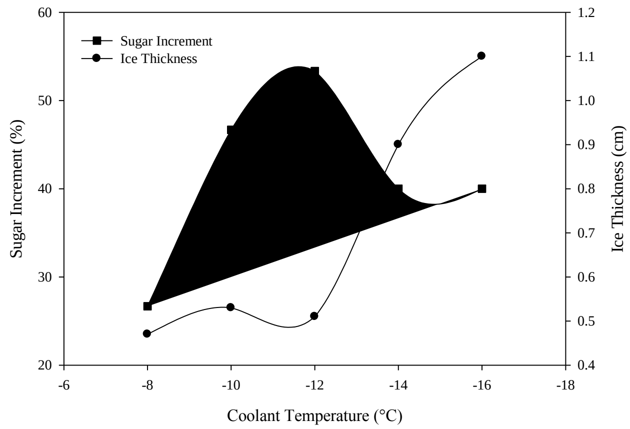


Figure 8 Effect of coolant temperature on sugar concentration

Throughout this process, heat is transferred from the solution to the coolant through the crystallizer wall. However, the accumulated ice at the inner wall of the crystallizer could act as an additional resistance to the heat transfer, giving reduction of thermal conductance. For this reason, it took longer for water to cool down and separates from the liquid phase, thus the concentrated solution produced will be not high enough. This is supported by the findings from Habib and Farid[19] who have stated that any increment in ice thickness was associated with a decreasing rate of freeze concentration. Therefore, the presence of thick ice also could be the reason why at low coolant temperature, the percentage of sugar increment is not high. This can be proved by the plotted graph shown in Figure 8.

■4.0 CONCLUSION

This work has proven that the CW can be concentrating efficiently by using PFC system and also proves that low initial concentration and intermediate value of coolant temperature resulted in better efficiency based on lower K value and high increments of sugar concentration. Nevertheless, the effect of other operating parameters like temperature of initial solution and size of solute on the system efficiency should also be investigated in order to know its best performance.

Acknowledgement

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