

Progressive Freeze Concentration of Coconut Water: Effect of Circulation Flowrate and Circulation Time

Mazura Jusoh*, Noor Naimah Mohamed Nor

Faculty of Chemical Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia

*Corresponding author: mazura@cheme.utm.my

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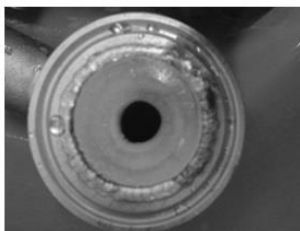
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Graphical abstract



A close-up of the ice layer formed

Abstract

Progressive freeze concentration integrated with a coil crystallizer was applied to concentrate coconut water in order to increase its sugar content. In progressive freeze concentration system, only a single ice is formed as a layer on cooled surface. It is introduced as an alternative in compensating the disadvantages of conventional method of freeze concentration, called suspension freeze concentration. In this research, the efficiency of the progressive freeze concentration system was determined through effective partition constant (K) value and percent increment of sugar content. Hence, the effect of circulation flowrate ranging from 2000 to 2800 ml/min and circulation time from 10 to 18 minutes on those two variables was then investigated through experiments designated using one factor at a time. From the experimental work, it was found that higher efficiency results at higher circulation flowrate of 2800 ml/min based on lower K and high percentage of sugar increment achieved. It was also found that the percentage of the sugar increment is high when the period given for circulating the solution is increased and the most suitable circulation time is 14 minutes.

Keywords: Freeze concentration; progressive freeze concentration; circulation flowrate; circulation time; coconut water

Abstrak

Pemekatan pembekuan progresif telah digunakan untuk pemekatan air kelapa bagi meningkatkan kandungan gulanya. Dalam sistem pemekatan pembekuan progresif, hanya ais tunggal terbentuk sebagai lapisan di permukaan sejuk. Ia diperkenalkan sebagai satu alternatif untuk mengatasi kelemahan kaedah konvensional pemekatan pembekuan, yang dipanggil pemekatan pembekuan penggantungan. Dalam kajian ini, kecekapan sistem pemekatan pembekuan progresif telah ditentukan melalui nilai pemalar pemisahan berkesan (K) dan peratus kenaikan kandungan gula. Oleh itu, kesan kadar alir kitaran antara 2000 hingga 2800 ml/min dan masa kitaran dari 10 hingga 18 minit terhadap kedua-dua pembolehubah disiasat melalui penyisatan yang dirangka menggunakan satu faktor pada satu masa. Daripada eksperimen yang dibuat, didapati bahawa kecekapan yang lebih tinggi terhasil pada kadar alir kitaran yang tinggi iaitu 2800 ml/min berdasarkan K yang rendah dan peratusan tinggi kenaikan gula yang telah dicapai. Ia juga mendapati bahawa peratusan kenaikan gula adalah tinggi apabila tempoh yang diberikan untuk kitaran larutan bertambah dan masa kitaran yang paling sesuai ialah 14 minit.

Kata kunci: Pemekatan pembekuan; pemekatan pembekuan progresif; kadar alir kitaran; masa kitaran; air kelapa

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1.0 INTRODUCTION

The change of consumer behaviors shifting from sweet artificial to more natural isotonic drinks has led to the growing demand for coconut water (CW) as energy drinks and sports drinks. This is due to the contribution of inorganic ions like sodium, potassium, magnesium and calcium contained in CW. These nutritious components help to replenish the body electrolytes lost through sweat.¹ It is preferable to consume CW directly from its fruit because the cavity of the coconut can sterilize the CW to ensure

its freshness. However, consumers come from many countries around the world, thus the fruits have to be transported to thousand miles away. This factor contributes to the high sale price of CW due to the high costs of storage, handling and shipping. Moreover, only twenty percent of the fruit is the wanted water, the rest is its fiber. 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 provide large quantity of CW to be further processed into ready-to-drink package with lower prices.

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).² Every process has their specific limitation of concentration attainable. Among those three, the highest juice concentration up to 65 °Brix can be achieved through evaporation concentration technique. The intermediate concentration is achieved by using FC which is limited at 50 °Brix and the lowest is via RO which limited at 22 to 30 °Brix 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.³ Besides, it does consume high energy for evaporators.⁴ 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 yield⁵ 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,⁶ 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).⁷ 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.⁸ PFC has been applied to concentrate fruit juices such as tomato juice,⁹ raspberry,¹⁰ orange juice,¹¹ apple and pear juices.¹²⁻¹³ 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 circulation flowrate and circulation time which are known to bring significant effects to 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 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 coil 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 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°C. 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°C.

The filled crystallizer was once again immersed in a precooled water bath at -12°C, and the solution was started to circulate at the desired circulation flowrate of 2000 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.

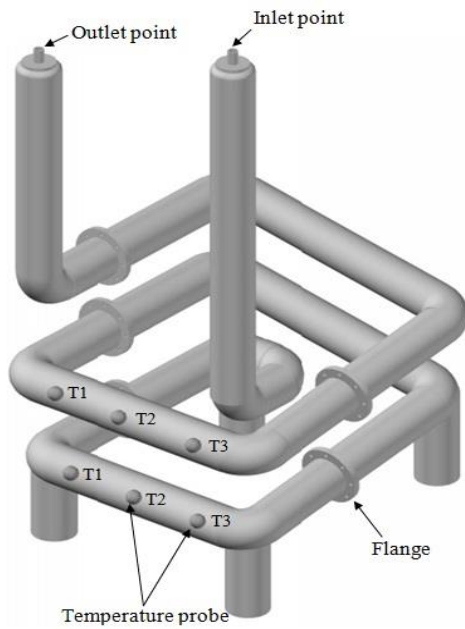


Figure 1 Stainless steel coil crystallizer

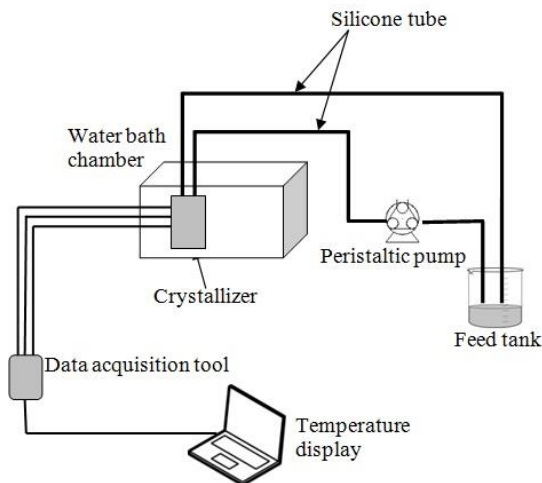


Figure 2 Schematic drawing for experimental set-up

Table 1 Value of varied and constant variables

Variables	Constant				
	Range	Flowrate (ml/min)	Time (min)	Initial Conc. (%Brix)	Coolant Temp. (°C)
Flowrate (ml/min)	2000	-	14	3.0	-12
	2200				
	2400				
	2600				
	2800				
Time (min)	10	2800	-	3.0	-12
	12				
	14				
	16				
	18				

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 and Figure 4 shows 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.

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

The quality of ice produced is one of the important determinant parameters in this study to determine the system efficiency other than sugar increment. Meanwhile, the increment of sugar concentration in this study was determined in percentage and calculated using Equation (2).

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

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

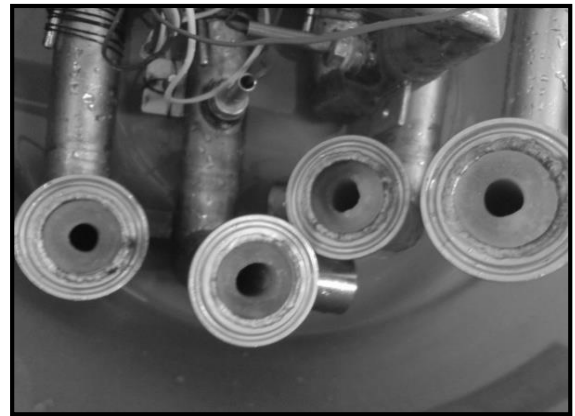


Figure 3 Ice layer formed

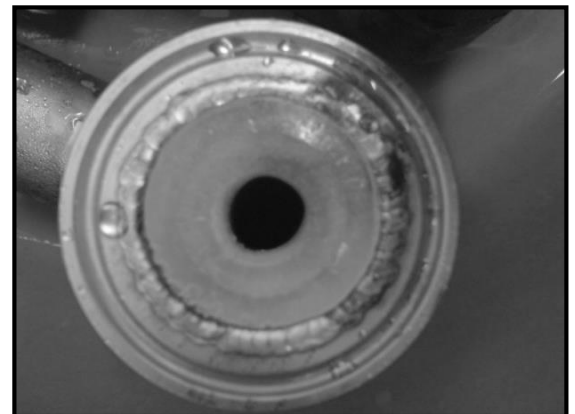


Figure 4 A close-up of the ice layer formed

3.1 Effect of Circulation Flowrate

The studied range of circulation flowrate in this study is 2000 to 2800 ml/min. This range was chosen based on the capacity of the available pump. For each flowrate level in this range, an

experiment was carried out. The other operating condition was kept constant as listed in Table 1. Graph is plotted against the determinant parameters, K value and the percentage increment of sugar content, for the purpose of indicating and explaining the effect of this parameter on the concentration process of CW through PFC system.

The effect of circulation flowrate on the purity of the thawed ice and sugar increment are shown in Figure 5. As can be seen from the graph, the increase in the circulation flowrate causes decreasing K. This shows that the higher the circulation flowrate used, the higher the resulting ice purity. This finding is acceptable based on what has been discussed by Miyawaki *et al.*¹⁴ and Ramos *et al.*¹⁰ who said that high circulation flowrate can produce high purity of ice and it is evidenced by the lower K value, which means better efficiency.

This finding can be explained as follows: increasing circulation flowrate will reduce the residence time of the solution to be in contact with the cooling surface, thus allowing less time for heat to be transferred from the solution to the coolant. Therefore, the speed of the ice front would be reduced and provide less time available for the ice growth to trap solute at the ice-liquid interface although the temperature of coolant is low. Hence, this is the reason why high circulation flowrate is capable of producing a lower K value and increase the ice purity.

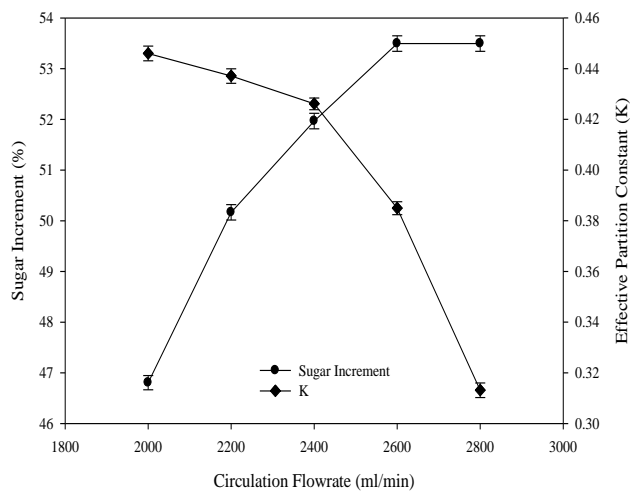


Figure 5 Effect of circulation flowrate on percent of sugar increment and K

On the other hand, for effect on sugar increment, the graph shows that the higher circulation flowrate can raise the sugar concentration more than 50 percent. This is proven when the best increment of 53 percent was achieved at the highest circulation flowrate applied in this study, which is 2800 ml/min. This circulation flowrate is not adequately high but is still capable to increase the sugar content up to 50 percent. Therefore, it can be concluded that the percentage of increment is high if the circulation flowrate is further increased.

This could be explained by occurrence of high shear force due to high circulation flowrate. According to Rodriguez *et al.*¹⁵, the shear force of the fluid flow is capable of carrying the solute in the solution. Therefore, this could be the reason why high circulation flowrate can produce high concentration of sugar in the CW solution at the end of the process in this study. High shear force due to high flowrate could bring the sugar away from the surface of the ice layer and also could easily remove the sugar that is trapped between the dendritic ice structure which has been

formed, thus leaving the sugar in the liquid phase. As a result, a concentrate with high sugar concentration was obtained.

As indicated by the two lines, the decreasing value of K resulted from the increasing percentage of sugar increment. It is evident that the smaller the amount of sugar in the resulting ice, the higher the concentration of sugar in the concentrate produced. This graph also proves that increasing circulation flowrate could give increasing performance of PFC system.

3.2 Effect of Circulation Time

The effect of circulation time to this process was also investigated through this experimental series. The same experimental method was used but with varying circulation time from 10 minutes to 18 minutes. This time, the circulation flowrate was kept constant at 2800 ml/min while the other two, initial solution concentration and coolant temperature was kept at 3.0 % Brix and -12°C respectively. For the purpose of indicating the effect of this factor on ice block contamination and total sugar concentration at the end of this process, graph is plotted against the K value and increment percentage of sugar respectively.

The K value correlate with the time used was summarized in Table 2 and its effect can be seen in Figure 6. The value of K increases with increasing time of PFC, as indicated by the line of the graph. It is observed that the K value is the lowest on the tenth minute which is 0.27. These values continue to increase in the next minutes and it is undesirable for this system. Therefore, it can be said that 10 minutes is a suitable circulation period of solution in order to operate PFC in high efficiency. This result can be supported based on a study made by Jusoh¹⁶ which had found that the solute content in the ice layer formed is satisfactorily low at 10 to 15 minutes of process duration. Below than 10 minutes, the ice layer produced is not solid and in dendrites structure which indicates incomplete PFC process.

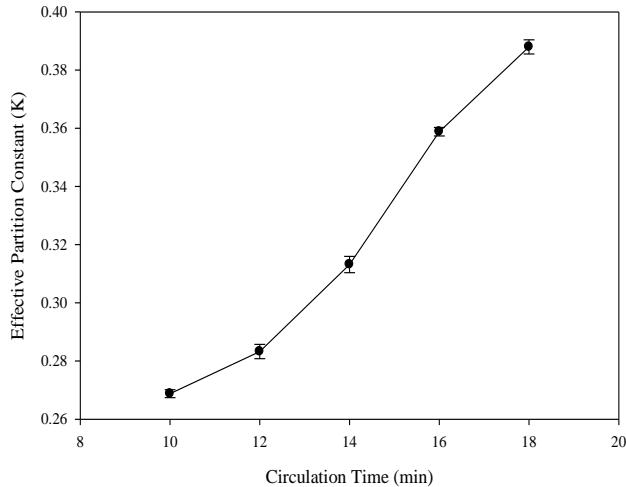
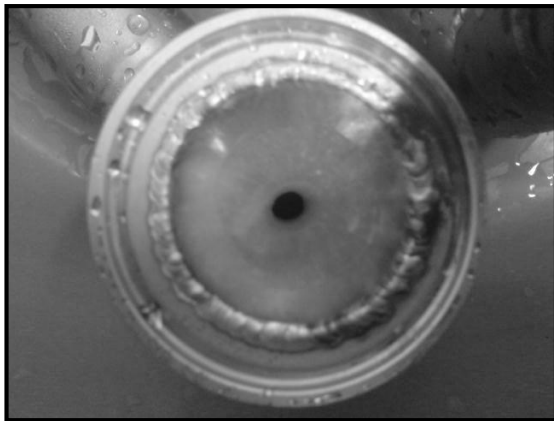
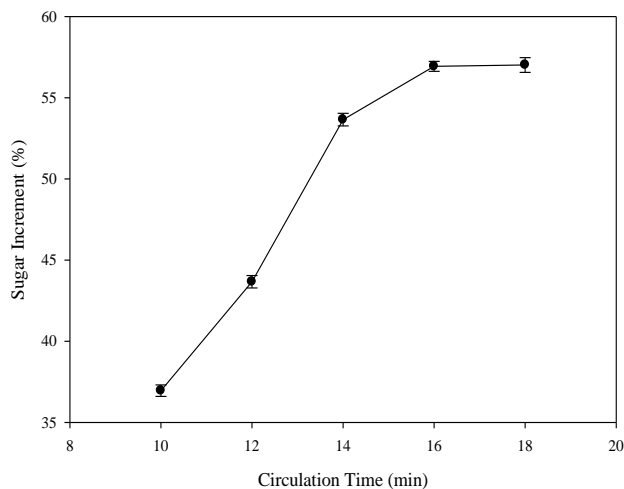
This finding can be explained based on what has happened when the process was carried out until 18 minutes during the experimental work. The ice formed almost filled the whole volume of the crystallizer. This is shown in Figure 7. This condition reduced the path diameter of the fluid flow, thus produces a narrow space for concentrate to circulate. Therefore, it becomes easier for the ice growth to trap the saturated solute in the concentrated solution, giving a higher value of K.

Figure 8 shows a plotted graph corresponds to the enhancement of sugar content by this parameter. After 10 minutes of operation, there is only 36 percent increment observed from the 3 %Brix sugar concentration of the original solution. The percentage value was then increased significantly in the next period of operation time of 12 to 16 minutes, up to 56 percent, and at 18 minute, the percentage is constant.

The greater influence given by circulation time is on the rate of solute concentration rather than on rate of ice formation.¹³ Bayindirli *et al.*¹⁷ had described the same trend in other studies. This can be explained as follows: in FC process, there is a combination of heat and mass transfer¹⁸ where the mass transfer rates in directional freezing is normally slower than the rate of heat transfer due to low mass diffusion coefficients and high thermal conductivity of ice. Therefore, there is more time available to promote mass transfer and increase the rate of concentration when the time given for PFC to take place is longer.

Table 2 Value of K obtained at each circulation time

Circulation time (min)	K value
10	0.27
12	0.28
14	0.31
16	0.36
18	0.39

**Figure 6** Effect of circulation time on K**Figure 7** Close up of nearly full crystallizer**Figure 8** Effect of circulation time on increment of sugar concentration

Besides that, it also gives more time to remove water from the liquid phase. As a result, a high increment of sugar is achieved. This fact can explain why in 10 minutes, the ice produced is in high purity but the sugar increment is lower than the subsequently minute.

However, too long of operation time is unfavorable for this process as discussed before. Therefore, from the two provided graph, it can be concluded that the most suitable period for the solution to be circulated is 14 minutes instead of 10 minutes as described previously. This is because, at 14 minutes, the K value is not really high and acceptable. Most importantly, at this minute of operation, the sugar content was increased more than 50 percent which is considered as good enough for this scale of study.

4.0 CONCLUSION

This work has focused on the effect of two important process parameters towards PFC process. The results revealed that high circulation flowrate and intermediate value of circulation time resulted in better efficiency based on K value and increment of sugar concentration. This work also has also proven that the CW can be concentrated efficiently by using PFC system.. Nevertheless, the effect of initial concentration and coolant temperature on the system efficiency should also be investigated in order to know its best performance.

Acknowledgement

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