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# Efficiency Study on Vertical-Finned Crystalliser for Concentration of Carrot Juice

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Vertical-Finned Crystalliser (VFC) is a new innovation of the progressive freeze concentration method which is applied in concentration of carrot juice. The aim of this research is to observe the efficiency of this newly designed crystalliser in preserving the beta carotene content in the carrot juice concentrate. The crystalliser is equipped with a cooling jacket and extended surface of heat transfer area which makes it possible to obtain high efficiency operation, highly concentrated carrot juice and pure ice layer in the process. The process started with feeding the carrot juice into the crystalliser and the ethylene glycol coolant was pumped into the cooling jacket. Both carrot juice and coolant were circulated in the crystalliser and cooling jacket using two different pumps, until a layer of ice was formed on the inner wall of the crystalliser, including the extended surface/fins. The range of coolant temperature investigated was -6 °C to -12 °C and circulation flowrate of 1,600 mL/min to 2,800 mL/min at constant operation time of 50 min. The efficiency of the process was observed based on the average concentration efficiency value, η (%) and effective partition constant, K using concentration value from the sample absorbance analysis result of UV-Vis spectrophotometer. It was found that the increase in freezing rate (-8 °C) brought about the increase in average concentration efficiency value, η and lower K-value. Meanwhile, the best circulation flow rate was 2,800 mL/min giving higher value of efficiency and lower K-value. The present concentration method using PFC with VFC will be applicable in the industry especially to produce a variety of concentrated liquid food.

## 1. Introduction

Carrot is one of the famous vegetables and always plays an important role in our lives as it provides us the nutrient that our body needs. To produce this juice, carrots juices are required to be concentrated; with high level of dissolved carrot in the juice. This is because carrot contains 88 % of water. Only 12 -13 % remains as the concentrated carrot itself. The remaining concentrated carrot includes nutrients such as phenolic, carotenoids and vitamins (Jabbar et al., 2014) and antioxidant (Aadil et al., 2013) that are good for skin protection. It also reduces the infection of acne, pre-wrinkles, dry skin and skin damage. The richest source of vitamin A and B inside the carrot juice also improves eye visibility, our defence against heart disease as it is enriched with Lutein, Beta-carotene and Alpha-carotene. The presence of fibre also helps to clean the colon inside the intestine and reduce fat formation in the liver. For mental health, it reduces depression level and improves a child's memory if it is consumed regularly.

Freeze concentration is a method to separate water component from a solution by freezing. The advantage of freeze concentration compared to other concentration methods such as evaporation and reverse osmosis is mainly on the fact that it requires less energy as the energy of fusion of water is lower than the energy of vaporisation, and it does not require high cost to attain osmotic pressure. Besides, it is also can avoid loss of aromatic compound. Generally, there are two types of freeze concentration, which are suspension freeze concentration (SFC) and progressive freeze concentration (PFC) and each is superior in some ways. Most of the large industries nowadays use suspension crystallization as the preferable method because it can be scaled up for large amounts of product. Ice crystals are produced as a suspension in the mother liquor as

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cooling is supplied while the solution is stirred. SFC is mostly used in production of dairy products, brewing, winery and distilling citrus fruits juice (Nistelrooij, 2005). However, the disadvantage of this method is that it requires a complex structure frame including scraped-surface heat exchanger for ice seeding and high pressurised system which results in higher operating cost.

As a result, the PFC method was later introduced, where in principle; ice layer is produced layer by layer on cooled metal surface. The concentrated solution can then be separated from the ice layer just by draining/pumping it out of the system. Commonly, there are three types of cooled metal used which are coil, stainless steel and aluminium. The reasons why this method is more reliable than SFC is because this process does not require ice crystals washing, it works at normal atmospheric pressure, no pressurises are needed and requires a simpler frame structure compared to the SFC. In addition, it requires low energy with reduced capital cost as no ice crystal washing and filtration are required. PFC is an attractive method as it could give high quality production with low capital and operating cost. It has already been tested with fructose, glucose and sucrose with sugar solution and the result shows 30° Brix of concentration was increased from the initial concentration and it has also been tested for pear and apple juice (Hernández et al., 2010). PFC also produces a large single block of ice crystal on the plate compared to the small ice crystals produced by SFC.

Many designs have been proposed by various researchers: tubular ice system (Miyawaki et al., 2016); pilot plant falling film cryoconcentrator (Hernandez et al., 2010); cylindrical sample vessel of stainless steel system (Liu et al., 1997); ice maker machine (Williams et al., 2013). However the designs still have some shortcomings and the main problem is that the production quality is much lower when compared to SFC. Thus, this new design of vertical finned crystalliser (VFC) was invented to tackle this shortcoming, by introducing some extended surface as fins on the inner surface of the crystalliser. Hence, the surface for ice crystal formation is increased compared to a crystalliser with no fins. With the presence of fins, the heat transfer area is extended to concentrate more solution with less handling (Amran et al., 2016). Hypothetically, the extended surface area would increase the productivity of such system. The aim of this research is to investigate the efficiency of this design in terms of the effect of coolant temperature and solution flowrate which would reflect the extent of improvement the fins could provide. This is to facilitate easier adaptation to the industry in terms of scaling up and handling of different capacities of target solution.

## 2. Materials and Method

### 2.1 Materials

The carrots were purchased from a local supermarket in Taman Universiti in the same grade and brand. Then, the carrot was ground by using Hanabishi Juicer to get the fresh juice of carrot in about 1.15 L to be put in the target solution tank before being fed to the crystalliser. The coolant in the waterbath contains 50 % volume (v/v) mixture of ethylene glycol and water, and pure acetone was added in the concentrated carrot juice as the extractor for beta carotene. Meanwhile, high purity beta carotene powder was used as a reference for comparison between the experimental and actual results.

### 2.2 Vertical-finned crystallizer

Stainless steel vertical finned crystallizer (VFC) that was designed by Amran et al. (2016) has been chosen because of its compatibility and high resistance against corrosion, as well as its high thermal conductivity (17 W/m.°C) (Perry and Green, 1997). The design of the crystallizer was chosen to be cylindrical to provide large and smooth surface for solution movement (Samsuri et al., 2016). The cylinder was covered by polyurethane foam to reduce the exchange of heat with the surrounding and to increase the rate of ice crystal formation during the experiment. In order to have larger surface contact for heat transfer between the solution and the coolant, the VFC was equipped with four vertical fins with 2 cm length, 1.5 cm width and 30 cm height where the fins would increase the surface area by 63.7 % as shown in Figure 1a. As a result, higher rate of ice production would occur as the larger surface area enhances the heat transfer. The crystallizer could hold up to 1.15 L solution. VFC cylindrical body was covered by the cooling jacket to enable circulation of coolant liquid to provide the cooling energy. To ensure that the system has enough cooling energy, the size of the coolant jacket was carefully designed in order to get a high rate of ice crystal formation. The target solution entered the crystallizer from the bottom inlet as shown in Figure 1a, to prevent bubble formation. In circulation, the target will flow out of the crystalliser from outlet at the top of the crystalliser. A relief valve is installed at the top of the crystallizer to facilitate the volume expansion of ice inside the crystallizer during the experiment. Insulated lid was also placed on top the crystallizer for ice visualization and sampling purposes. The solution, wall and coolant temperature were profiled using thermocouples placed at some designated positions. The temperatures then were displayed on a computer using a Picolog data acquisition tool during the operation.

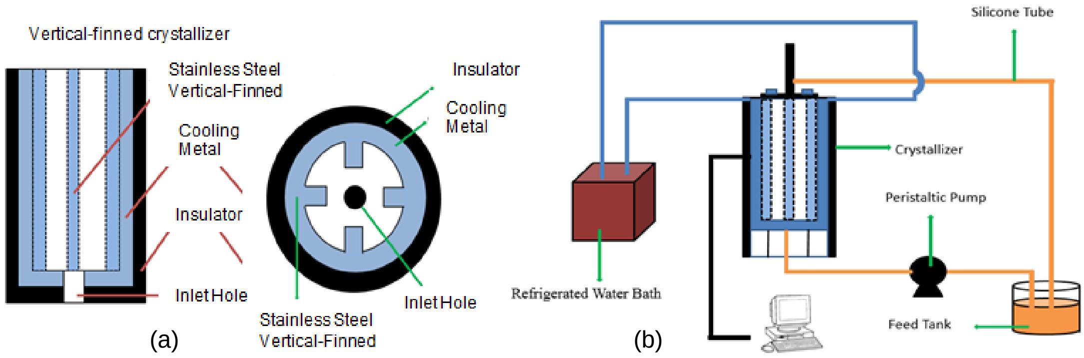


*Figure 1: (a) Inner structure and upper view of VFC (b) Experimental set-up*

### 2.3 Experimental procedure

Carrot juice was prepared by juicing raw carrot. 1.3 L of carrot juice was obtained and placed in the fridge and another 350 mL of carrot juice was frozen in the freezer. The juicing process should be done a day before the experiment took place. The experimental setup used is as shown in the Figure 1b next day. Ice seeding was performed before the carrot juice could be introduced in the crystalliser. Beforehand, the refrigerated water bath was turned on to bring the coolant to the desired temperature. As the coolant achieved the desired temperature, the coolant was pumped from the refrigerated water bath to the crystallizer cooling jacket for 5 min. Distilled water was then fed to the crystalliser and circulated through the crystalliser for 10 min with an average speed of 1 700 mL/s (300 rpm). Seed ice crystal layer formed on the inner wall of the crystalliser and uncrystallised water then was discharged from the crystallizer. Then, cold carrot juice was placed in the feed tank, mixed with frozen carrot solution from the freezer. This is to maintain the temperature of the carrot solution at 2 °C. A sample of this target solution was taken for analysis. The experiment began as the temperature of the refrigerated water bath and the flow rate of the peristaltic pump were fixed at the desired conditions for 50 min. All of the experimental procedures were followed as stated, and repeated at different temperature of refrigerate water bath and different flow rate of peristaltic pump for carrot solution. During the 50 min, the Picolog tool measured the temperature through the thermocouple attached to the crystallizer. The concentrated carrot solution was then collected to measure its volume by using measuring cylinder. After its volume was measured, 15 mL of the solution was taken for analysis. For detachment of the ice layer formed inside the crystallizer, the coolant first was discharged back to the refrigerated water bath. Then, the cooling jacket was filled with tap water in order to melt the ice layer. The tap water was connected via silicone tube for the inlet and outlet of the coolant jacket. The melted ice was collected through the bottom opening (inlet of crystallizer) of the crystallizer via silicone tube to the 1,800 mL beaker. After all of the melted ice was collected in the beaker, its volume was taken and recorded. 15 mL of the sample was taken for further analysis.

### 2.4 Analytical Method

UV-VIS spectrophotometer was used as an analytical measurement to record the absorbance of the beta-carotene in each sample. 16 experiments were carried out and each experiment consisted of three samples which including initial sample (sample 1), concentrated sample (sample 2) and ice sample (sample 3). Each of these 3 samples were analysed in order to obtain the absorbance of beta-carotene. Acetone were used as the solvent to extract the beta-carotene in the solution before being analysed using UV-VIS spectrophotometer. The samples were placed in the freezer for preservation purpose. 1 mL of the sample were taken and put into the test tube. 5 mL of 'chilled' acetone was added to the sample. The test tube then was placed on a centrifuge and was run at 2,500 rpm for 35 min. The test tube was then taken out and 5 mL more 'chilled' acetone was added. the test tube was placed again on the centrifuge for another 10 min at 2,500 rpm (Biswas et al., 2011). The sample was then analysed using the UV-VIS spectrophotometer to check for its absorbance. The mixed sample in the test tube was inserted to a cuvette of UV-VIS spectrophotometer and placed onto the UV-VIS spectrophotometer for measurement.

## 3. Results and Discussion

### 3.1 Determination of Beta-Carotene concentration

The highest peak for carrot solution absorbance throughout UV-Vis was obtained at 451 mm. From the reading of UV-VIS spectrophotometer, the concentration of beta-carotene in the carrot juice was calculated through the beta-carotene (carrot) standard graph (Javer et al., 2013) as shown Figure 2, where the slope is shown in Eq(1) below:

$$Y=0.1475X + 0.0025 \quad (1)$$

where, Y is referring to the value of absorbance that has been substituted in the equation to get the result of concentration of beta-carotene, X. Then initial sample, concentrated sample and ice sample calculation of beta-carotene concentration based on the standard graph were calculated, as tabulated in Table 1. From the table, it is clearly shown that the highest concentration belongs to samples run at coolant temperature at -8°C and circulation flowrate of 2,800 mL/min.

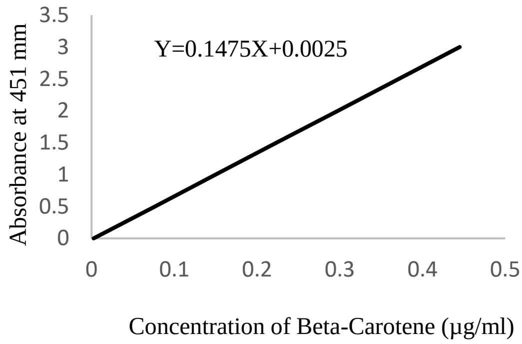


*Figure 2: Standard curve of beta carotene (carrot) graph for determination of sample concentration (Javer et al., 2013)*

*Table 1: All concentration obtained using the Beta-Carotene (carrot) standard graph*

| Temperature | Speed (mL/min) | Concentration of beta-carotene on initial sample (µg/mL) | Concentration of beta-carotene on concentrated sample (µg/mL) | Concentration of beta-carotene on Ice sample (µg/mL) |
|---|---|---|---|---|
| -12±1°C | 2,800 | 11.09 | 13.23 | 6.73 |
| | 2,400 | 12.40 | 14.33 | 8.37 |
| | 2,000 | 14.34 | 16.13 | 11.92 |
| | 1,600 | 12.54 | 13.98 | 11.87 |
| | 2,800 | 11.09 | 13.23 | 6.74 |
| -10±1°C | 2,800 | 10.44 | 14.11 | 6.47 |
| | 2,400 | 9.66 | 12.01 | 6.25 |
| | 2,000 | 10.21 | 12.53 | 8.69 |
| | 1,600 | 12.46 | 14.52 | 11.47 |
| -8±1°C | 2,800 | 14.11 | 18.67 | 6.58 |
| | 2,400 | 11.98 | 16.52 | 6.35 |
| | 2,000 | 11.24 | 13.92 | 8.16 |
| | 1,600 | 13.43 | 15.71 | 10.39 |
| -6±1°C | 2,800 | 10.22 | 12.09 | 4.69 |
| | 2,400 | 10.52 | 12.02 | 5.17 |
| | 2,000 | 12.57 | 13.44 | 8.51 |
| | 1,600 | 12.68 | 13.16 | 10.03 |

### 3.2 Average Concentration, η (%) as affected by coolant temperature and circulation flowrate

As the sample concentration was obtained, average concentration efficiency, Ƞ was determined through the following Eq(2) (Hernández et al., 2010):

$$\text{Efficiency (\%)}, Ƞ = (C_{FS} – C_H) \times 100/ C_{FS} \quad (2)$$

where, $C_{FS}$ indicates beta-carotene concentration of concentrated solution obtained while $C_H$ indicates the beta-carotene concentration left in the ice. In theory, the less beta-carotene left in the ice, the higher its concentration in the concentrated solution at the end of the process.

Figure 3a shows that the average concentration efficiency, η tends to increase as the temperature increases from -12 °C to -8 °C and starts to decrease after the temperature is brought down to -6 °C. Highest average concentration efficiency, η at coolant temperature -8°C is correlated to the dependency of the crystal growth onThe freezing rate of carrot juice, where lower freezing rate brings about ice crystals growth in layer with complete flat solid-liquid interface process. On the contrary, higher freezing rate resulted in low average concentration efficiency, η due to enlarged interface between the ice and carrot juice, hence enhancing crystal growth and attracting the beta-carotene content in the carrot juice. Figure 3a also indicates that the circulation flowrate used in the experiment at 2800 mL/s reached the highest result for average concentration efficiency, η. As time passes at constant operation time 50 min, the higher circulation flowrate influenced lower ice contamination. This is supported by Wiliams et al. (2013) where the highest result of average concentration efficiency was reflected by the reduced amount of trapped beta-carotene in the ice phase as flowrate increased (Wiliam et al., 2013).

**3.3 Effective partition constant, K by coolant temperature and circulation flowrate**

In freezing process, the effective partition constant of a solute, K, between the ice and the liquid phase at the interface is formulated as follows Eq(3) and Eq(4) (Liu et al., 1997):

$$K=C_S/C_L \tag{3}$$

where $C_S$ indicates the concentration of concentrated sample and $C_L$ indicates the concentration of ice sample. As the equation is integrated, another equation can be generated based on the concentration and volume of target (origin) and concentrated sample in order to find the value of K. The equation is stated as follows:

$$k = [\log(\frac{C_o}{C_L})/log\ (\frac{V_L}{V_o})] \tag{4}$$

where $C_o$ is concentration of initial carrot solution, $C_L$ is concentration of carrot, $V_o$ is initial volume of carrot and $V_L$ is volume of concentrated carrot (final volume of solution).
As for the relationship between the effective partition constant, K and the coolant temperature (Figure 3b), the K-value declined from -12 °C to -8 °C and increased back at -6 °C. This is because the lower temperature can lead to extreme freezing towards the carrot solution which can trap the beta-carotene into the ice during ice formation, thus increasing contamination. Meanwhile, the lower K-value resulted from temperature -8 °C is due to the fact that at this temperature the ice formation was slow and complete, reducing solute entrapment. However, at -6 °C, K increased as the formation of ice is not complete because of inadequate cooling energy and the ice crystal layer formed was dendritic in structure, thus leading to high beta-carotene entrapment in the interface.

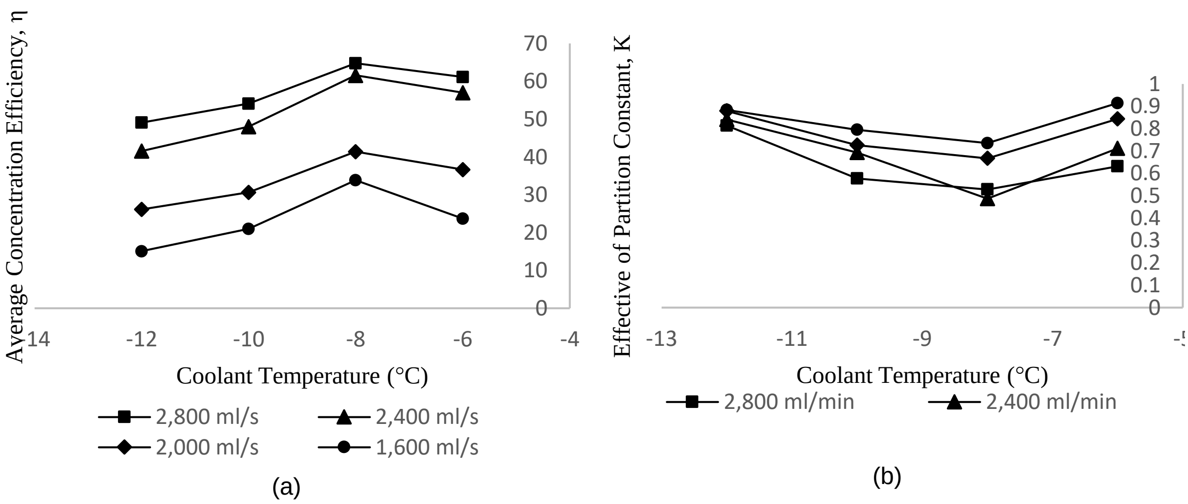


*Figure 3: (a) Average Concentration Efficiency, η vs Coolant Temperature (°C) and Circulation Flowrate (b)Effective Partition Constant, K vs Coolant Temperature (°C) and Circulation Flowrate.*

The flow rates applied for this research were 2,800 mL/min, 2,400 mL/min, 2,000 mL/min and 1,600 mL/s. The trend of the graph is inclined as the flow rate increases proportionally to the lower K-value. The lowest point obtained from the graph is at 2,800 mL/min. The four colour lines indicate different flowrates.

The graph also shows K-value increased as the flow rate increased. The lowest point of K-value is at 2 800 mL/min. Both η and K show positive result as the calculated results are in agreement with previous studies of circulation flow rate effect on effective partition constant in progressive freeze-concentration of 8% sucrose solution (Miyawaki et al., 2016) through tubular ice system by Miyawaki et al. (2016). In addition, other previous studies that support the result by Samsuri et al. (2015) in their study to investigate the effect of circulation flow rate on the performance of a spiral finned freeze concentrator using glucose solution (Samsuri et al., 2015).

## 4. Conclusions

As a conclusion, the performance of the vertical finned crystallizer (VFC) in terms of ice formation is indeed influenced by coolant temperature and circulation flow rate. From the result, it was found that the best parameter for the coolant temperature is at -8 °C as it shows the highest point for average concentration efficiency value, η (%) and lowest point of effective partition constant, K. Meanwhile, the best condition of circulation flow rate is 2,800 mL/min reflected by the higher value of efficiency, η and the lower value of effective partition constant, K.

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