

# Concentration of Cucumber Juice Using Progressive Freeze Concentration for Total Phenolic Content Increment

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## ABSTRACT

Concentration process of cucumber juice is an essential step in cucumber extracts and juice production in the industry in order to minimise the volume and weight for storage and handling purposes besides to keep the quality of the products. Enriching total phenolic content (TPC) in cucumber juice that contributes to anti-oxidants characteristics is one of the objectives in this study due to high demand in the industries especially in food and beverages, pharmaceutical and cosmetology. Studies and researches are highly needed to overcome the limitations and shortcoming of the current methods in concentrating fruit juices which are evaporation, reverse osmosis, and freeze concentration. This research aims to study the effects of operating parameters on concentration process of cucumber juice which involved in progressive freeze concentration (PFC) process includes coolant temperature, stirrer speed, operation time and initial concentration. New design of PFC called as multiple probe cryo-concentration system (MPCC) was used throughout this process. The multiple probes inside the system were freeze out the solution and form an ice block layer by layer. During the ice crystallisation process, formation of ice crystal lattice will reject all impurities and leave behind highly concentrated of cucumber juice. The effect of coolant temperature, stirrer speed, operation time and initial concentration was investigated in this work. The investigation revealed that lower coolant temperature led to a better performance efficiency of the system at -12 °C. Meanwhile, a higher stirrer speed and longer operation time gave a lower value of K and TPC increment at 350 rpm and 40 minutes, respectively. High initial concentration at 10 mg/ml was observed to give high value of K which is not favourable but at that value TPC increment is the best. Findings from this research have shown that PFC is a potential method to be engaged in fruit juice concentration, particularly for cucumber juice.

**Keywords:** Progressive freeze concentration, cucumber juice, ice crystallisation, fruit juice

## 1. INTRODUCTION

Cucumber juice is highly nutritious and a rich source of polyphenols which contributes to anti-oxidants characteristics, therefore the concentrate and extracts have been widely used for further application in the industries such as food flavouring, pharmaceutical, skincare and cosmetology. Companies are focusing on cucumber juice production and the researchers are always looking for the best techniques or methods to achieve good results and produce highly concentrated cucumber juice with enriched total phenolic content in the process of convergence for further analysis and reactions. For beneficial consumption in both the food or cosmetic industries, rather than using the whole cucumber, it is more practical to reduce the water content in its juice as the vitamins, minerals, and active components can be concentrated. In order to provide easy handling for import export process, concentration in food processing is essential to reduce the weight, volume, packaging, transportation cost [1], but the quality of the product is practically of concern. There are several ways

available for fruit juice concentration methods and the most popular are evaporation, reverse osmosis (RO) and freeze concentration.

Evaporation engages high temperature in the operation; thus, the high temperature operating condition will cause nutrition decomposition and disrupt the biological structure especially the total phenolic content (TPC) of the cucumber juice itself [2]. This reduces the concentrate quality and cucumber juice with high concentration could not be obtained. Meanwhile, RO is another increasingly popular method for fruit juices concentration which uses water selective membrane in the separation of liquid and the solute. However, in most cases, the process produces limited yield due to high osmotic pressure and clogging of the membrane can easily happen which will create low flux [3]. Meanwhile, freeze concentration which is a method of concentration by removing water constituent as ice crystals at sub-zero temperature has only the problem of low productivity of the concentrated product [4], however the process is free from heat abuse of sensitive food constituent or component. Looking at all the disadvantages of existing concentration methods, this research proposes the use a new

design of progressive freeze concentration to concentrate cucumber juice, which is expected to be able to preserve the juice's nutritional quality especially the TPC content and enhance the concentrated juice. Two modes of freeze concentration are suspension freeze concentration (SFC) and progressive freeze concentration (PFC).

SFC process starts from ice crystal nucleation and followed by the growth of ice crystal from the solution which is the second phase of the process. The formation of seed ice crystal is initiated by using scraped surface heat exchanger (SSHE) and uses a stirrer as the agitator to enhance the process of agglomeration. However, this separation method is quite complicated to be applied due to size limitation of ice crystals formed in the process and higher capital cost especially from the cost of SSHE and more unit operation involved in the ice crystal separation [5].

In PFC, the formation of ice crystal lattice on the cooled surface is formed layer by layer from the water content in the cucumber juice and as a result, producing a single ice block. In the end, the separation of ice block and the concentrate is much easier compared to SFC [4]. Besides, PFC requires low energy requirement and the process temperature is low thus inhibiting unwanted biochemical and chemical changes [6]; [7]; [8]; [9]; [10]. Moreover, highly concentrated cucumber juice can be achievable because theoretically, the formation of ice crystal lattice rejects all the unwanted impurities and leaves behind a highly concentrated cucumber juice. This process has never been widely practiced in cucumber juice concentration before. It is therefore important to study the effect of the concentration process on cucumber juice solutions to offer a new technique to produce a desirable cucumber juice concentration process.

## 2. EXPERIMENTAL

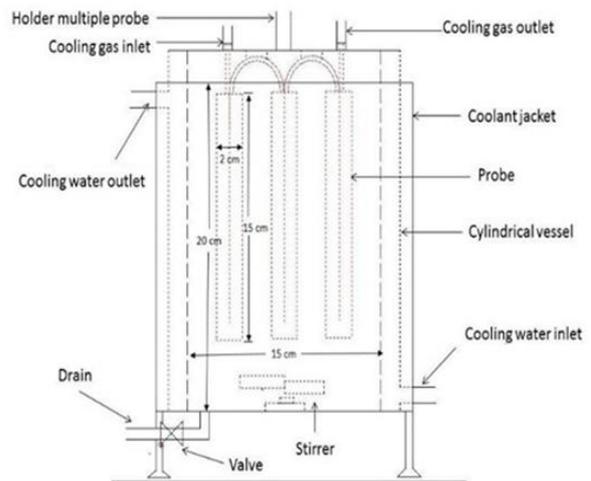
### 2.1. Materials

Cucumber juice was used as raw material in this experiment. Fresh cucumbers were purchased from Maslee Express, a local supermarket in Johor, Malaysia. Ethylene glycol was the coolant liquid used and mixed with distilled water (50/50 volume to volume ratio) in the water bath throughout the process. Folin-Ciocalteu reagent and anhydrous sodium carbonate powder were used for phenolic content analysis.

### 2.2. Experimental Setup

The new design of PFC system called multiple probe cryo-concentration (MPCC) is shown in Figure 1, which consists of a coolant gas system which is responsible for cooling through suitable refrigerated probes with attached motorized stirrer at the bottom of solution tank. The probe set is immersed in a solution tank and the five-finger probe can be rotated at 180° interchangeable in direction. On the

other hand, a stirrer was attached and the speed used was in the range of 250-400 rpm in order to maximize the ice formation on the outer surface of the probe and form a well-mixed solution.



**Figure 1** Schematic diagram of multiple probe cryo-concentration system

First, cool cucumber juice was placed in the solution tank and the temperature was set to be kept constant at close to the freezing temperature at 2°C which was provided by the coolant liquid used in the outer tank jacket. Next, the refrigerant gas circulation was activated by setting up the desired value of temperature and condition in order to supply cooling to the PCC system and as the temperature was reached, the probes were immersed in the solution and rotated at 180°. When cooling was adequate, the ice crystals started to develop on the outer surface of the probes and a concentrated cucumber juice was left behind in the tank.

A thermocouple was used to measure the temperature of the solution and the data obtained were displayed online throughout the experiment for easy monitoring. The experiment was stopped after applying the desired cooling rate and certain time duration, the concentrated solution of cucumber was then collected, and the layer of ice crystal was thawed. Next, the concentrated cucumber juice was determined and analysed for water content by using UV-Vis spectrophotometer and Folin-Ciocalteu method. Performance of the concentration system was evaluated using effective partition constant (K-value) via the following equation:

$$K = C_s / C_L$$

where  $C_s$  is the TPC in the solid (ice) and  $C_L$  is the TPC in the liquid.

### 3. RESULTS AND DISCUSSION

Figure 2 shows the colour comparison of the feed cucumber juice with the ice produced and concentrated cucumber juice obtained at the end of one of the experiments. It can be seen that the intensity of the colour of the concentrated cucumber juice is higher than the feed cucumber juice that indicates the effectiveness of the concentration process. Also, the colour intensity of the thawed ice solution is much less concentrated than the feed cucumber juice which indicates the ice crystal purity formed is suitable during the crystallisation process and proving that PFC process has high efficiency in the cucumber juice concentration process.

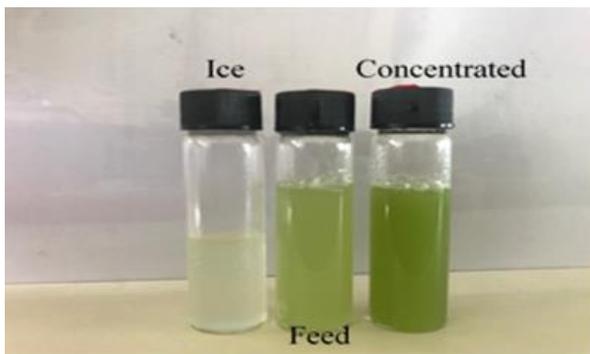


Figure 2 Comparison of feed cucumber juice and final products

#### 3.1. Effect of Operating Conditions towards K and TPC

Effect of operating condition is important to be studied in order to evaluate the performance of the system and obtain good results in achieving better concentration efficiency especially in TPC as well as productivity of the process by using the MPCC. Four operating parameters that give most effects to the PFC process include coolant temperature (CTemp), stirrer speed (StSp), operation time (OpTime) and initial concentration (InCon). The performance and response of the PFC process were evaluated based on the value of effective partition constant K and increment of total phenolic content (TPC) in the cucumber juice.

##### 3.1.1. Effect of Coolant Temperature

Coolant temperature is an important parameter that majorly influences the PFC system as it controls the growth of ice crystals and the ice front growth rate in the PFC process [11]. For the purpose of studying the trends of coolant temperature that affects the system, the coolant temperature was varied from -8 to -14 °C and all the other three operating parameters were maintained with operation time, stirrer speed and initial concentration of the cucumber juice solution kept constant at 30 minutes, 350 rpm and 6 mg/ml

respectively. The range of coolant temperature was decided based on the results from preliminary screening process where, the ice layer formation started to form at -8 °C. Figure 3 shows the TPC increment and value of effective partition constant K based on coolant temperature.

From Figure 3, it is shown that the optimum coolant temperature for cucumber juice concentration by using PFC system was at -12 °C, where the best increment of TPC and effective partition constant K were achieved with 22 percent and 0.24 respectively. The trend of TPC increment of cucumber juice solution increased when the coolant temperature applied increased from -8 °C until it reached its peak at -12 °C and started to decrease when the temperature approached -14 °C.

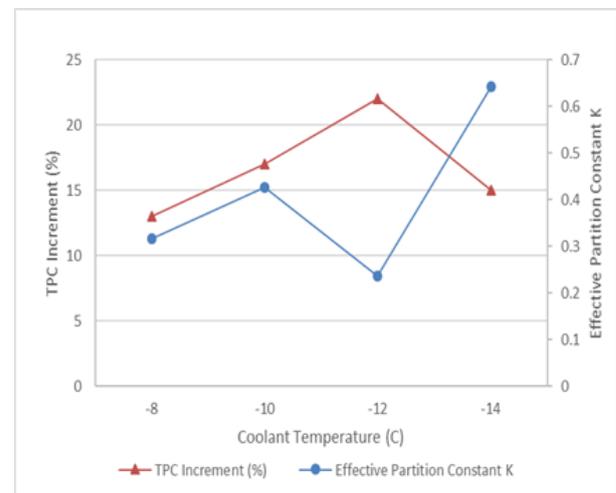


Figure 3 Changes of TPC increment and effective partition constant K with coolant temperature

The value of effective partition constant K increased when the temperature from -8 °C to -10 °C was applied to the system, and it started to decrease up to -12 °C and reached its minimum value with 0.24 which portrays the highest performance efficiency of the PFC system. Then, the trend of K value sharply increased at -14 °C.

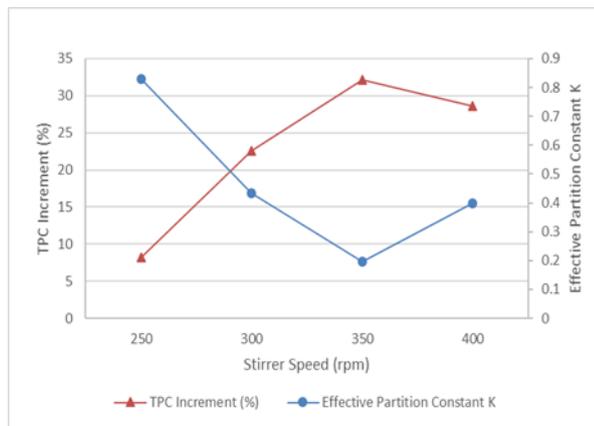
As mentioned by Miyawaki *et al.* [11], the formation of ice layer mainly depends on the coolant temperature applied. However, a too low coolant temperature will cause the ice growth rate per unit time increases [11]. Lower coolant temperature produces larger size of ice crystal formed [12]. The rate of ice growth is increased when the difference of temperature between the probes where the coolant gas is channelled and entering solution is high. This condition will increase the tendency of the solute being trapped in the outward movement of the ice front, and as a result, the effective partition constant K will be high which is unfavourable for the PFC system. This explanation has proved the reason why the K value sharply increased when approaching -14 °C.

It would be necessary to minimize the ice crystal entrapment in order to produce a higher quality cucumber juice with highest TPC increment. It is best if the

components of TPC remain in the solution in the process of impurities rejection during ice crystal lattice formation. The mass transfer of solutes must be larger than the rate of ice growth for the process of elution to occur [13]. The components have a high possibility to be trapped into the ice layer formed if the TPC components gather near the interface. In the process regarding TPC increment, the ability of hydrogen bond plays a major role to bind a large number of water molecules. The interstitial water is less available for freezing when the concentration of TPC in the solution is high. Hence, TPC components are trapped in the ice crystal and this will decrease the PFC process efficiency [14]. In the industrial scale, loss of valuable and precious phenolic contents in fruit juice production is common [15].

### 3.1.2. Effect of Stirrer Speed

Stirrer speed is one of the four important operating parameters involved in the PFC system. The influence of stirrer speed was investigated while the other three operating parameters were kept constant in the process. The stirrer speed was varied from 250 to 400 rpm while coolant temperature, operation time and initial solution concentration were kept constant at -10 °C, 30 minutes, and 6 mg/ml respectively. Figure 4 shows the TPC increment and effective partition constant K value based on stirrer speed.



**Figure 4** Changes of TPC increment and effective partition constant K with stirrer speed

From Figure 4, it is clearly shown that as the stirrer speed increased, the trend of TPC increment also increased and the trend of effective partition constant K decreased which indicates better efficiency of the system. The best achievement of 32 percent of TPC increment and 0.2 K value were obtained at the stirrer speed of 350 rpm. This result is consistent with the findings obtained by Miyawaki et al. [11] where the advance rate of ice front is lowered when a higher flowrate is applied to the system and producing high purity of ice [11]. Also, this outcome is in line with the findings by Okawa *et al.* [1] where a slower

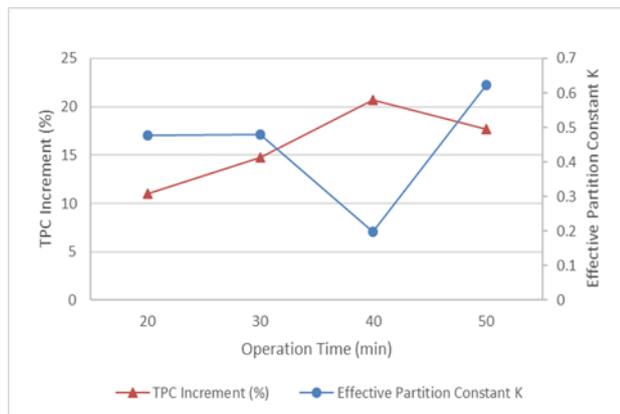
solidification rate is produced when higher flowrate is applied and hence, lowering the ice concentration captured, resulting in higher ice purity [1]. The mass transfer coefficient is increased when the solution velocity in the system is increased, and the condition encourages the solutes in the ice-liquid interface to be brought to the solution, thereby reducing the ice crystals inclusion [16]. Besides that, as stated by Rodriguez et al. [12] the fluid flow shear force is capable of carrying the solute in the solution. High shear force that occurred in the fluid flow when 300 rpm stirrer speed was applied has brought away the phenolic content that is entrapped between the ice layer dendritic structure and remained in the concentrated solution and hence, this could be the justification why high stirrer speed can produce high TPC increment as the final product of the process [12]. However, the effective partition constant, K value, obtained at 400 rpm which was also the highest stirrer speed applied to the process is higher than that for 350 rpm which indicates a lower efficiency. The possible justification behind this phenomenon is that there was a potential of the ice layer formed on the probe to erode when the stirrer speed applied was too high, and hence the concentration of solution in the liquid was reduced due to the increase of the volume of pure water in the solution. Consequently, the increment of the volume of pure water in the solution has affected and decreased the concentration of solution in the liquid after the erosion has occurred [17].

### 3.1.3. Effect of Operation Time

The same experimental method was conducted in investigating the effects of operation time towards PFC process. Operation time range was varied from 20 to 50 minutes while coolant temperature, stirrer speed and initial concentration of the cucumber juice solution were kept constant at -10 °C, 350 rpm and 6 mg/ml respectively. Theoretically, the operating parameter of operation time will produce high concentration of cucumber juice and higher purity ice crystal if a longer operation time is applied to the PFC process [18]. However, up to a point, if the operation time runs too long, the deterioration of the ice purity will start to occur because of cucumber solutes inclusion in the layer of ice. This condition happens due to the degree of saturation of the solutes of cucumber in the solution that rises over time and hence, it will induce the inclusion of solute in the layer of ice produced [19]. Figure 5 shows the trend of effective partition constant K and TPC increment based on operation time applied.

From Figure 5, it can be seen that the increment of TPC increased and reached its peak at 40 minutes of operation time with 20.6 percent increment and decreased at 50 minutes operation time. However, the trends of effective partition constant K is the other way around where it decreased and reached its lowest point at 40 minutes with 0.198 K value and increased back at 50 minutes operation time. Hence, it can be deduced that 40 minutes is an optimum operation time for concentration of cucumber juice for high efficiency by using PFC process. At operation time of 20 to 30 minutes, the ice layer produced was brittle

and not solid. It contained more dendrites and has less crystallinity; it would trap the solute in the ice layer formed which indicates incomplete PFC process.



**Figure 5** Changes of TPC increment and effective partition constant K with operation time

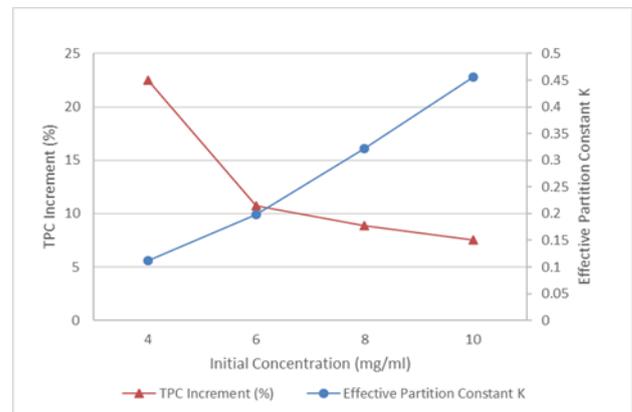
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Nevertheless, when the system operates too long, it might cause solute saturation in the remaining liquid. At this point, the trend of the solutes in the concentrate started to shift towards the ice crystal making higher amount of solutes trapped in the ice layer formed. It was proven that after the operation time has reached 40 minutes, effective partition constant K started to increase and TPC increment started to decrease and expected to continue onwards. This condition occurred due to the formation of the ice layer which get thicker and thicker until it touched the wall of the tank and nearly filled the entire space and volume of the solution tank, the fluid flow space was reduced and the possibility for the solutes to be trapped in the ice crystal formed was increased.

### 3.1.4. Effect of Initial Concentration

The study of the effects of initial concentration towards PFC system efficiency was done through the same procedure as before. The range of initial TPC concentration of cucumber juice solution was varied from 4 to 10 mg/ml while the other three operating parameters which are operation time, stirrer speed and coolant temperature were kept constant at 30

minutes, 350 rpm and -10 °C respectively. Figure 6 shows the TPC increment and effective partition constant K based on initial concentration of cucumber juice solution.



**Figure 6** Changes of TPC increment and effective partition constant K with initial concentration

From Figure 6, it can be seen that the lowest effective partition constant K and highest TPC increment were obtained at the lowest initial solution concentration which is 4 mg/ml. As the initial solution concentration increased, the K value also increased and TPC increment also decreased. This is due to the fact that solute content trapped in the ice crystal formed has increased. Specifically, for concentration process of fruit juice, the system requires more time than simulated solution due to the presence of foam that is formed at the initial process. The foam obstructed the heat transfer and might help in favouring mass transfer by creating larger turbulence at the solid-liquid interface. However, longer operation time created lower rate of ice formation. Both conditions of lower rate of ice formation and larger turbulence enable the solute diffusion from the interface of the ice-solution towards the concentrated juice. Hence, the amount of solutes trapped in the ice crystals is reduced [20].

As described by Miyawaki *et al.* [21], about the concentration polarization model, they explained that the solute concentration is highest at the ice-liquid interface (Miyawaki *et al.*, 1998). This is due to the solute accumulating at the interface after being rejected by ice crystal lattice. This phenomenon makes the inclusion of solute impossible during its growth process. Due to the increasing viscosity of the solution, the solutes have a high tendency to move from a higher to lower concentrated solution by mass transfer diffusion process. A phenomenon of constitutional supercooling would not occur if the concentration of solution is lower and this condition does not encourage formation of dendritic ice crystals which normally traps the solutes between its structures and creating a lower concentration of ice layer [22].

#### 4. CONCLUSION

The reliability and role of progressive freeze concentration method in concentrating cucumber juice by removing the water content and increase its total phenolic content (TPC) has proved that MPCC system can manage to give higher quality of the product. Effect of operating conditions revealed that lower coolant temperature led to a better performance efficiency of the system at -12 °C. However, the trend started to deviate when temperature lower than -14 °C was applied due to solute inclusion in the ice crystal formed. Meanwhile, a higher stirrer speed and longer operation time gave a lower value of K and TPC increment at 350 rpm and 40 minutes, respectively. Meanwhile, for high initial concentration, 10 mg/ml led to high value of K which is not favourable but at that value TPC increment was the best. This work has substantially proved that PFC has potential to be engaged in the industry for cucumber juice concentration, as well as for other fruit juices. Further research to optimise this process is needed to further strengthen the potential of this method.

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