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Preserving Flavours in Pomelo Juice using Progressive Freeze Concentration

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ABSTRACT

The purpose of this research is to determine the efficiency for the preservation of flavouring in pomelo juice by using Progressive Freeze Concentration (PFC). The flavouring component inside pomelo juice, which is 3-methylbutanal, underwent a concentration process and its existence before and after the process was determined. In this research, PFC was introduced due to its advantages of preservation. The research conducted concentration of pomelo juice at low temperatures using PFC Ice Crystalliser (PFCIC). The experiments were conducted with different coolant temperatures and circulation flowrates, with constant circulation time and the initial concentration at an optimum value. The products for this concentration process were the purge ice crystal and concentrated pomelo juice. The concentrated pomelo juice was then sent for analysis using Gas Chromatography-Mass Spectrometry (GC- MS) for its active compound composition. The findings demonstrate successful preservation of flavour in the pomelo juice using PFC.

1. Introduction

Pomelo is an Asian citrus fruit which has a similar appearance and texture as grapefruit. Pomelo owns a shape similar to a teardrop. The peel of pomelo is mostly green or yellow and it has quite thick and pale rind. Pomelo can grow to a large size which is almost similar to cantaloupe. The flavours of pomelo also have its own varieties but mostly are sweet and juicy [1].

The reason for pomelo to become a popular fruit among the consumer is the excellent nutritional content. For example, pomelo consists of various types of nutrients and also vitamins. Based on the statistics, the huge amount of Vitamin C can act as a powerful antioxidant for boosting our immune system which can help our body to damage from the harmful components [2]. Besides, pomelo also

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consists of several types of minerals such as potassium which can help the consumer to regulate their fluid balance and also the blood pressure [1].

The flavouring inside pomelo is very useful as it can be extracted for other uses such as flavouring agents. Thus, the business industry had introduced several methods in order to maximize the preservation of the flavouring. First example is to preserve the flavourings by changing its concentration such as dehydration. However, this is not a desired method as dehydration involves adding salt which may cause most of the original nutrients, flavours and texture of the raw fruits to be lost. Besides, there is also a heating method used for the preservation as well, this is also a less encouraged method as heating might destroy all the aroma, flavours and also nutrients inside the pomelo. Thus, in order to improve the efficiency of preservation, the flavouring of pomelo has to be separated out for preservation. This is because by this method, it can maximize the preservation of the flavourings and nutrients inside the pomelo.

The concentration methods available nowadays are gaining acceptance in several industries such as different kinds of food industries and also water treatment [3]. By concentrating, advantages can be brought to different terms for example such as manufacturing, packaging and also transportation [9]. All of the concentration methods are done by separation which is to separate out the desired components from the raw materials. For the several separation methods available based on current technologies to be used for enhancing the concentration method, there are evaporation, reverse osmosis (RO), and also freeze concentration (FC) [4]. These methods will be carried out based on different targeting concentrations.

Based on the situation, freeze concentration (FC) introduced as it is used in several cases of preservation in fruit juice. It is the process of concentration of liquid product by freezing the water content [3]. It is able to form pure crystals without solid bonds [4]. The uses of cold temperatures in this separation are very beneficial as it can reduce losses by volatility, chemical reactivity [5], and of corrosion [3]. There are currently 2 types of freeze concentration which is suspension freeze concentration (SFC) and also progressive freeze concentration (PFC). SFC is a process which will produce many small ice particles in the suspension of mother solution [6], while PFC will only form a single large ice crystal and grown on the cooling surface [7]. Based on the advantages of PFC, it should be introduced to the separation for concentration processes for this study. Thus, PFC will be used for the process of preserving flavouring inside the pomelo by using crystalliser.

The objectives of this research are to investigate the suitable ranges of coolant temperature to be used for the preservation of 3-methylbutanal in pomelo juice, as well as to study the effective ranges for circulation flowrate on preservation of 3- methylbutanal in pomelo juice via progressive freeze concentration.

2. Methodology

2.1 Materials

Pomelo juice at concentration at 7.3 %Brix was used as raw material. Fresh pomelo fruit was obtained from a fruit shop at Taman Universiti, Johore, Malaysia. The flesh was obtained, blended and filtered before used as the sample material for the research. Besides, coolant was also involved, distilled water was used in ice lining process, as well as 50% (v/v) ethylene glycol and 50% of distilled water was used at the coolant.

2.2 PFC Apparatus

Figure 1 shows the design of the progressive freeze concentration apparatus used, namely PFC Ice Crystalliser (PFCIC). The ice crystalliser was designed with materials contributing to high thermal conductivity, therefore it is very suitable to carry out concentration at low temperature. Besides, due to the design of PFCIC, the ice layer produced in each experiment after the concentration can be easily visualized.

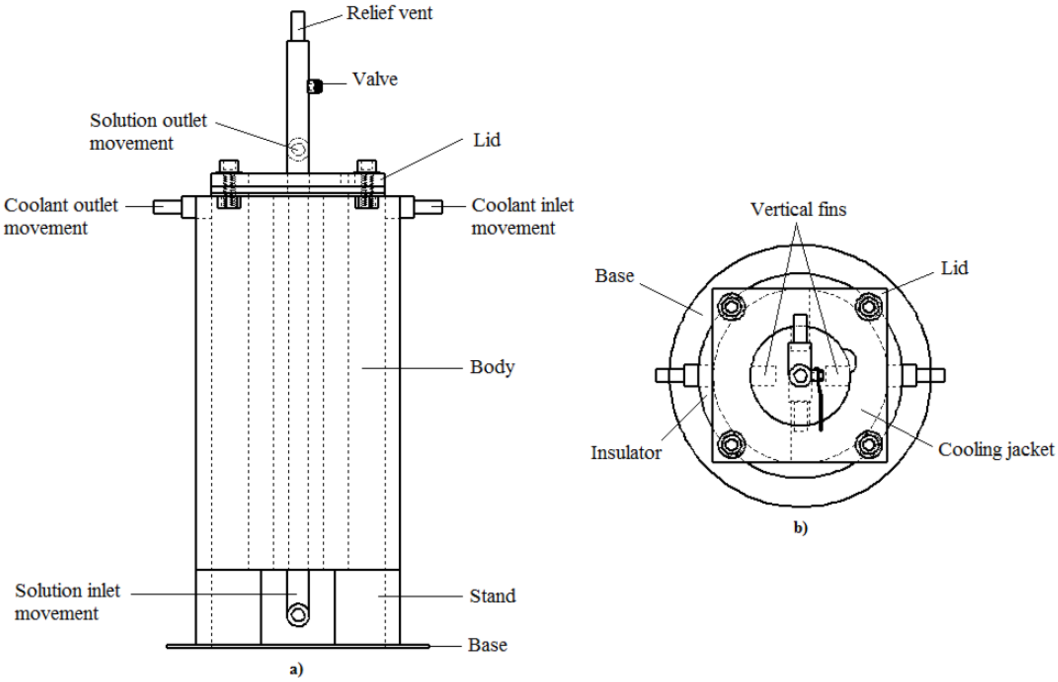

Fig. 1. Side (a) and Top (b) Views of PFCIC

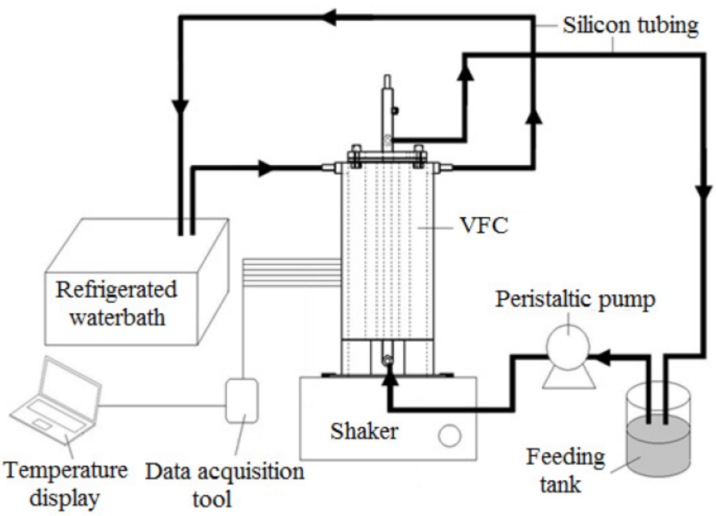

Fig. 2. Schematic diagram for experimental setup

2.3 Experimental Procedure

The experiment was divided into 2 parts, which is concentration of pomelo juice and analysis of flavouring in ice crystal. For concentration of pomelo juice, it which was prepared earlier was fed into PFCIC to undergo concentration. The temperature of pomelo juice was be maintained at almost similar to freezing point of water. Coolant was included in water bath and being switched on to reach temperature. After the coolant temperature has been achieved, coolant was allowed to flow outside of the crystalliser which is inside the jacket for 5 minutes. After that, distilled water was then fed into crystalliser using peristaltic pump to carry out ice-seeding process for 10 minutes. The parameters used was exactly the same for the upcoming experiment. After that, distilled water was then replaced by pomelo juice which is being fed into PFCIC from the feed container using the peristaltic pump. The feeding process was then stop after the PFCIC after the crystalliser is fully filled with the pomelo juice, by closing the valve at the feeding tank. The pomelo juice was allowed for circulating within PFCIC and silicone to wait for crystallisation to occur.

After the circulation process was done, the concentrated pomelo juice was then collected from both PFCIC and also silicone tubes. The volume of ice layer formed and also concentrated pomelo juice was measured and recorded before collecting the ice crystal sample in order to make the analysis purpose easier [8]. The experiment was conducted at different coolant temperature and circulation flowrate at shown in Table 1.

For analysis of flavouring in ice crystal, it was then identified by GC-MS. The concentrated pomelo juice was used for analysis of 3-methylbutanal content. For the GC-MS procedure, the concentrated juice was first being extracted and desorbed at 250°C for 3 minutes. Helium gas with high purity was then used as carrier gas for vial pressurization and circulated in (1:50) split mode. The flowrate for gas was maintained at 1mL/min. For oven temperature, it was initially kept at 40°C for 3 minutes, followed by increase of 2°C in 1 minute until the temperature reached 140°C, and another increase of 20°C in 1 minute to 250°C for holding around 6 minutes. For mass detection and source temperature, it was conducted with electronic impact mode at 70eV as well as temperature at 250°C. The mass spectrum was then scanned in the range of m/z 39 to 450 amu at 1 second intervals. Volatiles were then identified by comparing the mass spectra with data in National Institute of Standards and Technology (NIST08), and confirmed with retention index and aroma description [2]. For the results, it will then be displayed through a mass spectrometer. The amount of 3-methylbutanal present in the concentrated pomelo juice will then be compared with the pomelo juice prepared earlier in order to determine the efficiency for preservation of 3-methylbutanal.

Table 1
Value of varied and constant variables

Coolant Temperature (°C)	Circulation Flowrate (rpm)	Circulation Time (min)	Initial Concentration of Juice (%Brix)
-4	200	50	7.3
-6			
-8			
-10			
-12	250		
	300		
	350		
	400		

3. Results and Discussions

Effective partition constant (K) was related to quality of ice produced using Equation (1). In this equation, the initial volume and initial solute concentration of solution are represented by V0 and C0 respectively. While for the final volume and final solute concentration of the solution are represented by VL and CL respectively.

$$K = 1 - \frac{log\frac{C_O}{C_L}}{log\frac{V_L}{V_O}} \quad (1)$$

Solute recovery constant (K) was related to purity of ice produced using Equation (2). In this equation, the mass of solute in the concentrated liquid is represented by Ms,L. While for the mass of solute in the initial solution are represented by Ms,O. For Y value, it has a unit of g glucos/ g initial glucose [8].

$$Y = \frac{M_{s,L}}{M_{s,O}} \quad (2)$$

3.1 Effect of Coolant Temperature

Figure 3 shows the changes in K and Y value at different coolant temperature. Based on study, the rate of ice crystals growth in the freeze concentration process is mainly controlled by the coolant temperature [4,13]. Low temperature can enhance the heat transfer from crystalliser into the coolant as there is the presence of low surface temperature. Based on research as well, the value of K will decrease while the value of Y will increase between the temperatures of -4 to -10°C. However, the trends will be shifted from temperature of -12°C [8].

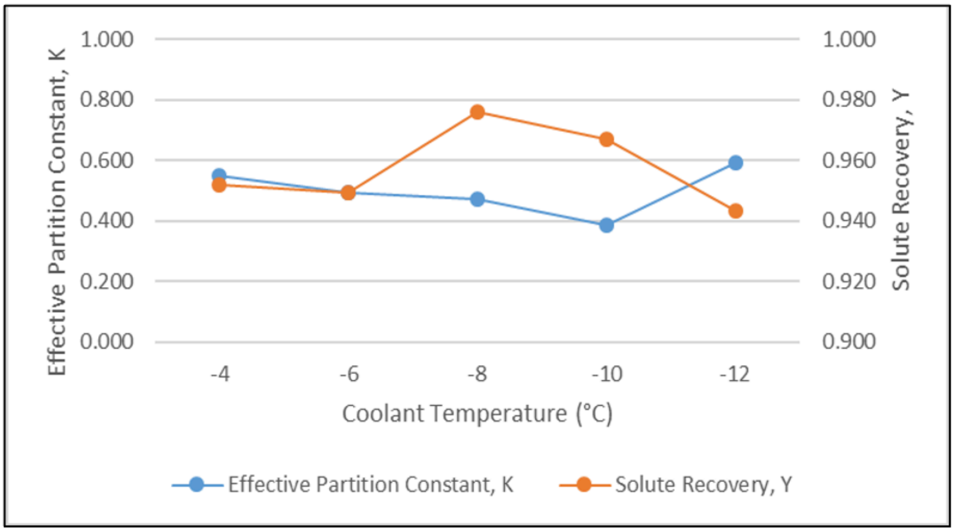

Fig. 3. Graph of changes in K and Y values at different coolant temperature

Based on the figure, it is clearly showed that K value decreased between temperature of -4 to -10°C, while Y value increased slightly between temperatures of -4 to -8°C. This shows that the highest efficiency based on coolant temperature selected was at -8°C, which was at low K value of 0.473 and high Y value of 0.976.

In general, the heat transfer within crystalliser wall and coolant used for the concentration process through ice crystallisation could be enhanced by low temperature of coolant [14, 10]. From the data, it clearly explained that K value decreased when the coolant temperature decreased from -4 to -10°C, which showed increase in the process efficiency. In this experiment, the process reached its highest efficiency at -8°C, where highest amount of solute was recovered.

However, it is inappropriate to further lower the coolant temperature below -10°C as solution inclusion might occurred in the ice crystal [8]. Due to high temperature difference between crystalliser wall and the entering solution, the ice growth rate will end up increased. As if the crystal grew faster than solute flowing out of crystalliser, solute might get trapped within the ice crystal formed. In result, the purity of ice will become lower caused by the contamination of solute. Thus, it will end up in decreasing low Y value due to lower process efficiency and less of solutes recovered [8]. In a nutshell, the coolant temperature which is used for PFC in pomelo juice is best to be controlled within the range of -4 to -8°C only.

3.2 Effect of Circulation Flowrate

Figure 4 shows the changes in K and Y value at different circulation flowrate. As solute inclusion is more likely to occur at low coolant temperature, the circulation flowrate needs to be controlled at high or adequate level in order to help remove the solutes from getting trapped within the ice crystal. The solution after the concentration process will also get higher concentration [4]. Based on research, the circulation flowrate should be controlled at a high value which is between 200 to 400 rpm, while these values will show the results of low K value as well as high Y [1].

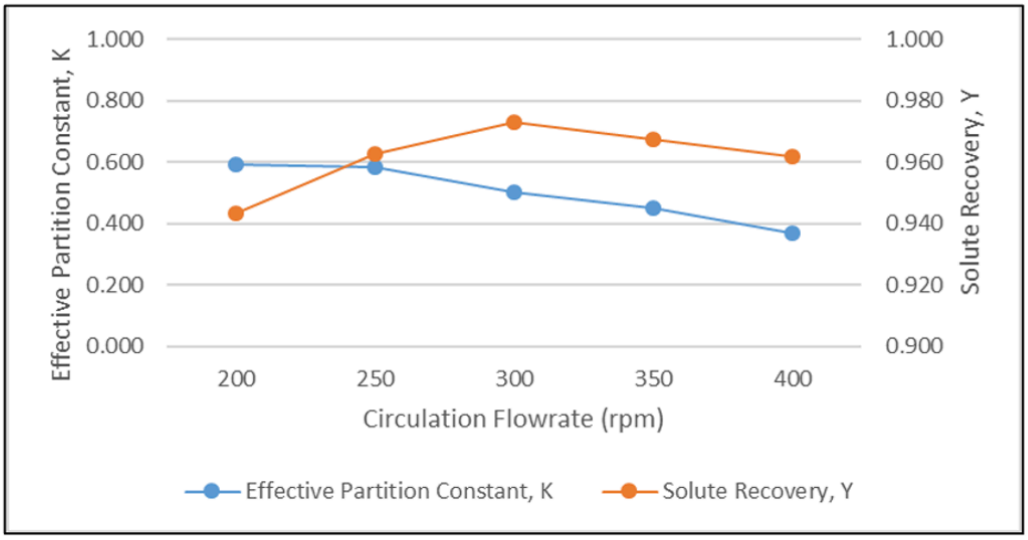

Fig. 4. Graph of changes in K and Y values at different circulation flowrate

Based on the figure, it is clearly showed that K value showed decreasing trend between flowrate of200 to 400 rpm, while Y value increased slightly between temperatures of 200 to 300 rpm. This shows that the highest efficiency based on circulate flowrate selected was at 300 rpm, which was at low K value of 0.503 and high Y value of 0.973.

In general, the transfer phenomena of heat and mass in PFC is also affected by fluid flow structure of solution [10]. Thus, a sufficiently high circulation flowrate is able to remove trapped solutes within ice crystal formed, in order to get a high concentration and purity product. However, if the circulation flowrate goes beyond the maximum limit, the heat transfer will get affected and hence causing the shear stress to dominate the solute movement within the crystalliser. This will end up causing the solute entrapment within the ice and causing the purity of ice to become low [8]. In a nutshell, the circulation flowrate which is used for PFC in pomelo juice is best to be controlled within the range of 200 to 300 rpm only.

3.3 Retention Time of 3-Methylbutanal

After GC-MS analysis, 3-methylbutanal was found within the chromatograms, which the sample used was the concentrated pomelo juice after the PFC process. 3-methylbutanal was also found with a peak within the chromatograms. According to NIST Retention Index Library, the retention time of 3- methylbutanal will be around 4 minutes. Table 2 shows the retention time of concentrated pomelo juice for different coolant temperature and circulation flowrate.

Table 2
Retention time of pomelo juice for different coolant temperature and circulation flowrate

Concentration Parameter	Retention Time (minutes)
Control: Initial Juice	4.073
(-4 °C, 200 rpm)	4.450
(-6 °C, 200 rpm)	4.763
(-8 °C, 200 rpm)	4.348
(-10 °C, 200 rpm)	4.265
(-12 °C, 200 rpm)	4.221
(-12 °C, 250 rpm)	4.742
(-12 °C, 300 rpm)	4.228
(-12 °C, 350 rpm)	4.149
(-12 °C, 400 rpm)	4.499

Figure 5 and 6 show the GCMS result for initial pomelo juice and concentrated pomelo juice at (-12°C, 400 rpm). Based on figures, it is clearly showed that 3-methylbutanal exist as 2nd peak in all the chromatograms. This shows that the flavouring component was success to be preserved in the pomelo juice after the concentration process. Thus, progressive freeze concentration is a successful method in order to preserve the flavouring component, 3-methylbutanal within the pomelo juice.

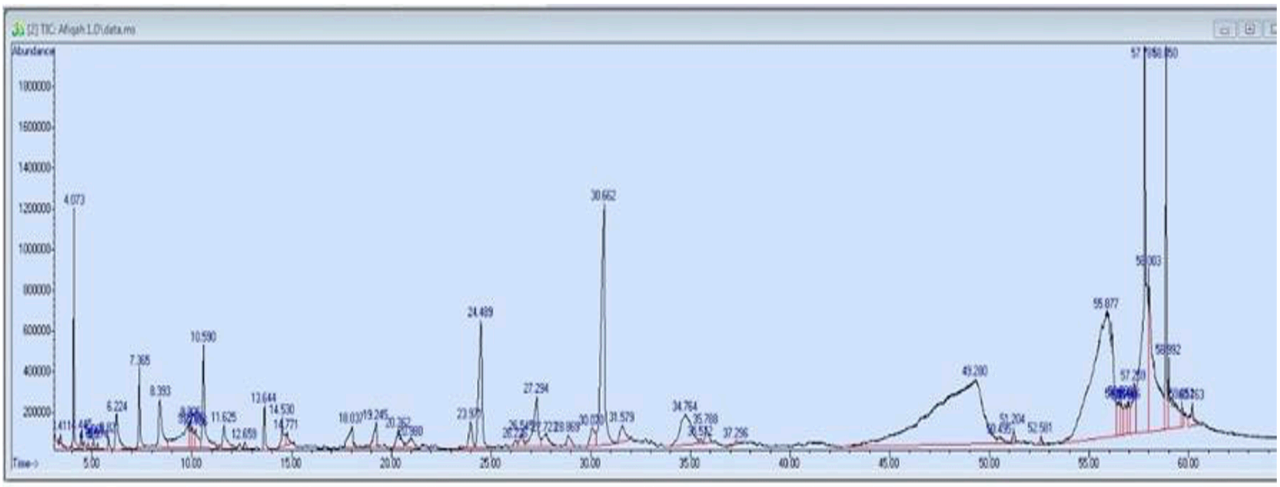

Fig. 5. GCMS result for initial pomelo juice

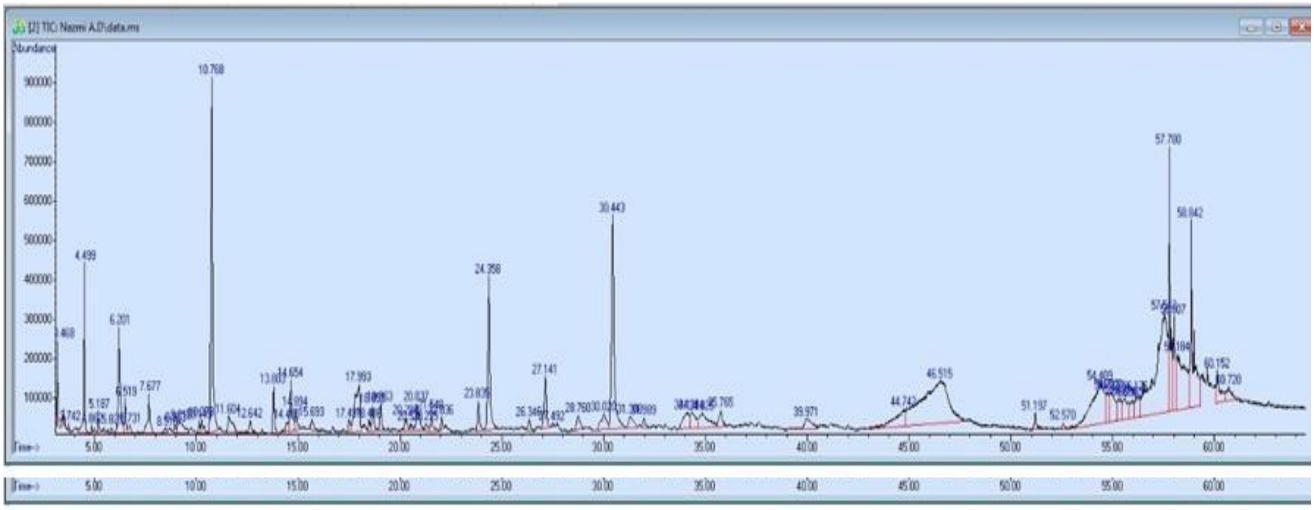

Fig. 6. GCMS result for concentrated pomelo juice at (- 12 °C, 400 rpm)

3.4 Composition within 3-Methylbutanal

Besides, different coolant temperature and circulation flowrate results in different composition of 3-methylbutanal being retained in pomelo juice after PFC. Table 3 shows the composition of 3-methylbutanal after PFC.

Table 3
Composition of 3-methylbutanal after PFC

Concentration Parameter	Composition
Control: Initial Juice	0.0125
(-4 °C, 200 rpm)	0.0129
(-6 °C, 200 rpm)	0.0094
(-8 °C, 200 rpm)	0.0223
(-10 °C, 200 rpm)	0.0220
(-12 °C, 200 rpm)	0.0086
(-12 °C, 250 rpm)	0.0076
(-12 °C, 300 rpm)	0.0240
(-12 °C, 350 rpm)	0.0233
(-12 °C, 400 rpm)	0.0152

Figure 7 and 8 show the composition of 3- methylbutanal at different coolant temperature and circulation flowrate. Based on figures, it is clearly showed that composition of 3-methylbutanal was found peak at coolant temperature of -8 °C and circulation flowrate of 200 rpm. This shows that the flavouring component was best preserved at these concentration parameters. Thus, PFC of pomelo juice should be conducted at these parameters in order to maximize the preservation.

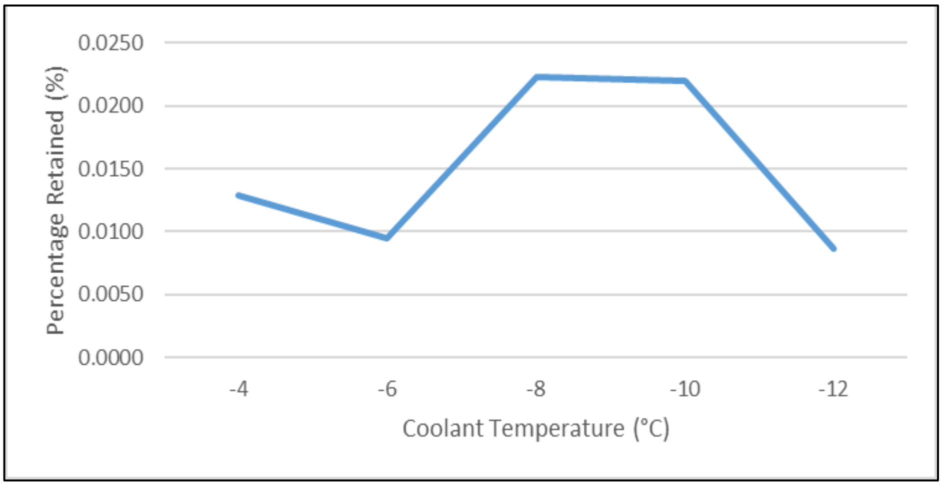

Fig. 7. Composition of 3-methylbutanal at different coolant temperature

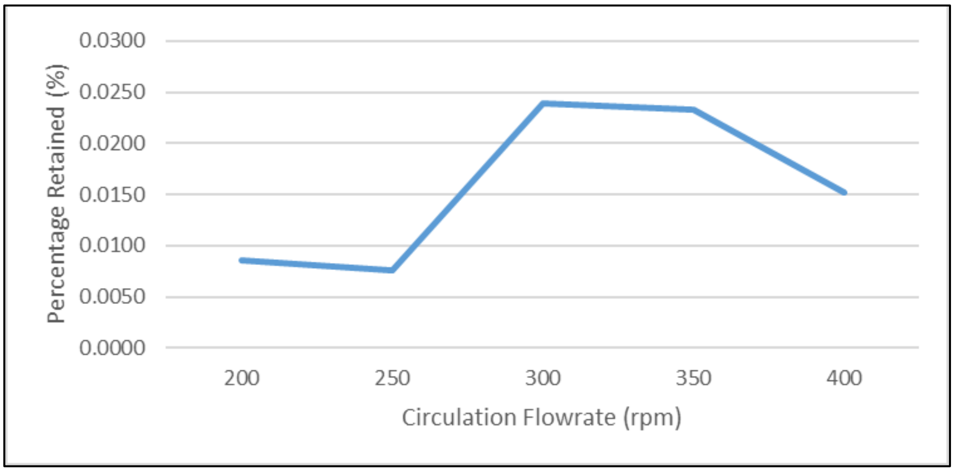

Fig. 8. Composition of 3-methylbutanal at different circulation flowrate

4. Conclusions

In PFC, it is important to observe the relationship between both coolant temperature and circulation flowrate towards the impurity of concentration of ice crystals. Although all of the pomelo juice had increment after the concentration process, but it is obvious that most of the solutes were trapped in the ice crystals after PFC was done as well. To further understand this relationship, the effect of changing experimental parameters were studied towards the efficiency of PFC process. In this finding, PFCIC was used as it owned capability of concentrating pomelo juice through PFC method. Besides, due to the fin design of PFCIC, it was easier to observe the ice formation within the crystalliser. For the performance of PFC, it has been evaluated through effective partition constant (K) and solute recovery (Y). Based on the performance as shown in findings, PFCIC is feasible to conduct the concentration process for pomelo juice. From the results, K and Y were found dependent on both coolant temperature and circulation flowrate. Based on the findings as well, the suitable range of coolant temperature to be used for preservation of 3- methylbutanal in pomelo juice is between -4 to -8 °C. Low and adequate coolant temperature will give high efficiency in PFC process, but further lowering the parameter will results in undesired value of K and Y. For the effective circulation flowrate on preservation of 3-methylbutanal in pomelo juice via PFC, it was best to keep the parameter between 200 to 300 rpm only. In general knowledge, high and adequate circulation flowrate is able to remove the trapped solutes within the ice crystals formed, leaving a highly purified ice and highly concentrated pomelo juice as product. However, further increasing the flowrate will result in the solutes getting trapped in the ice crystal again, hence lowering the purity for both products. Last but not least, preserving the flavouring components of 3-methylbutanal in pomelo juice using PFC was successful.

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