**RESEARCH ARTICLE**

# Progressive Freeze Concentration Performance Prediction based on Polynomial Curve Model for Star Fruit Juice Concentration

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Abstract Progressive freeze concentration (PFC) is a simpler freeze concentration process of removing water content in fruit juice through ice crystal formation in order to concentrate a solution. Vertical finned crystallizer (VFC) was used in the PFC system as the ice crystallizer in this study. A mathematical model is highly needed to be developed so that theories can be validated and to understand the system developed better with minimal risk and cost. Mathematical modelling is also essential to analyze the performance of the system. In this work, the use of mathematical model was explored based on a polynomial regression in analysing and predicting the performance of PFC system. The polynomials curve fitting were first performed to develop the models followed by the simulations to predict the target variables of effective partition constant ($K$ value) and solute recovery ($Y$ value). The relationship of operating parameters including coolant temperature and operation time on the PFC performance values were also discovered via the correlated polynomial regression models. Based on simulations result, the highest efficiencies of PFC process were achieved at approximately - coolant temperature of 10°C and operation time of 55 minutes. To validate the models' accuracy, the statistical assessment parameters of R-squared and Absolute Average Relative Deviation (AARD) were determined. The findings of this study conferred satisfactory results of the prediction performance of polynomial regression model, in which the least analysis error of AARD (i.e., below 10%) and the highest R-squared (i.e., above 0.97) were successfully achieved. It is concluded that polynomials-based predictive models are promising alternatives to replace time-consuming and expensive experimental evaluation of PFC process for fruit juices.

**Keywords**: Progressive freeze concentration, star fruit juice, effective partition constant, solute recovery, mathematical model.

## Introduction

Star fruit or its scientific name called *Averrhoa carambola* L., is a species that belongs to the *Oxalidaceae* family and available in almost all parts of the world [1]. Star fruit is named from its shape, like a star when the fruit is sliced diagonally with a five-pointed cross-section. The fruit is oblong in shape and has a waxy thin skin with a yellowish green colour. It typically ranges in size from 6.35 to 15 cm long and up to 9 cm wide. The fruit's skin is edible with a moderate sour taste in the flesh that makes it an ideal choice to be consumed fresh and processed for products in industries like foods and beverages. Basically, star fruit is rich in good nutrients as it is low in calories but loaded with vitamin C, fiber as well as polyphenolic

antioxidants like quercetin, epicatechin, and beta carotene [2]. Antioxidants have long been associated with reducing the risk of many diseases, including heart problems and certain cancers. For this reason, star fruit needs to be concentrated because it contains 90% of water. In fact, concentrating the star fruit juices can reduce the water activity (aw), which in turn prevents the growth of the microorganism and improves the shelf life of juice products [3].

Processes that can be used to concentrate star fruit juice include freeze concentration, evaporation, and reverse osmosis [4]. Among these processes, freeze concentration is the best to produce concentrated fruit juice with desirable volatile components retained as compared to the other two processes [5]. This is because freeze concentration is a process of removing water from fruit juices without heating it or changing the juice flavour [6]. Aside from fruit juices industry, the process is also applied in wastewater treatment [7, 8] and seawater desalination [9, 10] because this process has lower energy consumption compared to other processes. The concept of freeze concentration is for the water to form ice crystal at subzero temperature with the highest purity without any other components being trapped in its crystal lattice hence leaving all valuable solutes in the concentrated liquid [11].

In freeze concentration, the process can be categorized into two processes, which are suspension freeze concentration (SFC) and progressive freeze concentration (PFC) [12, 13]. PFC is the simpler way to tackle the difficulties encountered in conventional system, which gives easier separation of ice crystal from the mother liquor. A single large ice crystal is formed layer by layer on a cooled surface in PFC for heterogenous ice nucleation, thus eliminating the many small ice seeds formed in SFC [14]. Recent PFC designs in this lab include Vertical Finned Crystallizer (VFC) and Spiral Finned Crystallizer (SFC) [15, 16]. Both types have been designed in a cylindrical shape and developed to enhance the PFC efficiency by focusing on increasing the surface area that affects the heat transfer rate [17]. It was the crystallizer fin configuration that made the difference in order to improve the system performance, each in their own way.  In brief, VFC has vertical fins, while SFC has spiral fins that added to the crystallizer with the aim to raise the contact surface areas for better flow circulation of the solution from the inlet to the outlet inside the vessel.

Notwithstanding the crystallizer features, the most crucial part to evaluate in this separation system is the effective partition constant (*K*) that acts as an effectiveness index of PFC system between the ice and liquid phases [18] in concentration of any solution. Besides, the measurement of components amount recovered in the concentrated liquid during PFC process is also important in this research work. A mathematical model is essential to be created based on aforesaid measurements so that theories can be validated in order to better understand the behaviour of the system for concentration of each particular solution and its corresponding separation process with minimal risk and cost.  Thus, a simulation that is the implementation of the formulated model with the aid of a computer program offers a more flexible and convenient approach when analyzing the experimental data and system performance [19]. This is highly needed in the industry for testing of many PFC applications as it can often be conducted faster than real-time experiments. Besides, the experimental method is not always practical for scaling up and design purposes [15, 16, 17].

In this study, mathematical modelling allowed us to estimate and predict the behavior of the PFC process of star fruit juice using VFC under different operating conditions, which has not yet been exploited. Polynomial models are an effective and flexible curve fitting technique. In addition, the major benefit of polynomial models includes providing the great approximation of the relationship between the target and independent variables with error minimization. MATLAB R2016a was used as simulation software to execute the coding that can predict the effective partition constant and solute recovery values at different operating parameters, i.e., coolant temperature and operation time. The research outcomes of this paper could provide new prospects for future PFC model development.

## Materials and methods

### *Material Preparation*
Fresh star fruit juice was purchased from a local market in Taman Universiti, Skudai, Johor, Malaysia. Good quality star fruits were selected and subjected to some preparation activities like washing, slicing,

and extracting the juice using fruit juicer. Brix scale or refractive index measurement was applied using a Brix meter to determine the total sugar amount in the juice solution. Then, the juice was stored in a fridge at a temperature close to freezing point of pure water (i.e., 0°C).

## Chemical and Analytical Procedures

Ethylene glycol-water solution (50% v/v) was used as a coolant liquid. Folin-Ciocalteu reagent (10%, v/v) and sodium carbonate ($Na_2CO_3$) were utilized for total phenolic content (TPC) measurement of the sample [20]. Those chemicals were purchased from Merck (Germany). Gallic acid (Sigma Aldrich, USA) was used for standard calibration curve preparation in which the TPC amount is expressed as mg gallic acid equivalents per 1 gram of sample (i.e., mg GAE/ g). For analysis, 1 ml of star fruit juice sample was mixed with 5 ml Folin-Ciocalteu reagent, and left for 10 minutes at room temperature. Then, 4 ml of $Na_2CO_3$ was added and kept in the dark for 45 min at 37°C. After 30 minutes, the absorbance of sample was determined at wavelength 765 nm using UV-Vis Spectrophotometry (Mini UV 1240, Shimadzu, Japan).

## Progressive Freeze Concentration using VFC

A PFC system using VFC was employed to concentrate star fruit juice as shown in Figure 1. The experimental setup was according to Amran et al. [21], which comprised of VFC, feeding tank, refrigerated water bath, peristaltic pump, temperature display and data acquisition tool. Made out of stainless steel with cylindrical diameter size of 8 cm, the VFC design volume is approximately 1.15 L.
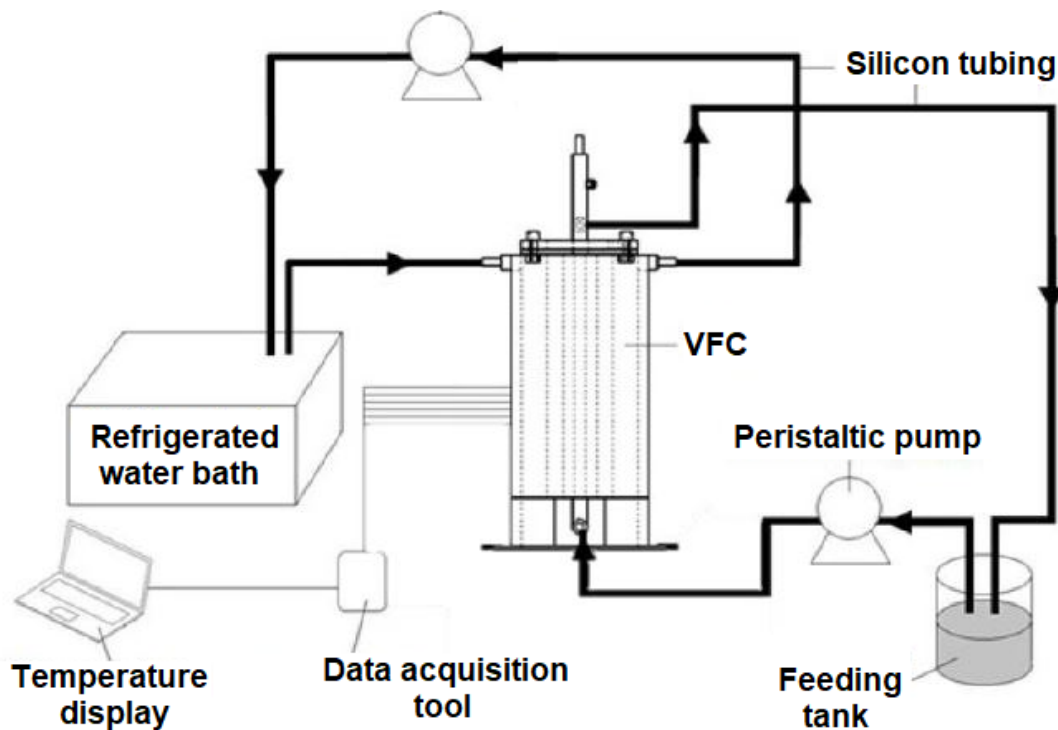


**Figure 1.** The schematic diagram of PFC using VFC

This crystallizer can basically be divided into five parts, i.e., body, stand, fins, lid and cooling jacket (refer to Figure 2). Four fins with vertical rectangular shapes were designed onto the inner wall of crystallizer in which each fin has a 30 cm height, 2 cm length and 1.5 cm width. The circulation flow rate (i.e., 300 rpm or equivalent to 3400 ml/min) of the feed solution (i.e., star fruit juice) from the feeding tank to the VFC was controlled by a peristaltic pump. Meanwhile, the water bath was used to control the coolant temperature ranged from -14 to 16 ± 0.05 °C. Six thermocouples (TC-08) were equipped to measure the juice solution temperature together with a data logger (Picolog) for computer online monitoring throughout the experimental process.
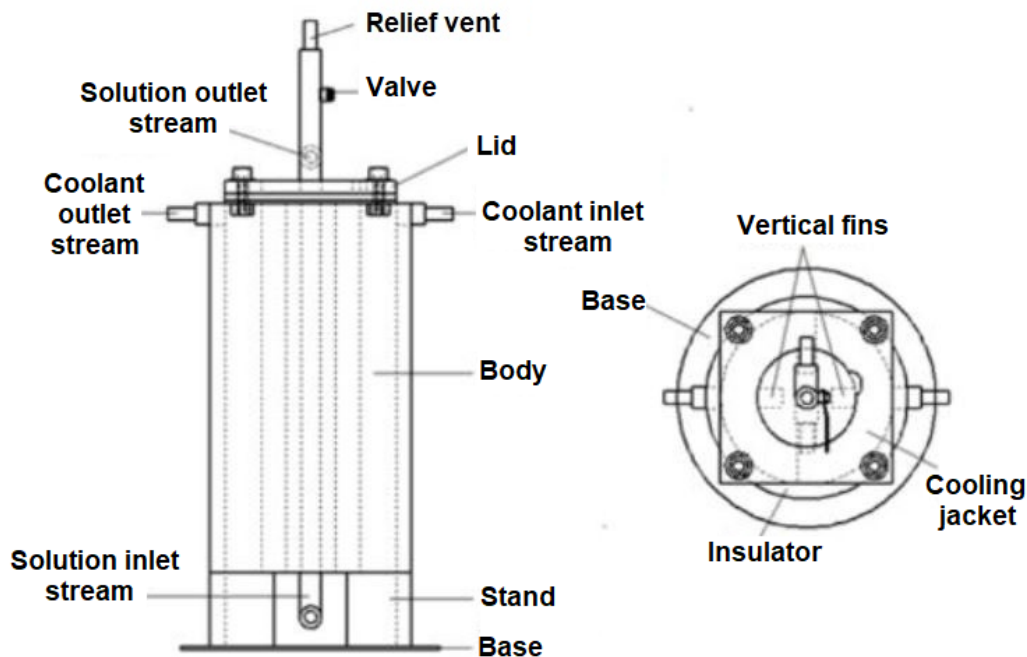
**Figure 2.** The diagram of VFC from the side view and top view

## Experimental Procedure

Firstly, the coolant from the refrigerated water bath was set at the desired temperature and then circulated within the cooling jacket. After 5 minutes, the juice at 0°C and 7.1 °Brix from feeding tank was transferred into the VFC until it filled up the entire tubing. Then, 1700 ml of juice was left in circulation for formation of ice crystal. The circulation process was stopped once achieving the desired operation time. The concentrated juice was then drained out and collected from the crystallizer as well as in the silicone tubing for analysis purpose. Eventually, the ice crystal that formed inside the VFC was detached from the cooling surface by letting it thaw using running tap water. The experimental work was performed in duplicate for reproducible result. In brief, in order to evaluate the PFC performance of star fruit juice, this study was accomplished by manipulating the effect of coolant temperature and operation time on the dependent variables, namely effective partition constant ($K$) and solute recovery ($Y$).

## Determination of Effective Partition Constant, K

$K$ value represents the separation efficiency of PFC process at the ice-liquid interface. In this study, it was calculated by measuring the Brix scale, which was determined by the sugar amount in the juice solution. As expressed in Equation 1, $K$ value was calculated experimentally based on ratio between solute concentration and concentrated liquid [18, 22]. The $K$ value ranges are from 0 to 1, where the lower value indicates a more efficient PFC process [23].

$$K = 1 - \frac{log\frac{C_O}{C_L}}{log\frac{V_L}{V_o}}$$

(1)

where $C_O$ and $C_L$ are the initial and final concentration of star fruit juice in °Brix, respectively. Meanwhile, $V_O$ and $V_L$ are the initial and final volume of star fruit juice in ml, respectively.

## Determination of Solute Recovery, Y

In order to identify the recovered solute in the concentrated star fruit juice, the solute recovery ($Y$) was

calculated on a TPC basis that has a unit of g TPC / g initial TPC. Thus, *Y* is interpreted as the ratio of the final TPC concentration of the solution over the initial TPC concentration of the solution as shown in Equation 2 [24].

$$Y = \frac{C_L M_L}{C_o M_o} \tag{2}$$

where $C_o$ and $C_L$ are the initial and final concentration of star fruit juice in mg GAE/ g, respectively. Meanwhile, $M_o$ and $M_L$ is the mass (g) of TPC in the initial solution and concentrated liquid (after PFC), respectively.

### *Polynomial Regression Model for PFC*

Basically, polynomial regression fits a wide range of curvature and can correlate between two or more variables from the data set that is not too complex. Since the PFC performance was hypothesized as curvilinear relationship for values of *K* and *Y* at different coolant temperatures and operation times, thus the polynomial function was applied that best suits the observed data. The general form of polynomials is expressed in Equation 3 [25];

$$Y = \beta_0 + \beta_1 x_i + \beta_2 x_i^2 + \cdots + \beta_k x^k + \varepsilon, \quad \text{for } i = 1,2,3,\ldots,n \tag{3}$$

where *Y* is the target variable, $\beta_0$ is the regression constant, $\beta_1$, $\beta_2$, $\beta_k$ are the coefficients of regression, *x* is experimental factor that influence the PFC process, *k* is the degree of the polynomial with $1 \leq k \leq 9$, and ε is the model error.

Firstly, the different order of polynomial equations for PFC performance were analysed. The coefficient of determination, denoted $R^2$ was then interpreted using Microsoft Excel spreadsheet system to determine how well the regression model fits the data set. R-squared is a statistical measure of the proportion of total variation outcomes elucidated by the model. Eventually, the polynomial curve fitting with the highest R-squared values were selected to assess the PFC performance of the star fruit juice.

### *Simulation using MATLAB R2016a*

The proposed regression models were solved by simulations using a MATLAB R2016a programming to predict the *K* and *Y* values at different operating parameters. This computer program greatly simplified the procedure of calculating the prediction values with a minimal data requirement and very straightforward. Figure 3 shows the algorithm and flowchart of MATLAB program by codes execution at selected parameter for each target variables.

### *Assessment of the Model's Accuracy*

Aside from the R-squared, the statistical assessment parameter of Absolute Average Relative Deviation (AARD) was used in order to evaluate the validity of the obtained models. The model's accuracy can be determined by this error analysis, where the data is said to be highly consistent when AARD is lower or around 5%, and considered as consistent when AARD is below 10% [26]. Equation 4 displays the standard equation for calculating the percentage of AARD.

$$AARD\ (\%) = \frac{1}{N} \sum_i^N \left| \frac{y_{exp} - y_{mod}}{y_{exp}} \right| \times 100\% \tag{4}$$

where *N* stands for number of data, $y_{exp}$ is the data from experiment and $y_{mod}$ is the data from modelling. Lastly, the experimental and predicted values were plotted in order to visualize the results of the model.
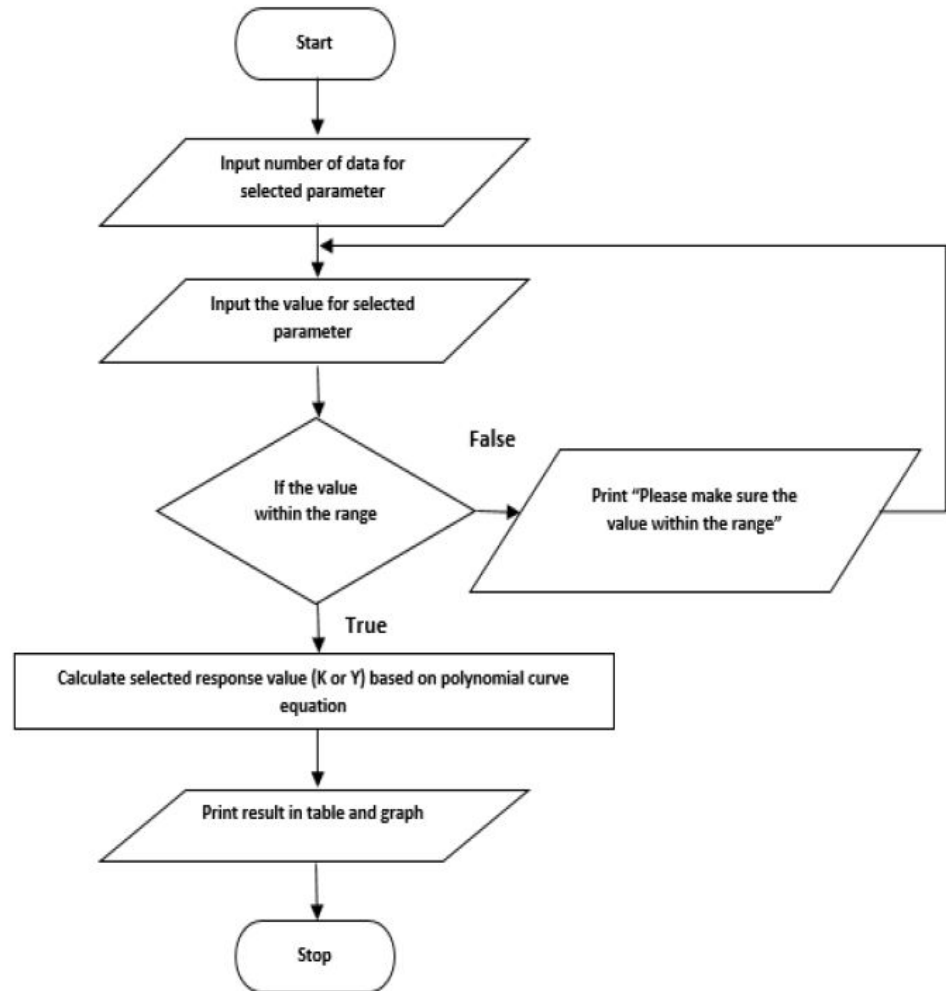
**Figure 3.** The algorithm and flowchart of MATLAB programming

## Results and discussion

### PFC Models Fitting

Regression model is commonly used in most data processing of manufacturing applications because it can produce high accuracy even with only a few sampling data. In order to find the appropriate regression expressions, the PFC performance of star fruit juice with different polynomials were compared. Figure 4 and 5 show the scatter plots of *K* and *Y* in PFC of star fruit juice at different operating conditions, which are mathematically represented by different polynomial degree. In this study, the trend line of first order linear was a bad fit ($R^2 < 0.3$). Thus, the least-square technique was extended for a better fit to the data of a higher-order polynomial. This procedure was used to minimize the differences between the data points and the curve plotted.
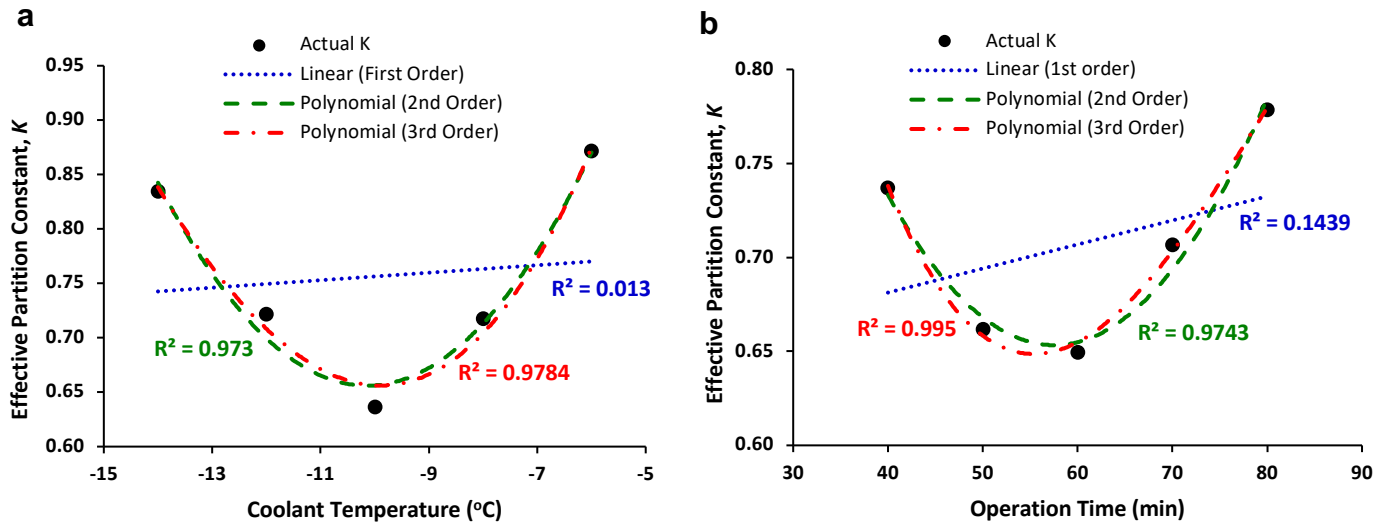
**a**



**b**

**Figure 4.** Representations of curve fitting of *K* at different operating parameters; (a) coolant temperature (b) operation time
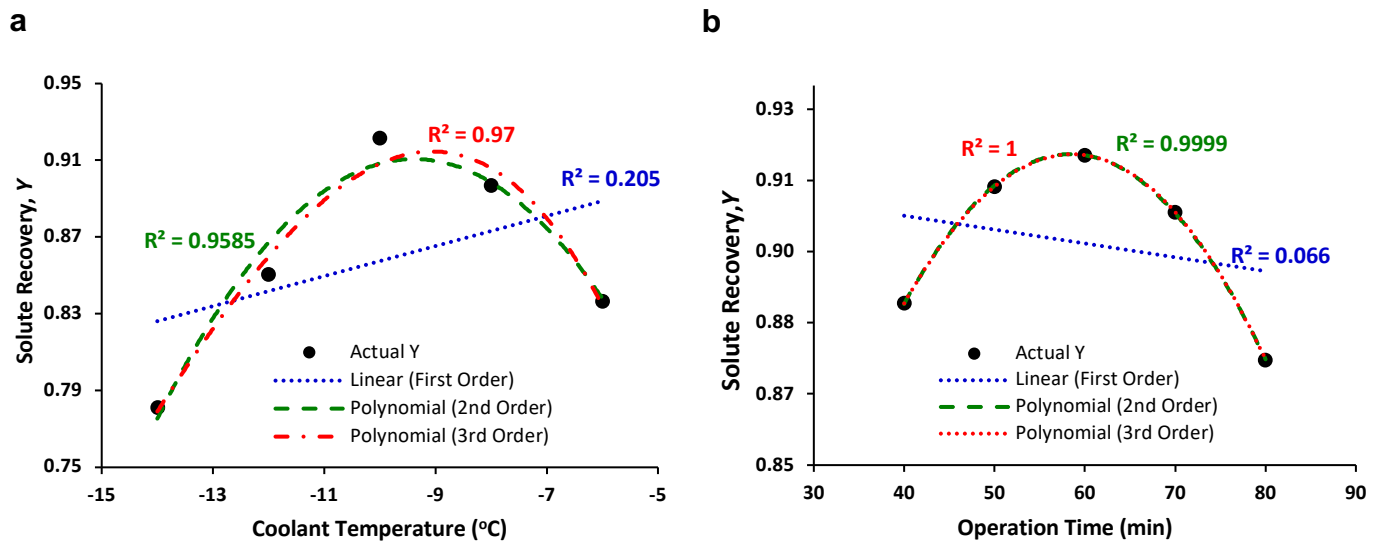
**a**



**b**

**Figure 5.** Representations of curve fitting of *Y* at different operating parameters; (a) coolant temperature (b) operation time

From the resulting graphs, it can be clearly seen that the polynomial regressions with the higher degree improve the models fitting. In brief, the 3rd order polynomial models have the highest R-squared values (i.e., $R^2 > 0.95$) for all PFC operating conditions. For *K* curves fitting, R-squared is equal to 0.978 and 0.995 against different coolant temperature and operation time, respectively. In other words, the results show that 97.84% and 99.50% of the original uncertainty have been explained by the models of *K* curves. Meanwhile, R-squared of 0.970 and 1.00 were achieved in *Y* curves fitted at varied coolant temperature and operation time, respectively.

Eventually, the best fitting models of PFC performance could be expressed by the developed curve equations as in Equation 5, 6, 7 and 8. Table 1 tabulates the data obtained from the experimental works (i.e., actual) and the developed models (i.e., predicted). It was obviously showed that the actual and predicted *K* values ranged from 0.6362 to 0.8713 and from 0.6246 to 0.8801, respectively. Meanwhile,

the actual and predicted $Y$ values varied from 0.7814 to 0.9218 and from 0.8070 to 0.9174, respectively. In brief, the lowest $K$ and highest $Y$ values could lead to greater efficiency of the PFC process.

Target variable of $K$ at different coolant temperature:
$$K = 0.0005x^3 + 0.0265x^2 + 0.3869x + 2.3436 \tag{5}$$

Target variable of $K$ at different operation time:
$$K = -0.000004x^3 + 0.001x^2 - 0.0722x + 2.3041 \tag{6}$$

Target variable of $Y$ at different coolant temperature:
$$Y = -0.0004x^3 - 0.018x^2 - 0.23x + 0.0174 \tag{7}$$

Target variable of $Y$ at different operation time:
$$Y = -0.00000009x^3 - 0.00008x^2 + 0.0099x + 0.6165 \tag{8}$$

**Table 1.** Actual and predicted values of $K$ and $Y$ obtained from PFC of star fruit juice

| Operation Time: 40 min | | | | | |
|---|---|---|---|---|---|
| **Coolant Temperature (ºC)** | **-6** | **-8** | **-10** | **-12** | **-14** |
| Actual $K$ | 0.8713 | 0.7177 | 0.6362 | 0.7218 | 0.8348 |
| Predicted $K$ | 0.8682 | 0.6884 | 0.6246 | 0.6528 | 0.7490 |
| Actual $Y$ | 0.8366 | 0.8969 | 0.9218 | 0.8507 | 0.7814 |
| Predicted $Y$ | 0.8358 | 0.9102 | 0.9174 | 0.8766 | 0.8070 |

| Coolant Temperature: -8ºC | | | | | |
|---|---|---|---|---|---|
| **Operation Time (min)** | **40** | **50** | **60** | **70** | **80** |
| Actual $K$ | 0.7372 | 0.6620 | 0.6495 | 0.7070 | 0.7787 |
| Predicted $K$ | 0.7601 | 0.6941 | 0.7081 | 0.7781 | 0.8801 |
| Actual $Y$ | 0.8841 | 0.9087 | 0.9153 | 0.9033 | 0.8722 |
| Predicted $Y$ | 0.8787 | 0.9003 | 0.9031 | 0.8866 | 0.8504 |

### Modelling Correlation by Simulation

Based on the obtained models of the greatest fit, the predicted $K$ and $Y$ were defined using MATLAB simulation. The programming system executed the coding and simultaneously produced the prediction values at different coolant temperature and operation time. Figure 6 and 7 represented the results generated from the simulations. From the graph (Figure 6), the lowest $K$ values were found, which are 0.6246 and 0.6941 at -10°C coolant temperature and 50 minutes operation time, respectively. Meanwhile, from the graph (Figure 7), the highest $Y$ values were obtained, which are 0.9174 and 0.9031 at -10°C coolant temperature and 60 minutes operation time, respectively.

This study revealed the effects of coolant temperature and operation time on the PFC performance of star fruit juice, specifically on $K$ and $Y$ values. Theoretically, the lower the effective partition constant ($K$ value), the higher the separation effectiveness [27, 15]. From Figure 6, the lowest separation efficiencies were observed at the highest $K$ values, i.e., at -6ºC coolant temperature (Figure 6a) and 80 minutes operation time (Figure 6b). Meanwhile, the highest efficiencies of the PFC process were achieved at the lowest $K$ values, i.e., at -10.5ºC coolant temperature and 53 minutes operation time. However, it can be seen that the $K$ values increased when the coolant temperature applied was higher and lower than the region of -11 to -9ºC. This is an agreement with the theory reported by Zhang and Liu [28] where the ice growth rate in PFC is greatly controlled by coolant temperature. Too low coolant temperature led to low purity of ice crystal caused by the supercooling effect occurred. At this condition, higher solute inclusion could be trapped in the ice crystal [16]. Consequently, ice crystals formation was increased and created in larger amounts. The similar pattern of $K$ values was also observed when varying the operation time of PFC (Figure 6b). The $K$ values increased when the operation time employed was higher and lower than the range of 45 to 60 minutes, which was undesirable in PFC due to lowering the efficiency of the system.
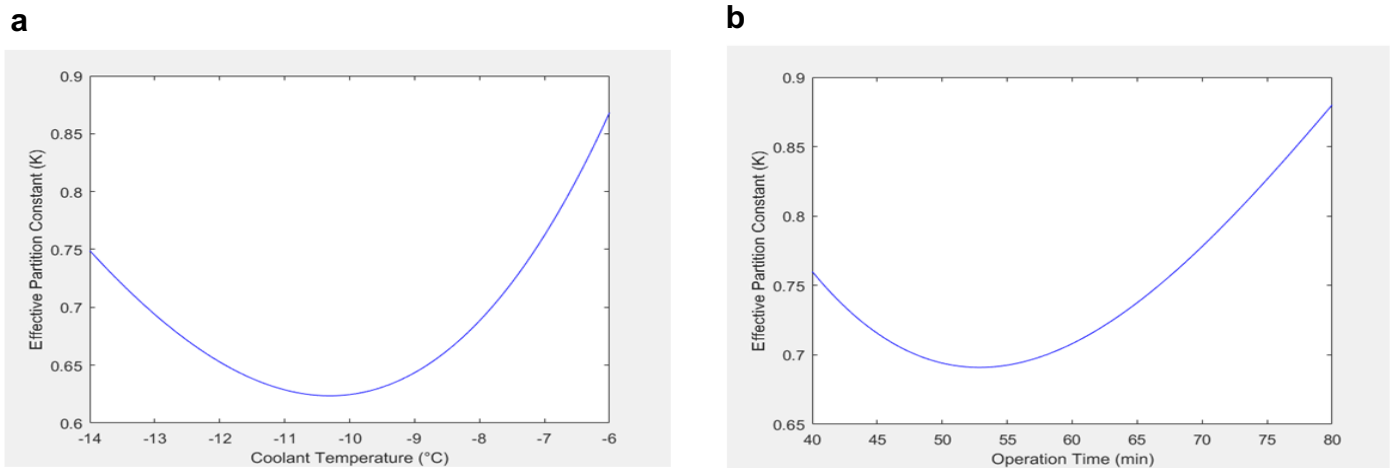
**a**



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**Figure 6.** Graph of *K* at different a) coolant temperature and b) operation time
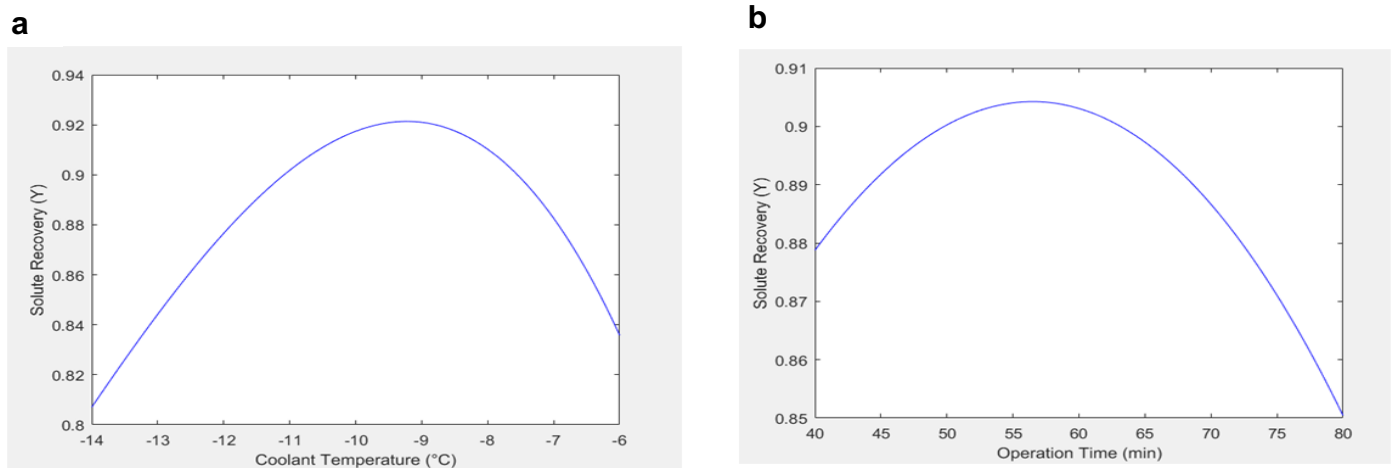
**a**



**b**



**Figure 7.** Graph of *Y* at different a) coolant temperature and b) operation time

From Figure 7, it can be seen that the highest solute recovery (*Y* value) was obtained at -9.2°C coolant temperature (Figure 7a) and 57 minutes operation time (Figure 7b). The *Y* value in the star fruit juice concentrates represents the ice crystal purity in PFC. Thus, high retention of TPC in the concentration solution is the main target to describe the PFC performance. The *Y* values showed an increase when the coolant temperature was set approximately between -11 to -8°C, and the operation time was conducted in range 50 to 65 minutes. Less and more than these ranges contributed to low *Y* values, which means lower effectiveness of the separation process. Basically, the ice crystal formation is increased when PFC operated with longer operation time, which subsequently raised the solute recovery. However, too long operation time might shift to the decrement trend of *Y* value due to solute saturation in the concentrates [21]. At these circumstances, continuously increasing the ice surface thickness might reduce the ice growth rate because of the solutes contamination in the ice crystal. This situation is also supported by other studies that discovered that the higher solid fraction could contribute to lower ice purity [29].

### Model Error Analysis

The quantitative error analysis of the polynomial regression models for PFC of star fruit juice were compared in terms of calculated percentage of AARD. Figure 8 depicts the visual comparison between

AARD (%) for different approaches in estimating the PFC performance. It is evident that the predicted *Y* values fall in the range of 1 to 5% of AARD meaning that the data is highly accurate, while predicted *K* values fall in the range of 5 to 10 % of AARD which implies that the data is accurate. The results were comparable with R-squared obtained ($R^2 > 0.97$) where the values are close to 1, revealing the models match the data more accurately. This concluded the validity, reliability and accuracy of the polynomial models to predict the solute recovery and effective partition constant in PFC process.
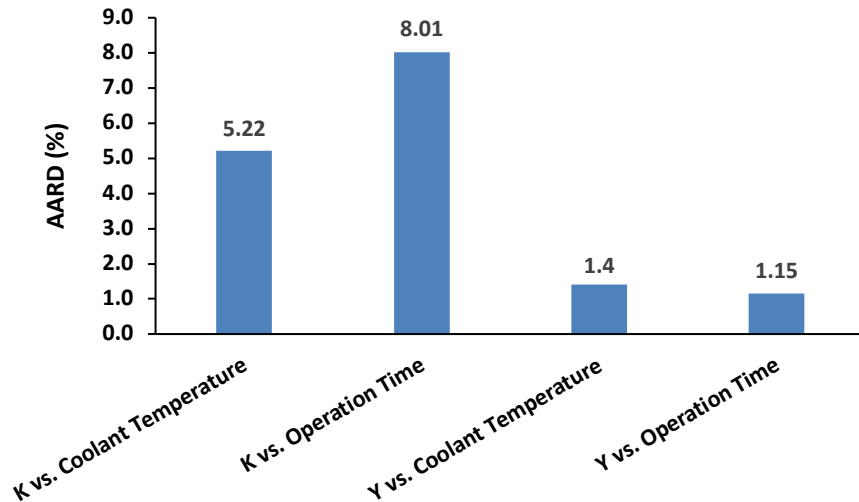


**Figure 8.** PFC performance comparison of the employed operating conditions based on AARD

## Graphical Verification of the Models

In addition to verifying the accuracy of the model, Figure 9 and 10 indicate the scattering points of PFC performance to compare between experimental (or actual) and predicted values for effective partition constant and solute recovery. As it can be seen from the aforementioned graphs, both actual and model predicts the target variables with satisfactory accuracy. In other words, the results produced by models were closer to actual ones with lower average deviation. Briefly, the prediction performance of polynomial regression model as functions of coolant temperature and operation time provides the good results with the least analysis error of AARD (i.e., below 10%) and the highest R-squared (i.e., above 0.97).
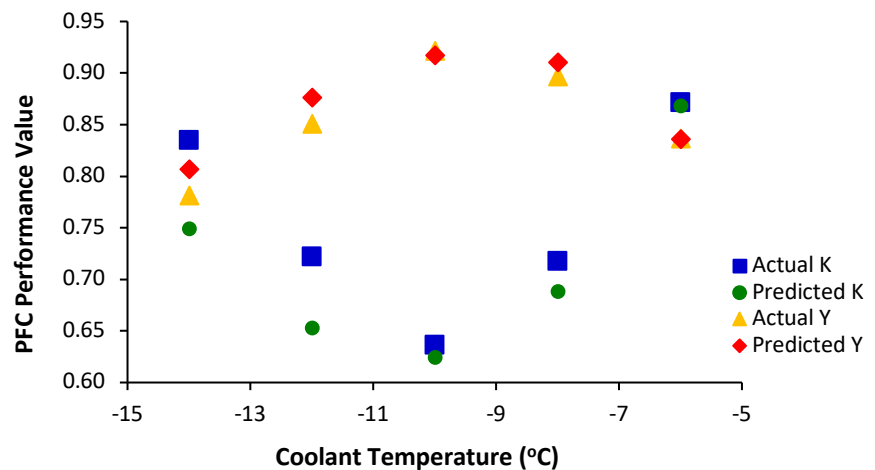


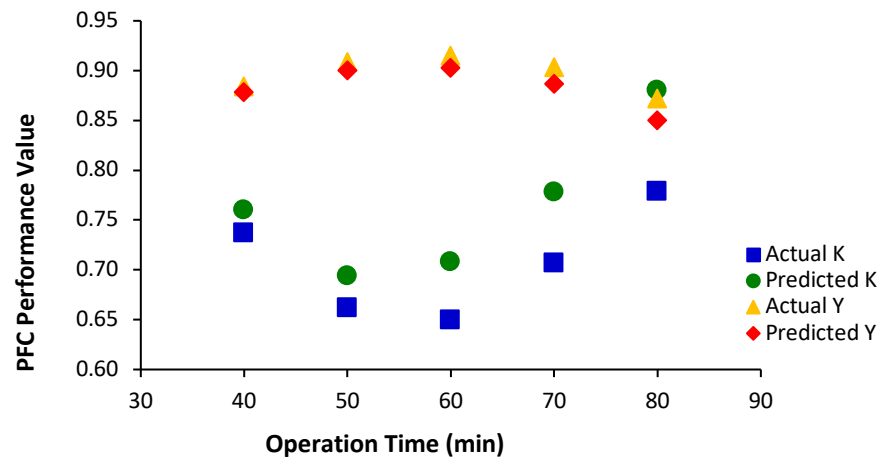**Figure 9.** Prediction performance of polynomial model for *K* and *Y* as a function of coolant temperature

**Figure 10.** Prediction performance of polynomial model for *K* and *Y* as a function of operation time

## Conclusions

PFC using VFC crystallizer can be defined as one of the effective methods to concentrate star fruit juice. Throughout this study, the use of polynomial regression model is an important tool to analyse the performance of PFC. With the help of MATLAB, simulations have been performed for the detailed analysis of the effective partition constant and solute recovery with respect to the parameters of coolant temperature and operation time according to the selected system approaches. For validation strategy, the models' accuracy was assessed through R-squared and error analysis of AARD. In conclusion, the mathematical model generated has shown satisfactory results in simulating a PFC process for the concentration of star fruit juice. It is also possible that the use of modeling method might solve the shortcomings of experimental works being time-consuming, high cost of initial investment, and the complexity of controlling the ice crystal growth.

## Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

## Acknowledgements

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