Fractional Crystallization for Wastewater Treatment from Food Industries: Effect of Operation Time and Solution Concentration

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Abstract**—** Fractional crystallization is a process where the water component in a solution is frozen and crystallized as ice so that the more concentrated solution will be left behind. This process is used to recover the water component in the waste solution from food industries during the wastewater treatment. The experiments were carried out to examine the effect of operation time and solution concentration on fractional crystallization process for the wastewater treatment. The performance analysis was executed by using glucose solution as simulated wastewater and was evaluated by the value of effective partition constant, K and percentage of water recovery, R_w. Operation time studied was recorded optimum efficiency at 15 minutes with high purity of ice formed, lowest K-value of 0.49 and highest water recovery of 42%. 1mg/ml showed the optimum concentration for solution concentration studied with the best separation efficiency. It was recorded the lowest K-value of 0.271.

Keywords— Fractional crytallization; Simulated wastewater; Water recovery; Effective partition coefficient; Freeze concentration

1. INTRODUCTION

As increasing the food processing industry, the wastewater treatments play the main role together with the rapid growth. According to the Food Processing Environmental Assistance Center at Purdue University, study shows that one of the food processing industry which is dairy sector generated wastewater in the primary waste output during dairy processing. Most of the waste is in liquid form, somewhere between 60 to 95 % of water was stated by US Dairy Sustainability Report , 2011[1].

Food industrial wastes from various industries contains substances which led to corrosive or toxic effects, cause damage to the sewage network, reduces the self-purification of rivers and have an unfavorable effect upon the living conditions for the organisms in these water. Food can be contaminated by protozoa, viruses and pathogens that may be spread from contaminated water. The effluent may contaminate the river water if there is no proper wastewater management is done. The quality of fresh water resources has become scarce due to urbanization process[2]. As the result, the poor quality of water can cause spread of chronic and acute illness as it use in the food production and preparation process. The substances that contained in the industrial effluents such as alkalis, chloride, acids, solids and salts should be remove by physical, biological and chemical method or at least reduced the concentration to an acceptable level.

Therefore, there is some treatments that present today to do the task of wastewater treatment which is evaporation and reverse osmosis. Evaporation is the simplest method but it uses a large amount of energy to supply the heat of vaporization of water[3]. If the concentrated contains volatile organic compounds, this technology is not suitable to be used as it can easily turn into dangerous and hazardous vapor when heated.

While, reverse osmosis is a process which separates the solute and the liquid phase through a water selective membrane. Due to involve no phase changes, reverse osmosis uses the least amount of energy and can produce water with high purity. However, clogging of the membrane can easily occur and the replacement of membrane will involve high cost.

Therefore, a new technology has been introduced to overcome the weakness of previous technology which is fractional crystallization. Crystallization techniques have been extensively utilized in the practical applications of wastewater treatment all over the world[4]. Progressive freeze concentration (PFC) method is the division under fractional crystallization technique that does not only produce the high purity of water in the form of ice, but the process of treatment also is much easier compared to biological wastewater treatment. . Besides that, the production of ice crystals are good for air conditioning system by keep it as cold heat storage[5]. In this study, the effect of operation time and solution concentration towards the partition coefficient constant, K-value, and the percentage of water recovery were determined to evaluate the efficiency of the crystallization process by using glucose solution and ethylene glycol as simulated wastewater and coolant medium.

2. METHODOLOGY

2.1 Chemicals and materials

Glucose solution was used as a substituted to actual wastewater. Glucose was diluted with distilled water to obtain the desired concentration of glucose solution. 50% v/v of ethylene glycol with water was used as the coolant medium inside the water bath.

2.2 Experimental Set-up

The laboratory equipment for PFC as shown in Figure 1 was composed of four parts which are a chiller, a motor, a stirrer, and a cylindrical vessel. The chiller was filled with ethylene glycol as a cooling medium at -8^{o}C. A stirrer was located near the ice front and stirs the solution. The aim of employing the stirrer was to reduce the concentration near the ice front, and equalize the whole concentration of the liquid phase. Other equipment needed for the experiments were UV-vis spectrophotometer with cuvette, beaker, measuring cylinder, and weighing scale for analytical method.

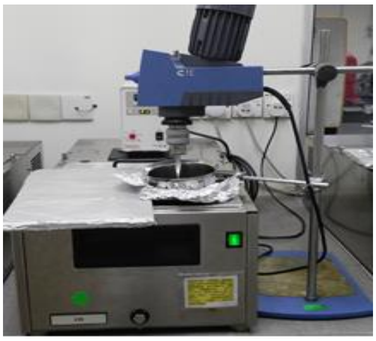

Figure 1: Equipment setup

2.3 Experimental Procedure

A water bath was filled with ethylene glycol with 50% v/v with water. The coolant was being pre cooled at -8^{O}C and the solution flowrate was constant at 200 rpm.

For the first experiment is to investigate the effect of operation time, the solution concentration was fixed at 7 mg/ml where was dissolved with distilled water. The solution was put into the cylindrical vessel to be immersed inside the water bath. After 5 minutes, the stirrer was stopped and the sample vessel was taken out of the cooling bath. The ice crystal was separated from the concentrated mother solution. The volume, V_L and solute concentration C_L in the mother liquor were analyzed. The C_L and ice concentration were analyzed using UV visible spectrophotometer. The procedure were repeated at operation time of 10, 15, 20 and 25 minutes.

For the second experiment, the same procedure was repeated at fixed operation time of 15 minutes to investigate the effect of solution concentration at 1, 2, 3, 4 and 5 mg/ml.

The results on both parameters then were analyzed on the effective partition constant, K and percentage of water recovery.

2.4 Effective Partition Coefficient, K

The effective partition constant evaluated by the concentration of solutes in the ice and bulk liquid phase[6] as defined by equation 1:

$$K = C_s / C_L \qquad \text{Equation 1}$$

where C_s and C_L are solute concentrations in ice and solution phases, respectively. According to Miyawaki ,2005, the value of K should be calculated experimentally, which is carried out by developing a linear equation from a mass balance of the process[7].

When K is assumed constant, the concentration can be determined using below equation:

$$(1 - K)\log (V_L/V_o) = \log (C_o/C_L) \qquad \text{Equation 2}$$

where V_o and C_o are the volume and the solute concentration at the beginning in the solution phase, respectively. The value of K is between 0 to 1, where K=0 when there is no freeze concentration and K=1 for complete freeze concentration.

2.5 The percentage of water recovery, R_w

Percentage of water recovery is the other parameter to determine the efficiency of PFC method. Equation 3 was used to obtained the percentage of water recovery.

$$R_w = [\,(C_O - C_s) / C_O] \times 100 \qquad \text{Equation 3}$$

Where C_o and C_s are the initial concentration of simulated wastewater and concentration of solutes in ice or solid respectively. The concentration was measured by using the UV visible spectrophotometer. The absorbance of mother liquor which is glucose in the solution was determined to obtain the concentration from the calibration curved prepared.

3. RESULTS AND DISCUSSION

Standard of glucose solutions with different concentration were prepared for the absorbance to be measured. Figure 2 shows the glucose calibration curve with an R^2 value of 0.9985 which shown the good curve fitting of the results to the curve constructed. The samples were diluted before being analysed to scale down the concentration to meet the range of glucose calibration curved.

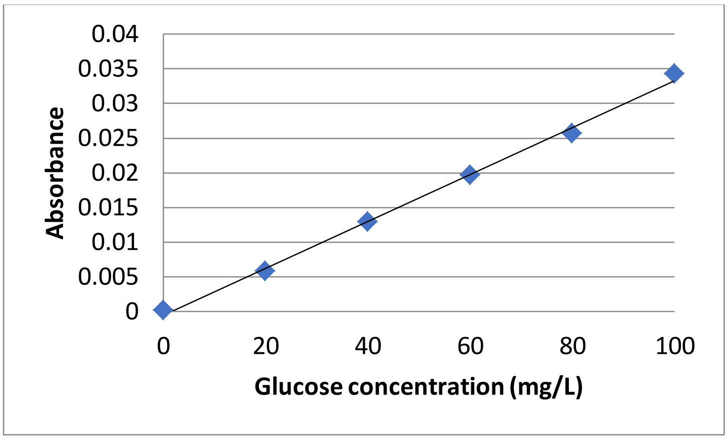

Figure 2: Glucose calibration curve

During freezing, ice crystals were formed on the surface of the cylindrical vessel. Figure 3 shows the ice layer formed in the cylindrical vessel at the end of the experiments. The thickness of the layer varied with the operating conditions varied throughout the experimental works.

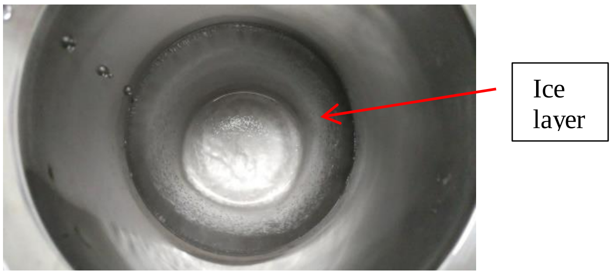

Figure 3: Ice layer formed in the cylindrical vessel

3.1 Effect of Operation Time

In order to study the effect of operation time to this process, the time was varied at the minutes of 5 to 25 while the solution flow rate, solution concentration and operation temperature were kept constant. Figure 4 shows the amount of glucose concentration in solid phase at different operation time. At 5 minutes, the ice produced seems to have quite high concentration of glucose with just a reduction of 2.3% of the original concentration due to the incomplete crystallization. At this operation time the separation or crystallization process are not started. Glucose concentration in the solid phase was found satisfactorily low at 10 and 15 minutes with a reduction of 36% and 42% of the original concentration. This indicates a good freeze concentration process has occurred at these operation times. In a meantime, the concentration of glucose shifted its trend at 20 and 25 minutes where it starts to increase from a decreasing trend. This is due to the concentrated solute was begin to formed as ice.

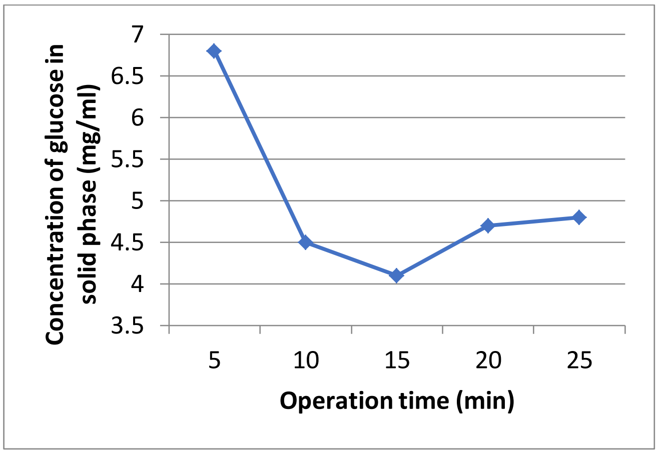

Figure 4: Concentration of glucose in solid phase at different operation time

Based on the result of operation time, the data was evaluated on the K and R_w value. Figure 5 shows the relationship of operation time with K and R_w value. From the figure, at operation time of 15 minutes, the optimum efficiency was achieved as it shows the highest value of water recovery, R_w and lowest value of K at 0.49 and 42.44%. Besides that, from the results stated in Figure 5, 15 minutes of operation time was achieved the highest purity of water formed in solid phase.

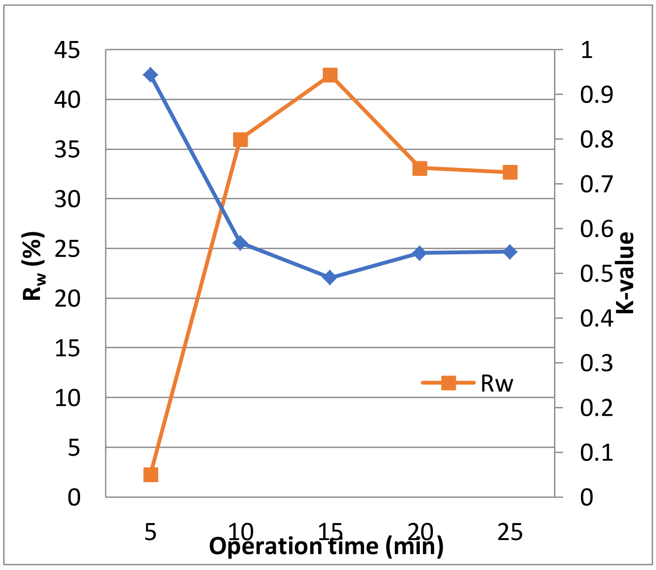

Figure 5: Effect of operation time on R_w and K-value

3.2 Effect of Solution Concentration

The range of solution concentration studied was 1 – 5 mg/ml, where the solution flow rate, operation time and operation temperature were kept constant. The operation time was continuously constant at 15 minutes.

From the analysis of concentration of glucose in solid phase, it showed that the range of reduction of glucose content was from 33% to 46%. The pattern of reduction was not depends on the concentration gradient. Figure 6 shows the concentration of glucose in solid phase at different solution concentration. At 1mg/ml solution concentration, the highest reduction of glucose concentration was recorded with 46.7% reduction. At this point, it indicates a good freeze concentration process.

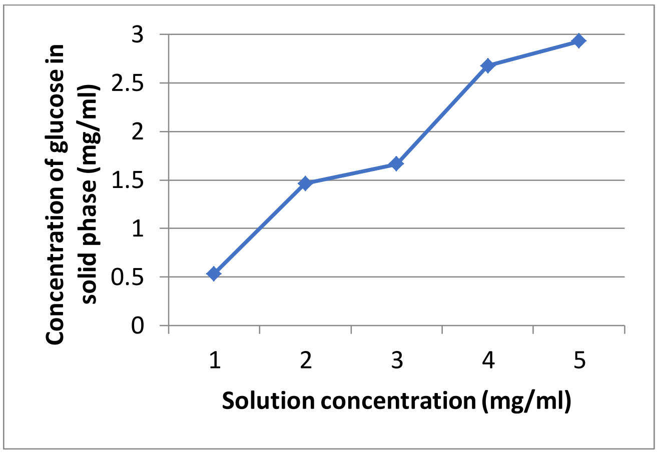

Figure 6: Concentration of glucose in solid phase in different solution concentration

The effect of solution concentration was further evaluated with the value of R_w and K-value to determine efficiency of the PFC process. Figure 7 shows the relationships of solution concentration on both efficiency parameters. From the Figure 7, the better separation efficiency between solid and liquid happen at 1mg/ml solution concentration. It was recorded with highest value of water recovery and lowest value of K with 46.8% and 0.271. It can be conclude that better separation with better efficiency should be run at 1mg/ml solution concentration. This indicates at the lower concentration, the amount of solute is less and the chance to get trapped in the ice is low. At higher solution concentration like 5mg/ml, the concentration of glucose recorded in solid phase was high at 2.932. This is due to the high amount of solute will give higher chances of solute to get trapped in the ice.

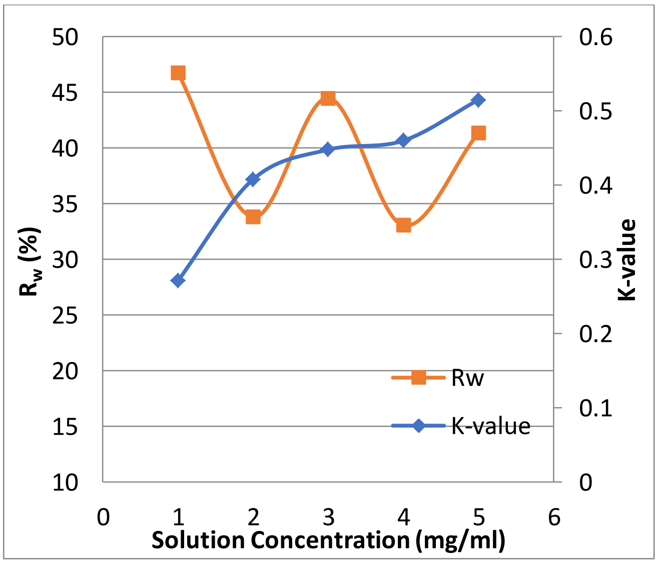

Figure 7: Effect of solution concentration on R_w and K-value

4. Conclusion

This work has proven that the designed of PFC is capable of producing ice layer and is seemed relevant for the purpose of wastewater treatment especially in food industries. The best operation time is determined at 15 minutes with K-value and R_w is 0.49 and 42.44% respectively. Meanwhile, with the K-value, 0.271 and R_w, 46.8% recorded, 1 mg/ml is the best solution concentration in crystallization process. The parameter studied should be further investigated in order to discover the most optimum operating conditions, where it could produce ice layer with highest purity.

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