Optimization of Stormwater Purification: Coolant Temperature and Operation Time

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***Abstract*— Shortage and lack of sources of clean water is a big issue nowadays. This problem happens because of the increment of population. New sources of water need to be discovered. Therefore, the sources of water must be explored in order to get continuously supply of pure water. This paper recommended a new technology to get pure water which is by purifying the stormwater using progressive freeze concentration, (PFC). This method produces single ice crystal block that have high purity of clean water. The parameters that were investigated in this paper are coolant temperatures which from -6°C to -10°C and operation time from 10 minutes to 50 minutes and the constant parameter are rotation speed at 250 rpm and initial concentration at 2mg/l. In order to determine the effectiveness of this technology, effective partition constant (K) and concentration efficiency (Eff) were examined. To determine the optimization of operating condition, response surface methodology (RSM) was used by producing an appropriate design of experiment using STATISTICA Software Version 8. From the experiment, the system attained the optimum condition at temperature – 8.9°C, operation time at 41.12 minutes. The optimum value of K and Eff predicted by ANOVA are 0.1096 and 89.0235 respectively.**

Keywords— crystallization stormwater, progressive freeze concentration, response surface methodology.

I. INTRODUCTION

The world population in 20th century is tripled but the water consumption is six times higher than amount of people. Most of the water is used for drinking, bathing, cooking, cleaning, watering plants and also for industrial uses [1]. However, there are many signals that warn the world that water supply is in critical condition due to high population and urbanization [2], [3]. This problem might lead to damaging the ecosystem and people in urban area getting less water.

One of the ways to solve this problem is by finding other resources of water which is by purifying storm water. Stormwater is water resulting from rain or snow. It may falls on streets that contain oil and grease, soil that contain fertilizer and pesticides and many more. This water will be drained to lakes and nearby river which is the sources of water supply [1].Stormwater management is very crucial because it can avoid damage to property and human from flooding, preserve the ecosystem and as our water resources [4]. This water can be used as ground water recharge and flood protection [5]. When flood happen, people in that area will having shortages of clean water supply. By doing this purification process, the flood water can be used as clean water supply to flood victim. Therefore, by managing this stormwater it provides benefit to people and also environment.

To purify the stormwater, many researchers found that there are several technologies that can be used to purify stormwater such as multistage flash (MSF), electro dialysis (ED) and many more. But the recently used are evaporation, progressive freeze concentration (PFC) and reverse osmosis which is known as RO. Reverse osmosis, (RO) is a technique that use semi-permeable membranes in order remove dissolved materials, nitrate, colour and other chemical from water. It is commonly used to treat municipal waste water. Mostly RO is being implemented for desalination of seawater. RO process consists of four steps which are pre-treatment system, high-pressure system, membrane system and post-treatment [6]. The process is very effective but it removes the entire component in the water that makes the water acidic.

Evaporation system is commonly used as water purification because it can produces high purity of water. There are several category of evaporation method include multi-effect distillation (MED) and mechanical vapour recompression (MVR). Some of the experiment shows that evaporation method is related to direct contact between air and water but it is also can used to purify waste water. Stormwater contain many impurities thus, the techniques to purify between stormwater, air and water are different. Besides that, evaporation can remove all the heavy metals and, dangerous chemical in waste water resulting in the high purity of water but the power consumption is too high. This will leads to highly cost process and installation [7].

Researchers are still investigating to find another alternative to purify waste water such as stormwater because RO and evaporation is not preferable. They found that progressive freeze concentration (PFC) has the high potential to replace the previous technology since it can produce the high purity of water. PFC can be defined as a process that produces a big single ice crystal. It separates the original solution by making an ice crystal. Ice crystal that forms is the pure water that contains no impurities [8]. The advantages of PFC are it is unique and simple process which has lower cost and energy consumption. There are many designs that have been introduced to get cost-effective and easy-to-handle process. In PFC process, it produces single block of ice crystal that will make the division process of ice crystal block and concentrated solution become simpler thus can produce very low maintenance cost [9].

To search the optimization condition for this method, RSM has been used by evaluating the design of experiment. This optimization will produce the most suitable operating condition for this process [10], [11]. RSM is known as the most relevant optimization method that can be used as sensible progression for purification of stormwater using PFC technologies. The aims of this experiment are to investigate the optimum condition for coolant temperature and operation time for stormwater purification through PFC system and to study the interaction between coolant temperature and operation time. To determine the effectiveness of PFC system, effective partition constant (K) and concentration efficiency (Eff) were examined.

II. METHODOLOGY

A. Materials

In this experiment, the simulated stormwater was used as raw material by diluting 100 mg/l of zinc chloride solution to 2mg/l to get more accurate reading of concentration. For coolant, 50w/w of ethylene glycol and 50w/w of distilled water were mixed. The feed samples contain distilled water and zinc chloride and must be perfectly mixed until dissolved solution obtained. Zinc chloride is chosen as simulated stormwater because it is the most dangerous particulate and need to be removed from water and it also has higher composition in stormwater [12]. The material that acts as coolant in this experiment is ethylene glycol. Ethylene glycol is one of the chemical that can transfer heat in very low temperature process [13].

B. Laboratory Equipment and Experimental Set Up

In order to perform this experiment by using PFC, there are four types of apparatus involved which are cooling bath, stirrer, crystallizer and UV-Vis. Cooling bath acts as cooling source to crystallize the solution. The appropriate temperature need to be set up so that the crystallization process can occur. Crystallizer acts as equipment that used to convert sample liquid into ice crystal [9]. To increase the performance and effectiveness for PFC process, stainless steel crystallizer was used [14]. Besides that, the purpose of using UV-Vis is used to estimate the water quality which is it convey spectra that related to many aggregate stormwater quality parameter [15]. The purpose using stirrer to introduce the movement of the solution which is purposely to give a consistent circulation flow thus, it can reduce between liquid and ice's accumulation [13], [16].

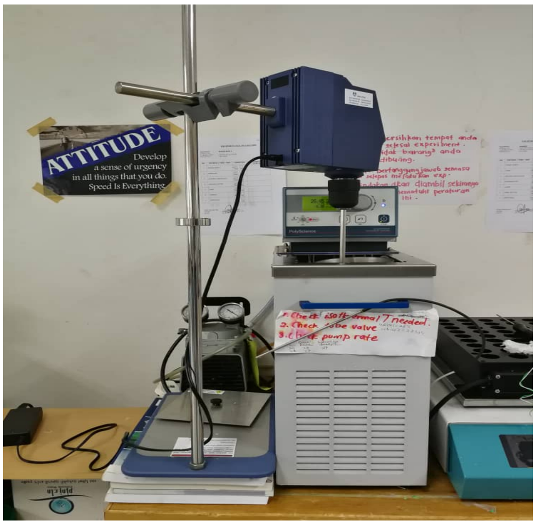


Figure 1: Experimental set up

C. Experimental procedure

Simulated stormwater was performed by using zinc chloride and distilled water by diluted 100 mg/l of zinc chloride solution to 2mg/l since the initial concentration of simulated zinc chloride is constant at 2mg/l**.** For coolant which is ethylene glycol, the coolant was diluted with distilled water to get 50%v/v and it takes around 2 hours to achieve required temperature at -6 °C to -10 °C After that, the 500 ml of simulated stormwater solution was fed into crystallizer. The crystallizer was immersed directly in the cooling bath. This is because to encourage the crystallization process to happened. The rotation speed was constant at 250 rpm since the variables are only operation time and coolant temperature [16]. To make the process become more effective, the initial super cooling must be avoided from happen because it can cause high impurities [17]. During the running process, ice layer started to appear on the wall of the crystallizer.

The crystallizer was removed from cooling bath to be thawed right after the rotation was stopping completely after the selected time which ranging from 10 minutes to 50 minutes. Then the sample of ice layer that form during process was collected while the concentrate of the simulated stormwater was drawn off [16].The solution that contain more solute was fully draw off and a sample of the ice layer generated was assembled [13]. Designs of experiment involving parameters which are coolant temperature and operation time are determine by using STATISTICA Software Version 8. The constant variable is initial concentration which is constant at 2mg/l and the other is rotation speed which is remaining at 250 rpm. At the end of experiment, the volume and concentration of both concentrated solution and ice form were recorded and collected to further investigation**.** By using analysis of variance (ANOVA) in STATISTICA Software, the optimum condition for operating time and coolant temperature were obtained.

D. Experimental design for RSM

In order to discover the optimum and standard condition for this process, experimental work was performed by suing RSM. The central composite design (CCD) was used to investigate the relationship between all the process variables and identify the best optimum condition for purification process by using RSM [11].Experiment was started using STATISTICA software version 8.0 Statsoft Inc., USA to determine the data spreadsheet. Then, the experiment was started based on the spreadsheet which has the parameter of X that consists of coolant temperature and operation time and Y that consists of effective partition constant (K) and concentration efficiency (Eff).

III. EVALUATION SYSTEM EFFICIENCY

A. Effective Partition Constant (K)

To determine the effectiveness of progressive freeze concentration, it depends on effective partition constant, K value. This is because the elimination of the solute molecules from ice front motion and boundary between solution phases and ice the core process of concentration in PFC. K value can be expressed by the following equation:

$$K=CS/CL \qquad \text{(Eq. 1)}$$

CS is known as ice concentration while CL is concentration of stormwater solution. The equation of K can be integrated:

$$(1-K) \log (VL/Vo) = \log (Co/C) \quad \text{(Eq. 2)}$$

Means that Co as initial concentration of stormwater solution, VL represents concentrate volume [9], [13], [14].

B. Concentration Efficiency (Eff)

For the concentration efficiency (Eff), it indicates the relationship between the increasing in the concentration of the solution in liquid phase corresponds to the initial concentration of the mixture, as given in Eq. (2) [11]:

$$\text{Eff} = \frac{CL - CO}{CL} \times 100 \quad \text{(Eq. 3)}$$

where Eff is the concentration efficiency (%), CL is the concentration of zinc chloride liquid solution after the process, and CO is the concentration of the ice crystal. Effectiveness and performance PFC system can be known from the value of K and Eff [18] [19].

IV. RESULTS AND DISCUSSION

A. Model adequacy and fitting

To run this experiment, process if determining the design of experiment by using STATISTICA Software Version 8. Figure 2 shows the image of ice crystal generated during the experiment. The arrows elucidate the thickness of ice formed. Table 1 shows the experimental range used in this study and Table 2 show the design of experiment and the response from the experiment. The effective partition constant and concentration efficiency, Eff were related to the manipulated variables which are coolant temperature and operation time that known as the responses of the process by using multiple polynomial regression analysis.

Figure 2: Ice crystal formed

Table 1: Experimental Range

	Range				
Type of Parameter	-α	-1	0	+1	+α
Coolant Temperature (^{0}C) (X_1)	-5.2000	-6	-8	-10	-10.8000
Operation Time (minutes) (X_2)	1.7000	10	30	50	58.2000

Table2: Design of experiment and response

Run	Manipulated Variable		Process Variable	
Run	Coolant Temperature (X_1)	Operation Time (X_2)	K	Eff
1	-10.0000	10.0000	0.2866	71.3398
2	-10.0000	50.0000	0.1356	86.4351
3	-6.0000	10.0000	0.4711	52.8833
4	-6.0000	50.0000	0.2542	74.5818
5	-10.8284	30.0000	0.2079	79.2399
6	-5.2000	30.0000	0.4007	59.9286
7	-8.0000	1.7000	0.5164	48.3566
8	-8.0000	58.2000	0.1844	81.5626
9	-8.0000	30.0000	0.1466	85.341
10	-8.0000	30.0000	0.1666	83.3374

Multiple regression equations for K (Y_1) and Eff (Y_2) known as a function of coolant temperature (X_1), and operation time (X_2) and their interaction using linear and quadratic regression coefficient of main factors and linear-by linear regression coefficients of interaction are calculated in equation:

$$Y_1 = 1.915304 + 0.303266X_1 - 0.021546X_2 + 0.015933X_1^2 + 0.000217X_2^2 - 0.000412X_1X_2 \quad \text{(Eq. 4)}$$

$$Y_2 = -91.4975 - 30.3112X_1 + 2.1557X_2 - 1.5920X_1^2 - 0.0217X_2^2 + 0.0413X_1X_2 \quad \text{(Eq. 5)}$$

Y is the predicted responses from the experiment. In order to determine the fitted model for the process, coefficient of determination (R^2) and analysis of variance (ANOVA) was used.

B. Analysis of variance

The adequacy of the regression model also can be generated by using ANOVA method. F-value can be calculated from ANOVA result. Generally, F-value calculated from ANOVA must be greater than F-tabulated at r (F0.05, 5, 4) at 95% confidence level to avoid hypothesis saying that all regression coefficient is zero. In this experiment, F-values calculated were 27.6148 and 27.7242 while F-tabulated is 6.2561 as tabulated in Table 3 and Table 4. This shows that both F-values are higher than F-tabulated, Thus, the hypothesis is rejected. To get a good fit model, the R^2 should greater than 0.75 [20]. R^2 from this experiment are 0.97185 for K and 0.97195 for Eff. This means about 97.2% of the sample is fit to the variable while only 2.8% is irrelevant to the variable. Therefore, the model is very fit for the model.

Based on ANOVA analysis, P-value must be lower than 5% for all factor so that it will bring more effect to K and Eff and vice versa. Table 5 and Table 6 show the regression analysis that will be referring as method to determine the consequence of regression coefficients of the model for responses of K and Eff. Basically, the variable that has lower value of P and higher value of F will bring the most significant effect to the process. From table 5 and 6, the operation time (X_2) has the lowest P and highest F which are 0.001063 and 71.80626 for K and 0.001055 and 72.07366 for Eff [11]

Table 3: ANOVA for quadratic model for K.

Sources	Sum of Squares (SS)	Degree of Freedom (d.f)	Mean Squares (MS)	F-value
Regression (SSR)	0.168554	5	0.0337108	27.6148
Residual	0.004883	4	0.00122075	
Total (SST)	0.173437	9		

Table 4: ANOVA for quadratic model for Eff.

Sources	Sum of Squares (SS)	Degree of Freedom (d.f)	Mean Squares (MS)	F-value
Regression (SSR)	1686.462	5	337.2924	27.7242
Residual	48.664	4	12.166	
Total (SST)	1735.126	9		

Based on ANOVA analysis, P-value must be lower than 5% for all factor so that it will bring more effect to K and Eff and vice versa. Table 5 and 6 show the regression analysis that will be referring as method to determine the consequence of regression coefficients of the model for responses of K and Eff. Basically, the variable that has lower value of P and higher value of F will bring the most significant effect to the process. From table 5 and 6, the operation time (X_2) has the lowest P and highest F which are 0.001063 and 71.80626 for K and 0.001055 and 72.07366 for Eff [11].

In Figure 3 and Figure 4, both bars exceeded to the right of the line p = 0.05 indicates significant factors with the both linear term of operation time and coolant temperature and both quadratic term of operation time and coolant temperature were rated as the most and least significant respectively. The other factor can be determined as irrelevant to the process.

Table 5: regression analysis for K

Factor	Coefficient Estimation	Standard Error	F	P
X_1	0.303266	0.066952	33.94379	0.004323
X_1^2	0.015933	0.004085	15.20980	0.017542
X_2	-0.021546	0.004312	71.80626	0.001063
X_2^2	0.000217	0.000041	28.20139	0.006044
X_1X_2	-0.000412	0.000437	0.88936	0.399048

Table 6: Regression analysis for Eff

Factor	Coefficient Estimation	Standard Error	F	P
X_1	-30.3112	6.68370	34.11236	0.004284
X_1^2	-1.5920	0.40784	15.23722	0.017490
X_2	2.1557	0.43051	72.07366	0.001055
X_2^2	-0.0217	0.00408	28.31217	0.006002
X_1X_2	0.0413	0.04360	0.89599	0.397458

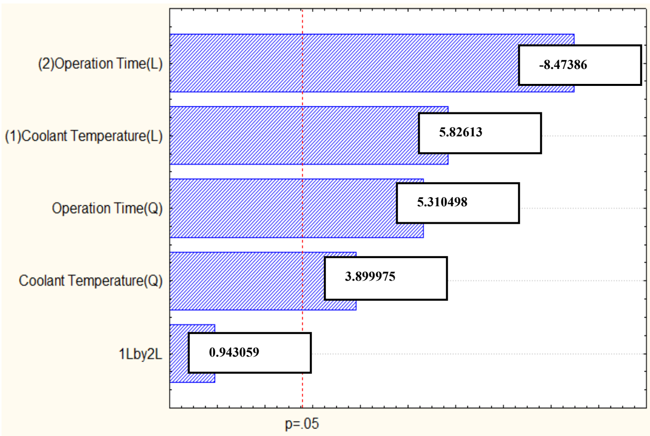


Figure 3: Pareto chart of standardized effect on K

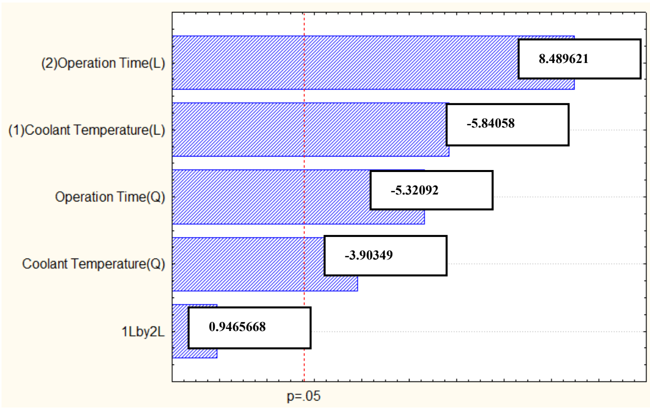


Figure 4: Pareto chart of standardized effect on Eff

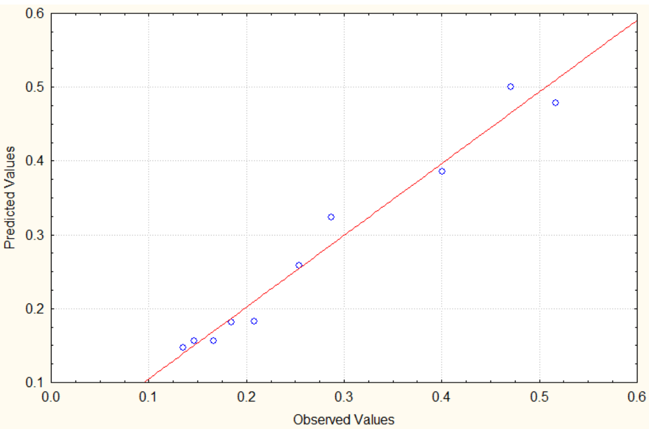
A predicted value of this experiment can be created by using regression model that are listed in Table 7 and 8. Figure 5(a) and 5(b) represents the observed value and predicted value of K and Eff. The residuals in Table 7 and 8 shows the difference value between observed and predicted data. From figure 5(a) and 5(b), both observed value of K and Eff were very near to straight line of predicted value. This clearly shows that errors in this experiment are less. From residual value, the negative and positive value represent that these experiments have good quality of errors scattering. From the result, it obviously shows that data are fit to the second order polynomial models.

Table 7: Observed value and predicted value of K

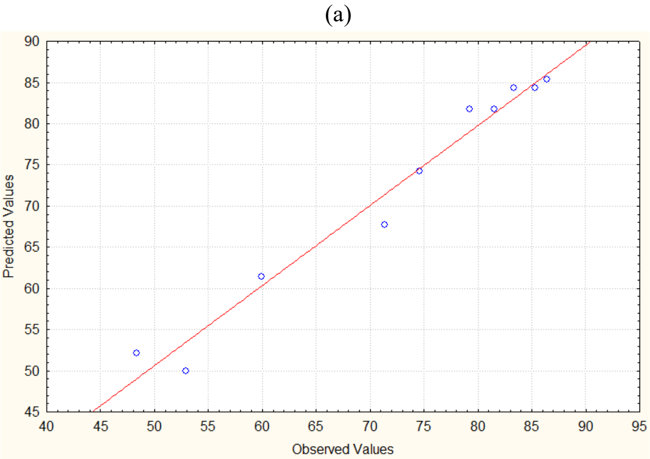
Run	Observed Value	Predicted Value	Residuals
1	0.286600	0.323345	-0.036745
2	0.135600	0.146940	-0.011340
3	0.471100	0.500235	-0.029135
4	0.254200	0.257930	-0.003730
5	0.207900	0.182281	0.025619
6	0.400700	0.385844	0.014856
7	0.516400	0.478199	0.038201
8	0.184400	0.182126	0.002274
9	0.146600	0.156600	-0.010000
10	0.166600	0.156600	0.010000

Table 8: Observed value and predicted value of Eff

Run	Observed Value	Predicted Value	Residuals
1	71.33980	67.67500	3.66480
2	86.43510	85.31195	1.12315
3	52.88330	49.96838	2.91492
4	74.58180	74.20852	0.37328
5	79.23990	81.78918	-2.54928
6	59.92860	61.41740	-1.48880
7	48.35660	52.17285	-3.81625
8	81.56260	81.78442	-0.22182
9	85.34100	84.33920	1.00180
10	83.33740	84.33920	-1.00180



(a)



(b)

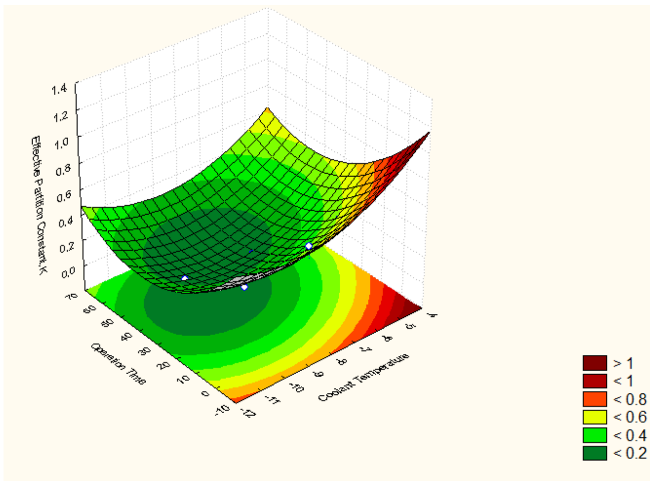
Figure 5: (a) Observed value versus predicted value of K; (b) Observed value versus predicted value of Eff.

C. Response Surface Contour Plots Analysis

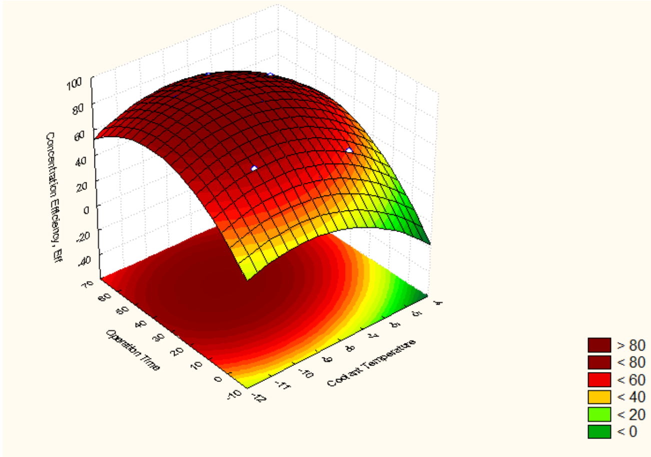
To evaluate the effect and interactions between responses whether at the middle point which optimum point of the other variables, it can be getting from contour plots. Figure 6(a) and Figure 6(b) illustrate the value K and Eff as a function of independent variable which are coolant temperature and operation time. The constant variable in this experiment are initial concentration at 2mg/l and rotation speed at 250 rpm. Figure 6(a) 3D plot shows that lowest value of K can be obtained at the range of -7.8^0C to -11.3^0C for coolant temperature and 24 to 60 minutes for operation time. Value of K seems to decrease in inclination and can change when the coolant temperature achieves at certain value. From the theory, it can be said that when the temperature is too low, the tendency of ice crystal form rate is quite higher, thus the inclusion of concentrate at ice also high which contribute to higher value of K [21]. As the coolant temperature increases, the K value is reducing meaning that crystallization process was successfully.

On the other hand, too high coolant temperature also can cause inclusion of solute to ice crystal since the concentrated solution contain too high solute[22].The value of efficiency of the system was observed to be increased as the coolant temperature decreased. Thus, the solid was able to grow in more ordered pattern since the rate of heat transfer is slower at higher coolant temperature. The value of K seems to be at low value when the operation time was increased. However inclusion of solute from solution into ice crystal can happened when solution contain too much solute at certain time.

The relationship between coolant temperature and operation time for responses of Eff is illustrated in Figure 6(b) .The figure shows that higher Eff can be obtained between -7.2^0C to -10.8^0C for coolant temperature and 28 to 52 minutes for operation time. This means that ice crystal can be generated more in ordered pattern when the operation time is higher. It was notice that intermediate operation time and coolant temperature bring a higher efficiency of the process result is lower K and higher Eff.



(a)



(b)

Figure 6: (a) 3D contour plot for interactions between factors affecting response K; (b) 3D contour plot for interactions between factors affecting response, Eff.

D. Optimum condition

The predicted or optimum value that generated from this RSM can be referring in Table 9. This shows that with this optimum condition, the concentration of zinc chloride in ice will be lower compared to other conditions. At these conditions, the ice growth rate is higher and the inclusion level solute from solution to ice is lower.

Table 9: Optimum condition for K and Eff.

Responses	Coolant Temperature(^{0}C)	Operation Time (minutes)	Predicted
K	-8.9000	41.1300	0.1096
Eff	-8.9000	41.1200	89.0235

V. CONCLUSION

PFC system is proven that it could be successfully implemented purify the stormwater. The ice crystal formed shows that pure water can be produced through PFC process. In addition, RSM also was successfully produce a good outcome or optimum condition in order to run PFC system. Coolant temperature and operation time bring significant effect to the process. From the result, it indicates that regression model generated is a fit model since the R^2 are 0.97185 for K and 0.97195 for Eff. This means about 97.2% of the sample is fit to the variable while only 2.8% is irrelevant to the. According to the STATISTICA Version 8 software, it can be concluded that the optimum K and Eff for this system could achieve coolant temperature at -8.9˚C and 41.13.

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