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Optimisation of stormwater purification in progressive freeze concentration: Stirring speed and initial concentration

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Abstract

A method has been earnestly studied which is progressive freeze concentration (PFC). In this study, zinc chloride dissolved in distilled water has been used to signify stormwater as its amount is the highest and high toxicity in actual stormwater. Response surface methodology (RSM) was employed to determine the optimum condition of the process. The experiment was run by using the data generated from STATISTICA software with constant coolant temperature and rotation time which is at −8 °C and 15 minutes. The interaction between the process conditions gave significant effects to the effective partition constant (K) and solute recovery (Y). The predicted optimum condition for the system within the experimental ranges would be a stirring speed of 245 rpm and initial concentration of 6.846 mg/L with the best value for K and Y are predicted at 0.3357 and 0.5856, respectively. Shortage of clean water supply, flooding and excessive stormwater runoff became a concern towards the human being as these problems cannot be simply solved by building a specific pipeline system. Thus, an excellent approach and process needed to manage and purify the stormwater as well as transform it as one of the clean water resources.

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1.0 Introduction

Earth is a planet that is approximately covered with 14,108 km^3 of water (Yahya et al., 2017). Water is an important source for living things and industries that exist on the earth. However, the largest water that occupies the earth is saltwater, while by far freshwater is required for most living things and industries. Clean water is mainly supplied from rivers, lakes, and groundwater. Lakes and rivers are valued as water sources for various kinds of usage such as water transport, recreation, and for tourism. Furthermore, treated lakes and rivers are supplies for drinking purposes. Freshwater from the lake is one of the important water sources for home usage, industries and businesses (Yahya et al., 2017).

Due to the rise of industrialisation, tourism, and agriculture development, the amount of water usage is increasing at an alarming rate (Li et al., 2018). Out of all the land-uses of water supply, the major vicissitudes to volumes and pattern flow running from catchments to watercourses was caused by urbanisation. The urban water system is divided into three different elements, which are water supply, wastewater, and stormwater. Thus far, managers of urban water resources mainly focus on water supply imported from rivers, groundwater, and treated wastewater. Despite the volume of stormwater like the existing water supply, stormwater is rarely considered by water resource managers as water supply (Walsh et al., 2012).

Most parts of the world witnessed the common phenomenon of increase in urban areas as a result of the apparent welfare of metropolitan living. Thus, increasing living standards is exerting a burden on water demand. This situation directed to unmaintainable withdrawal of surface and groundwater resources and deteriorating the water quality in metropolitan regions. Globally, stormwater reuse is not widespread and popular. The reason is stormwater reuse offers a numeral of distinctive defies. Depending on its source, stormwater can be extremely contaminated. In certain conditions, it can be extra polluted than secondary treated sewage (Goonetilleke et al., 2017). Nutrients in the stormwater overflow instigate from the wash-off of impermeable exteriors and surrounding soils, along with atmospheric wet and dry deposition. Superfluous nutrients in the stormwater overflow can degrade the surface water quality. The earliest flush of stormwater overflow is enriched with nutrients that cause the eutrophication of downstream water physiques and damage of marine natural balance (Xu et al., 2017). Stormwater has becoming a concern as it causes flooding problems, pipeline breakdown, and drainage system failures. Therefore, the implementation of stormwater as one of water supply resources could be an alternative way to solve the problems regarding the large volumes of water (Ortega Sandoval et al., 2019).

There are several common water purification processes implemented in industry such as evaporation and reverse osmosis (RO). Evaporation process is the simplest and leading technology in the industry. However, it suffers drawbacks such as high energy requirement for the evaporation process, hence making it an unfavourable method. (Kirkham, 2014). Besides, evaporation produces low quality of water as small amount of the solutes are trapped in vapour during the vaporisation process. The remaining solution has fewer concentrate and the vapour is extremely contaminated (N Yahya et al., 2017). As per RO, the process required the least amount of energy and is able to produce highly pure water. However, membrane blockage can effortlessly happen in most cases and thus the replacement of membrane required a huge amount of investment. Sometimes, RO's efficiency is restricted by the compatibility of the membrane and the chemical component of the solution (Khan et al., 2014). An advanced technology has been presented for the enhancement of solution concentration such as a progressive freeze concentration (PFC) technology.

PFC is a method where the solvent and solution was separated into a single ice layer. This method is simpler in nature and easier to operate. Furthermore, it is an appropriate method to separate numerous categories of solvent from solution. It is also able to enhance the concentration of solution. It is believed to be able to treat water at lower capital and maintenance costs because of the low energy requirement by the process in comparison to the evaporation technique (Samsuri et al., 2016). The fact that it does not require any heating, makes it an appropriate technology used for separation of heat-sensitive products. It can be considered that PFC is one of the best methods for water treatment (Yahya et al., 2017).

There are several factors that affect the efficiency of the PFC system such as stirring speed, initial solute concentration, coolant temperature, and rotation time. In this study, only the stirring speed and initial concentration of the solution have been focused on. The stirring speed plays one of the main roles in determining the PFC's system efficiency. This factor will enhance the flow movement of the solution, hence encourage a more efficient heat transfer process. Meanwhile, the initial concentration of the sample solution also gives a significant impact on the system performance. The amount of solute in the sample solution will affect the performance of the separation process.

The method of one-factor-at-a-time (OFAT) is commonly used for process condition optimisation. In this method, one parameter will be examined by keeping constant the other parameters. OFAT is a traditional method used mostly in previous studies. However, this method is time-consuming and there is no reflection of actual changes in the environment as this method often misses the interaction between factors which are simultaneously present.

Among the statistical experimental design methods, response surface methodology (RSM) is the most convincing and suitable tool to be applied in order to identify the effect of the factors involved in the process and their interaction with one another which ensure that the optimisation process is effective (Jusoh et al., 2013). RSM is the fastest statistical design and more economical in collecting the results. RSM is the preferable experimental design for fitting the polynomial model in order to analyse the response surface of various combinations of factors (Cojocaru & Khayet, 2011).

The experiment conducted in this study aims to optimise the process conditions by using the data generated by the statistical design of experiment (DoE) and RSM. DoE will simultaneously all the factors that affect the PFC system within the experimental runs. The relationship between the factors can be determined by this structured and systematic method. For this study, optimisation was done by using RSM in order to obtain the optimum condition of stirring speed and initial concentration for stormwater purification via PFC.

The main mechanism of concentration in the PFC system is the segregation of solute molecules from moving ice front and the interface between the ice and solution phases. The PFC system performance is depending on the effective partition constant (K). The quality of ice produced is related to the K value which can be calculated by using the following equation (Miyawaki et al., 2005):

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

where, V_L is the concentrate volume and V_o is the initial volume of the solution. C_o is defined as the initial concentration of the solution and C_L is the concentration of concentrate. The efficiency of the PFC system is higher when the K value is lower. The initial concentration of the solution can affect the efficiency of the PFC system because the amount of solute plays role in determining the separation efficiency and K value is dependent on it.

In the crystallisation process, the solute concentration will be increased when it is in liquid form. However, by depending on the process conditions, there might be an amount of solute entrapped in the ice phase. Thus, the solute recovery (Y) which signifies the amount of solute recovered in the concentrated liquid from the original solution defined the effectiveness of the PFC system. The solute recovery (Y) can be calculated by using equation as shown as follows :

$$Y = M_{s,L}/M_{s,o} \quad (2)$$

where, $M_{s,L}$ represents the mass of solute in the concentrated liquid and $M_{s,o}$ is the mass of solute in the initial solution. In theory, the solute recovery (Y) has a unit of g of zinc chloride/g of initial zinc chloride.

2.0 Methodology

2.1 Material

Zinc chloride (ZnCl) solution at various initial concentrations ranging from 2 to 10 mg/L was used throughout this experiment to represent the real stormwater. In order to produce the simulated stormwater, zinc chloride was dissolved in the distilled water. In this study, zinc chloride was chosen because its concentration and toxicity are the highest based on the properties of real stormwater (Egemose et al., 2015). In addition, ethylene glycol 50% (v/v) with water was used as a coolant in this study. Commonly, ethylene glycol was purposely used to transfer heat in very possible low temperature processes.

2.2 Methods

The experiment was started by preparing the simulated stormwater with varied concentration in a range of 2 to 10 mg/L. The solution was stirred well to ensure that zinc chloride is dissolved completely. After that, the solution was transferred into a 1000 mL volumetric flask. This step is needed to ensure that all the zinc chloride was transferred completely into the flask. After the preparation was done, the cooling bath was filled up with coolant and the temperature of the coolant was set up to achieve the desired temperature. For the feeding process, the zinc chloride solution was fed into the crystallizer at the room temperature. The schematic diagram of apparatus setup is shown in Fig. 1.

In order to let the crystallisation process occur, the crystalliser was immersed into the cooling bath at desired operating condition. The volume of the sample fed into the crystallizer is about 500 mL for each run according to the size of the crystalliser. Therefore, the level of the sample solution will be below the coolant level to be fully immersed. The stirrer was then put properly in the middle of the sample solution and the stirring speed was set at the range of 150 to 350 rpm. After 15 minutes, the stirrer was stopped. The concentrate was drained out completely and was collected in a container. The volume as well as the mass of the collected concentrate and melted ice layer was measured and recorded. The concentration of the concentrate and melted ice samples were measured by using UV-Vis.

The concentrates and melted ice layers collected from the experiment were analysed by using UV-Visible Spectrophotometry (UV-Vis) (Perkin Elmer Lambda 750) to determine the concentration of the solution. The range of the wavelength used to analyse

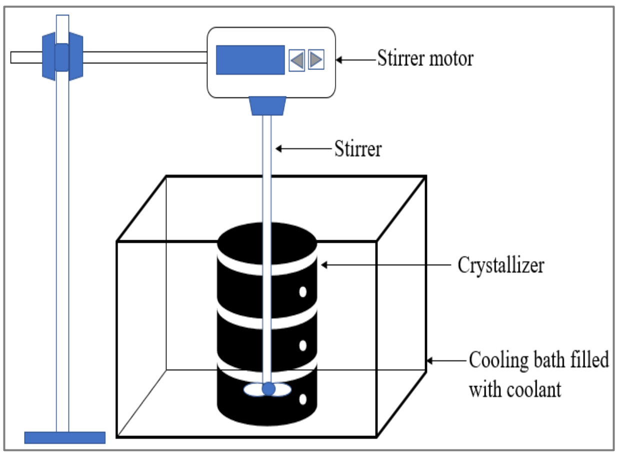

Fig. 1: Schematic diagram of apparatus setup

the solution is from 180 to 400 µm. The process was repeated at different operating conditions.

2.3 Optimisation

The optimum conditions of stirring speed and initial concentration of the solution that give the best effective partition constant (K) and solute recovery (Y) for the PFC system were determined by using RSM. In order to study the relationship of process variables and to predict the best optimum condition for stormwater purification, central composite design (CCD) has been utilized.

STATISTICA software (version 8.0 Statsoft Inc., USA) was run to obtain a series of experimental data. The experiment was implemented based on the data generated which consists of stirring speed and initial concentration of ZnCl. In this study, it is believed that stirring speed and initial concentration of solution gave significant impact to the PFC system performance. Hence, these two parameters were varied in order to analyse their effects on the PFC process of the simulated stormwater. The stirring speed was controlled through the speed of the stirrer. The range of stirring speed was determined based on the capacity of stirrer motor (from low to high speed). Meanwhile, the initial concentration was regulated through the concentration of prepared simulated stormwater which represents the range of real stormwater concentration.

Table 1: Range of parameters for the PFC process

Parameter	*Range and Levels*				
Level	$-\alpha$	-1	0	+1	$+\alpha$
Stirring speed, X_1 (rpm)	109	150	250	350	391
Initial Concentration, X_2 (mg/L)	0.34	2	6	10	11.66

Table 2: Design of experiment and responses

Run	X_1	X_2	K	Y
1	150	2.00	0.3664	0.85
2	350	2.00	0.5028	0.85
3	150	10.00	0.4664	0.52
4	350	10.00	0.2037	0.34
5	250	0.34	0.1468	1.88
6	250	11.66	0.3607	0.69
7	109	6.00	0.6561	0.70
8	391	6.00	0.5900	0.36
9	250	6.00	0.3398	0.81
10	250	6.00	0.3545	0.52

The rotation time and coolant temperature employed in this study were kept constant at 15 minutes and -8°C. Both parameters were decided based on the preliminary experiments where all the conditions used obtained the best result.

3.0 Results and discussion

3.1 Model Adequacy and Fitting

For the first stage of the experiment, the screening process parameters consist of two variables which are stirring speed and initial concentration were used in the screening experiments. The range of the operating variables employed for this PFC process is shown in Table 1 and the responses of K and Y for each run are tabulated in Table 2.

The variables are correlated with the response, K, when the second order polynomial which is multiple regression analysis is applied. Eq. (3) and Eq. (4) represents the regression equation for K and Y as a function of stirring speed (X1) and initial concentration (X2) as well as the interaction of both variables using linear and quadratic regression coefficients of main factors.

$$Y1 = 0.530425 - 0.003262X1 + 0.099913X2 + 0.000008X12 - 0.002860X22 - 0.000249X1X2 \quad (3)$$

$$Y2 = 0.882994 + 0.006568X1 - 0.233060X2 - 0.000013X12 + 0.015195X22 - 0.000112X1X2 \quad (4)$$

where, Y1 and Y2 represent the predicted effective partition constant (K) and solute recovery (Y), respectively. Coefficients with one factor indicate the effect of the specific factor and the coefficients with two factors indicate the interaction between the two variables. Meanwhile the coefficients with second order terms portrayed the quadratic effect of the factor. The sign in front of each coded variable which is the positive and negative sign represent the parallel and adverse effect of the factors to the response. The selection of the models was done based on the highest order of polynomials where the model is not aliased, and it is significant (Tan et al., 2008).

By using the same software, an analysis to evaluate the model adequacy was done. It is to judge the appropriateness of the model by determining the R-squared value. The total variation of the observed values of activity about its mean was revealed by the R-squared value (Keshani et al., 2010). In order to obtain a good fit for the model, R-squared value should be larger than 75% (Muralidhar et al., 2001), (Safiei & Shaikh Alaudin, 2021). The R-squared for the regression model that relates the effects to K and Y is 0.77 and 0.84, respectively. The R-squared value for both models were considered as acceptable at describing the adequacy of the model generated as 77% and 84% of the sample variation able to attribute to the variable and only 23% and 16% of total variance cannot be clarified by the model.

3.2 Analysis of Variance (ANOVA)

ANOVA method also used to evaluate the adequacy of the generated regression model. In ANOVA, F-value is an important value that needs to be experiential in order to evaluate the adequacy or accuracy of the model. F-value is the ratio of mean square due to regression to the mean square due to the residual error. The calculated F-value for K and Y model are 2.75 and 3.81. It is very advantages when the variable which may affect the process is identified once the validity of the regression model has been evaluated. Table 3 and Table 4 show the ANOVA for both response of K and Y value.

3.3 Response Surface Contour Plot Analysis

The effect and the interaction between the factors towards the response at the middle point of the two variables could be observed on the contour plots of the response towards the variation of studied variables at a time. A 3D surface plot of the response against the two independent variables can be plotted to observe the effect of the independent variables towards the response as shown in Fig. 2.

Based on Fig. 2(a), the contour plots portrayed the value K as a function of stirring speed and initial concentration with a constant coolant temperature and rotation time which has been set to -8°C and 15 minutes throughout the experiment. This figure shows that when the initial concentration and stirring speed decreases, it will affect K to be decreased also. The optimal range for initial concentration and stirring speed are 8 to 12 mg/L and 200 to 300 rpm. It can be concluded that the lower the initial concentration and moderate stirring speed, the better the K value obtained from the experiment. Lower K value shows that the PFC process occurred satisfactorily. Hence, the efficiency of the process is high and the purity of the ice obtained from the experiment is also high. When the initial concentration is higher, there will be a high amount of solute entrapped in the ice during solid-liquid interface. Thus, the concentration of the ice layer will be high resulting in higher K value (Ab Hamid et al., 2015). It can be concluded that the separation process performed better at lower initial concentration (Miyawaki et al., 2012).

Table 3: ANOVA for response of K value

Source	Sum of squares of error (SS)	Degree of freedom (DF)	Mean squares (MS)	F-value
Regression (SSR)	0.1451	5	0.0290	2.75
Residual	0.0422	4	0.0106	
Total (SST)	0.1873	9		
R^2	0.7700			

Table 4: ANOVA for response of Y value

Source	Sum of squares of error (SS)	Degree of freedom (DF)	Mean squares (MS)	F-value
Regression (SSR)	1.4473	5	0.2895	4.06
Residual	0.2853	4	0.0713	
Total (SST)	1.7326	9		
R^2	0.8400			

Furthermore, Figure 2(b) represents the value of Y as a function of initial concentration and stirring speed with constant coolant temperature and rotation time throughout the experiment which is at -8°C and 15 minutes. The value of Y will be high if the initial concentration is brought down and the stirring speed is increased. The optimal range for initial concentration and stirring speed for Y are 8 to 12 mg/L and 200 to 300 rpm. In conclusion, lower initial concentration and higher stirring speed will give better Y value. This means that the higher the Y value resulting in the more solute entrapped in the liquid phase during solid-liquid interface. When the solute recovery rate is higher, the ice obtained from the experiment will be high in purity as the solute is entrapped in the liquid. Therefore, the efficiency of the PFC system is increased as the stirring speed increases (Md-Zamani et al., 2015). With higher stirring speed, the solute will be washed away from the solid front as the solution is being stirred which leaves the solid with higher purity (Moharramzadeh et al., 2021).

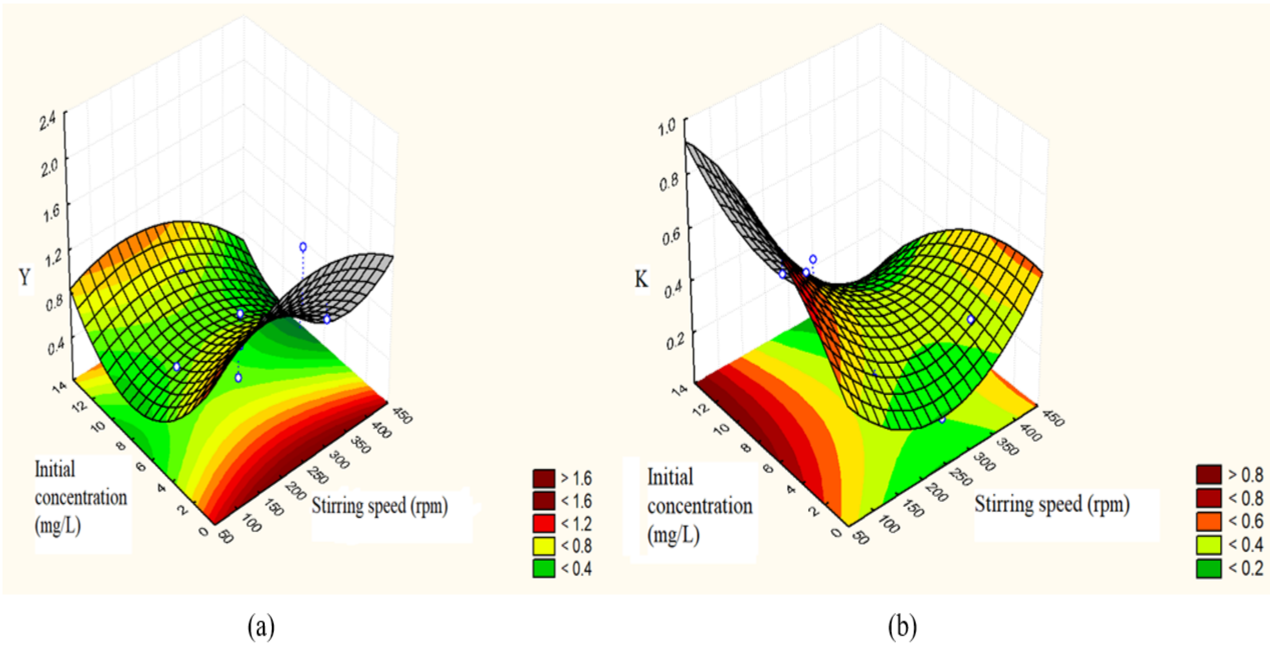

Fig. 2: 3D contour plot for interaction between factors affecting response (a) K and (b)Y

Table 5: Optimum condition from the model for response K and Y

Response	Stirring speed, X_1 (rpm)	Initial Concentration, X_2 (mg/L)	Prediction
K	280	5.248	0.3357
Y	209	8.443	0.5856

Each operating condition will obtain the optimum values which are considered as the best optimum conditions for both responses K and Y. The average optimum condition of stirring speed and initial concentration for responses K and Y obtained from the statistical software is 245 rpm and 6.846 mg/L. The range of the studied variables towards the response is summarized in Table 5.

4.0 Conclusions

RSM is proven to be the best tool to optimize the operating conditions investigated for PFC systems on stormwater purification. The fitted model generated by the STATISTICA is also accurate by observing the R-squared and F-value for response K and Y. The R-squared value for response K and Y is 77% and 84%, respectively. Based on the statistical software, the optimum condition of stirring speed (245 rpm) and initial concentration (6.846 mg/L) obtained the best K and Y value of 0.3357 and 0.5856, respectively. With the K value approaching 0 means that the PFC process is perfectly complete and Y value is approaching 1 which means that the solute entrapped in the ice layer is low. Even though the Y value obtained from the generated model is quite low, which is reflected the moderate accuracy of the prediction, but the model of K is satisfactory enough to be used to predict the K value. In this study, it is proven that the PFC method is capable of being a new technique for purifying stormwater with some modification needed especially in determining Y value.

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