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Research Paper

Box Behken design for optimization of COD removal from Palm oil mill effluent (POME) using Reverse osmosis (RO) membrane

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Abstract
The optimization of COD removal from palm oil mill effluent (POME) using the Reverse Osmosis (RO) membrane was investigated. Experimental conditions for reduce the COD value of POME were achieved successfully using the Box Behken design. The values of affecting factors (POME concentration, pH and Transmembrane pressure were optimized according to the polynomial regression model. The predicted conditions to produce lower COD values were found to be POME concentration (vol. %) =28.30, pH =10.75 and Transmembrane pressure= 0.69 kPa. The predicted of COD value was 24.137 mg/l which in good agreed with experiment value as 25.763 mg/l was obtained.

Keywords
POME, RO membrane, Box Behken, COD

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1. INTRODUCTION

Palm oil mill effluent (POME) has been reported as a main wastewater produced from industry of palm oil mill. The main process of crude palm oil extraction from fruit is required to use huge amount of water which estimated 5-7.5 tons of water for produce of 1 tons of crude palm oil. Unfortunately, more than 50% of the water has been produced as POME which causes to contaminate the environment (Ahmad, 2003). The common method to handle POME is an integrated anaerobic and aerobic pond which disadvantages large area and long residence times have been reported earlier (Yejian, 2008). Last few years, some of previous studies are trying to still this problem by including biological and physical-chemical (Chan, 2010; Mohammed, 2014; Shak, 2014). Recently, the new method of membrane technology has been applied for POME treatment which improved the quality of final process effluents (Said, 2005).

The application of membrane especially Reverse Osmosis (RO) for POME treatment has many advantages such as capability to produce clear water in relatively short amount of time, small area and energy consumption. Higher efficiency to reduce many parameters related to POME treatment process quality such as BOD, COD, and, TSS to acceptable levels set by regulatory agency (Ahmad, 2006; Wu, 2010). Effects of several parameters such the pressure and concentration on the POME treatment have been investigated experimentally for each parameter which led to increase the experiment runs and consuming time (Wu, 2007). In carrying out an experiments POME treatment which containing many affect variables, a tool such as Response Surface Methodology (RSM) is required to optimize the process response (Mohajeri, 2010).

The Response Surface Methodology (RSM) has been applied as a statistical technique to study of affecting process variables and build of experimental model with interactive variables (Muneer, 2013b). As main advantages to apply RSM is, the higher interaction between all process variables and lower runs of experiment which attributed to consuming time of the process compared the traditional optimizations (Box and Draper, 1987). For this propose, the Box Behken design with lowest run of experiment has been selected for other chemical process which exhibited more efficient and accurate for the final process response (Ismail, 2005). The

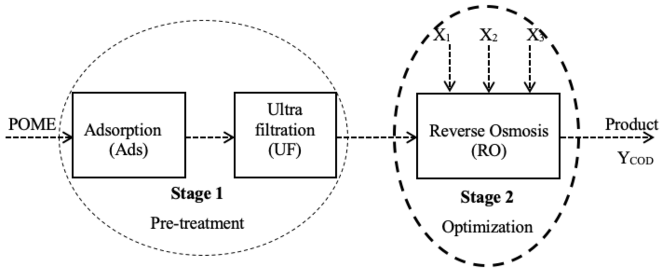

Figure 1. Experiment stages of POME treatment process

main objective of this work is to investigate the ability of RSM based on the Box Behken design to optimize the affected process variables to reduce the COD using Reverse Osmosis (RO) membrane.

2. EXPERIMENTAL SECTION

2.1 Materials and method

Raw POME was collected from a local palm oil mill in Selangor, Malaysia. To adjust of POME pH within the treatment process, sodium hydroxide and hydrochloric acid from R & M chemical, Malaysia and Merck Company respectively, were used. The POME treatment process consists of two main stages as the first pre-treatment and second optimization was designed as shown in Fig. 1.The first stage of pre-treatment includes Adsorption and Ultrafiltration membrane (UF) was used to reduce suspended solids from the raw POME. About 10 L contains different volume % (vol. %) of raw POME concentration was feed to adsorption column using dosing pump under constant flow rate at 2 ml/min. The POME flows downward along gravity, and exits at the bottom of the column which was feed into the UF membrane. The ultrafiltration pretreatment were carried out in cross flow unit with a hollow fiber membrane. Permeate from the UF membrane also simultaneously serves as feed into the RO membrane. In the stage of RO membrane, the POME feed was pumped through a spiral wound (RE2012-LPF, CSM filter) and recycled back to the UF feed reservoir. The COD values of the POME feed sample and permeate were performed and analysed using a DR/2010 portable data logging spectrophotometer (HACH, USA).

2.2 Statistical design of experiments

In this study, the Box Behken was applied based on the Design Expert software version 6.0. Determination of the DOE aims to reduce the number of experiments and obtain the optimum response (Y) according to the interaction of all the factors (Xi) were involved. The response (Y) was related with three factors can be described by polynomial Equation (1):

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \beta_{ij} X_i X_{ij} + \varepsilon \quad (1)$$

The relationship among the three factors mathematically based on the second order as given in Equation (2) is:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_{12} + \beta_{22} X_{22} + \beta_{33} X_{32} + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 - \beta_{23} X_2 X_3 \quad (2)$$

where,Y is the predicted response, βi is the coefficients, βii is coefficients of the quadratic terms, βij is the coefficients of the interactions of factors ε is random error.

The response of this study was value of COD (Y) while the affecting factors were concentration of POME (X_1), pH of solution(X_2) and Transmembrane pressure (X_3). All factors and their levels affects on POME treatment process were selected as concentration varying between 10 to 90 and of solution adjusted between 3.0 to 11.0 using hydrochloric acid and sodium hydroxide, while the Transmembrane pressure between 0.5 to 2.5 kPa as summarized in Table 1.The total number of experimental runs was 17 based on the Box design and given in Table 1.The effects of the interaction among all the factors to reduce COD value were evaluated through an analysis of variance (ANOVA) according to experimental results. Moreover, it was important to check the adequacy of the model using diagnostic graphs and validity of model by comparing the predicted to experimental results as the main steps (Muneer, 2013a,c).

Table 1. Parameters and levels of Box Behken design

Independent Factors	Xi	levels -1	0	1
POME Concentration, (vol. %)	X1	10	50	90
PH	X2	3	7	11
TMP, (kPa)	X3	0.5	1.5	2.5

3. RESULTS AND DISCUSSION

3.1 Model Fitting of Box Behken design

The effects of POME concentration, pH of solution and Transmembrane pressure on the final value of COD were investigated using the quadratic polynomial model. The mathematical model of POME treatment optimization was estimated based on the experimental results using Box Behken design with the respective coefficients as given in Equation (3):

$$YCOD = 78.36563 - 0.49438X_1 - 8.46437X_2 + 0.40000X_3 + 0.011198X_1^2 + 0.38547X_2^2 - 1.25000X32 - 0.027594X12 + 0.10831X13 + 0.52063X23 \quad (3)$$

The final model creates by the Box Behken design was involved all the coefficients which shown as a quadratic regression. The theoretical (predicted) values of final COD using the created model were compared to experimental results as listed in Table 2. The analysis of variance (ANOVA) results of the Box Behken model was presented in Table 3. The significance degree of the model and all the factors (X_1, X_2 and X_3) are estimated according to the P-value. If this value that less than 0.050 which was considered to be significant, and any other value that is greater than 0.050 was not significant (Jafarzadeh, 2011). The higher significant factors of POME concentration (X_1), the pH (X_2) and then Transmembrane pressure (X_3) well as the interaction between the POME concentration and the pH (X_{21}) were observed. However, the lack of fit of Box Behken model with F-value of 0.9193 exhibits to be insignificant relative to the pure error (Zhang, 2009).

Additionally, Other important terms are the accuracy and variability of the Box Behken model, which can be estimated according to the R-Squared (R^2) value which is between 0 to 1, with a value closer to1 being a better prediction of the response (Karacan et al., 2007). The R^2 of the Box Behken model showed a higher value, which was 0.9692. On the other hand, the Adj R-Squared (R_{adj}) coefficient was also found to be 0.9297, which was agreed to the R^2 value. However, adequate precision term of Box Behken was used for evaluated the predicted range of responses relative to the associated error. For this process, the value greater than 4 as 18.019 was found which attributed to support the fitness of the final model (Korbahti and Rauf, 2009).These values shows a good correlation between the all factors of the POME treatment process using the Box design.

3.2 Adequacy Check of the Box Behken Model

Some plots to investigate the optimization POME treatment process using the Box Behken design are given in Fig.2. The normality term which indicates the relationship between the student zed residuals with normal probability (Fig 2 a) showed all points were close to the line. This result confirmed there no obvious problems with the normality of the design based on experiments result of COD. Fig.2 b show the plot between the studentized residuals and the predicted COD values which as random scattering of all the points rather than a funnel-shaped pattern was obtained. This results, confirms that the response was an original observation of variance and no problem with the response [20]. The values of the studentized residuals were almost at intervals of between -3.5 to +3.5 as shown in Fig.2 c, and the observed response value was not considered for any value beyond these values. This model was a studentized residual value that was lower than ± 3.5, which gives a good fitting of the model to the response surface (Rauf et al., 2008). The outlier of the experimental runs POME treatment process clearly gave that all the points in the range of the outlier

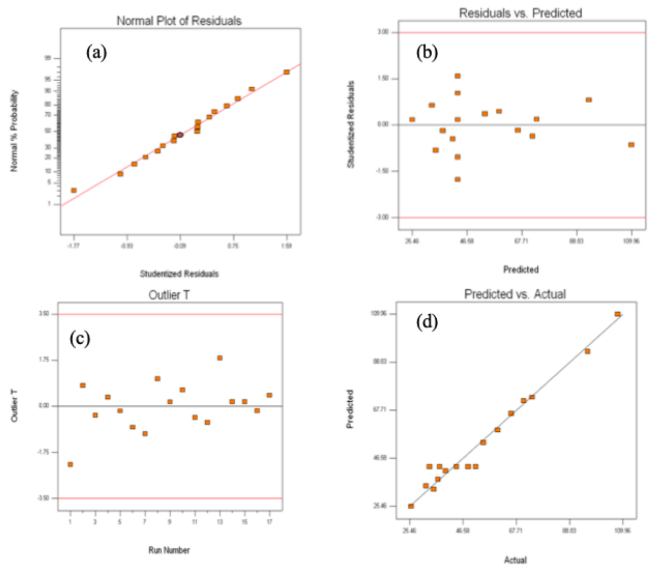

Figure 2. All diagnostic plots of optimization of COD using Box Behken design, (a) Normality, (b) studentized residuals, (c) Outlier T, (d) Actual and predicted.

as a good distribution for the Box Behken design model. The actual value of the final COD from the experimental was nearly the same as the value predicted by the model as shown in Fig2 d, which due to the higher values of R^2 and R^2_{adj} terms.

3.3 Response Surface Plotting and Optimization of Box Behken Model

3.3.1 Effects of concentration and pH solution on COD value

The effects of the POME concentration and pH on the COD value of POME treatment were investigated by the RSM based on a Box design. The 3-D response surfaces and contour graphs were used to explain the effects of the interaction between two factors as shown in Fig. 3 (a,b). It can be observed that the final COD value decreases as the pH was increased from 3.0 to 11.0. This behaviour leads to changing of surface properties of impurities in POME. At higher pH of solution indicates to the charge of the impurities could be equal to the charge on the surface of the membrane. The similarity of these charges gives more hydrophilic nature of the membrane which leads to the impurities not stick to the membrane surface and the trapped to the bulk solution (Ahmad et al., 2005). However, at lower pH of solution, the attraction force between the impurities and surface of membrane was increased. This phenomenon guides the impurities to easily attach to membrane surface and then pass through the pores. The impurities was not only included the solid particles but also the organic molecule. The existence of organic molecule can be assumed as the COD value in solution (Said et al., 2014).

The liner relationship between the final COD and con-

Table 2. Experimental runs of Box Behken design with Actual and Predicted results of COD response

Std	Run No.	(X_1)	(X_2)	(X_3)	COD(mg/L) (Actual)	COD(mg/L) Predicted
1	4	10	3	1.5	54.67	53.55
2	12	90	3	1.5	108	109.96
3	10	10	11	1,5	35	33.04
4	3	90	11	1.5	70.67	71.79
5	6	10	7	0.5	32	34.5
6	14	90	7	0.5	74	73.42
7	5	10	7	2.5	36.67	37.25
8	2	50	7	2.5	96	93.5
9	17	50	3	0.5	60.33	58.96
10	9	50	11	0.5	26	25.46
11	16	50	3	2.5	65.67	66.21
12	11	50	11	2.5	39.67	41.04
13	8	50	7	1.5	48.67	43
14	7	50	7	1.5	37.33	43
15	1	50	7	1,5	33.33	43
16	13	50	7	1.5	51.67	43
17	15	50	7	1.5	44	43

Table 3. ANOVA results for quadratic model based on the Box Behken design

Source	Sum of squares	Degree of freedom	Mean square	F-value	P-value
Model	8245.45	9	916.16	24.51	0.0002
X1	4528.19	1	4528.19	121.12	< 0.0001*
X2	1720.79	1	1720.79	46.03	0.0003*
X3	260.83	1	260.83	6.98	0.0334*
X21	1351.73	1	1351.73	36.16	0.0005*
X22	160.16	1	160.16	4.28	0.0772
X23	6.58	1	6.58	0.18	0.6874
X12	77.97	1	77.97	2.09	0.1919
X13	75.08	1	75.08	2.01	0.1994
X13	17.35	1	17.35	0.46	0.5176
Residual	261.7	7	37.39		
Lack of fit	27.72	3	9.24	0.16	0.9193**
Pure error	233.98	4	58.49		
Total	8507.15	16			

*Significant at < 0.05% level; ** Not significant, R^2= 0.9692, R^2_{adj} =0.9297, Std. Dev. = 6.11, Mean=53.75, C.V= 11.38, Adeq Precision=18.019.

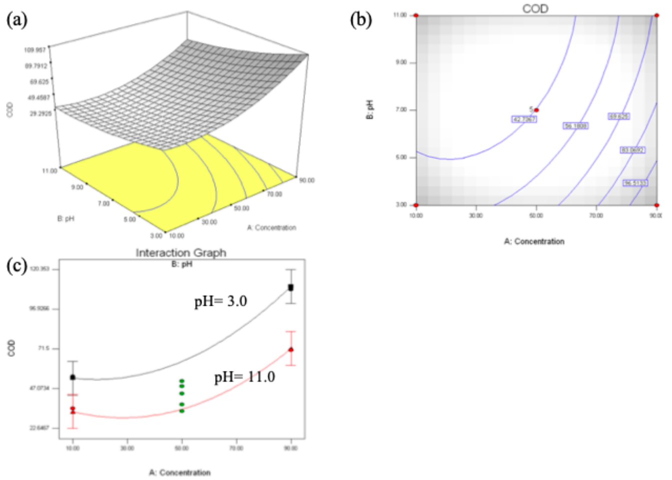

Figure 3. The effect of the concentration and pH on COD value of POME treatment (a) 3-D, (b) contour, (c) interaction

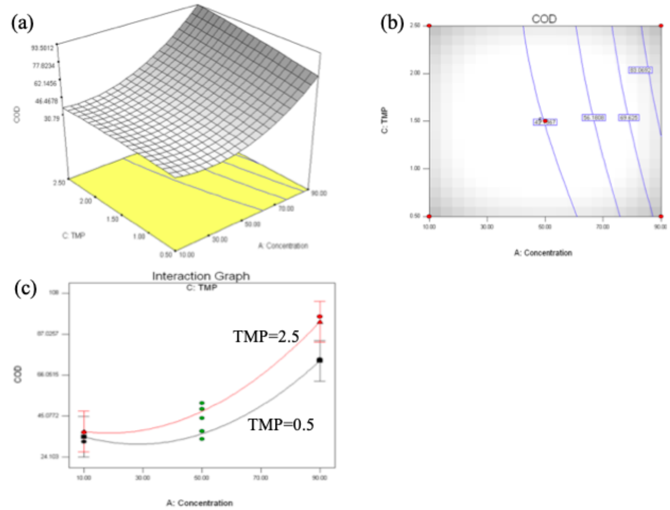

Figure 4. The effect of the concentration and Transmembrane pressure on COD value of POME treatment (a) 3-D, (b) contour, (c) interaction

centration was observed. By increasing of concentration of POME, the final COD after treatment was increased because higher concentration of POME contained more of impurities and organic molecules. The effects of interaction between the POME concentration and pH to produce on lower value of COD were shown in Fig. 3(c). Lower value of COD at lower concentration of POME was found which less than 50 mg/l at higher pH of 11.00. However, the interaction between the POME concentration and pH exhibits more affecting at all values which also was confirmed by significant of ANOVA of concentration analysis in Table 3 as p-value, $0.0005 < 0.05$.

3.3.2 Effects of concentration and Transmembrane pressure on COD value

The effects of concentration of POME and trans membrane pressure on COD value were estimated with different several values as presented in Fig.4. Slightly affecting of trans membrane pressure on COD with different values especially at lower concentration of POME (10 vol.%) was reported in Fig. 4 (a,b). This result is probably due to the lower concentration of POME as indicator to presence of smaller impurities. In addition, the increase in trans membrane pressure makes higher chance of the small particles to pass through the membrane pores (Said et al., 2014). At the same time, the COD value was found to increase as the concentration of POME increased due to the presence more amount of impurities in POME solution. To explain that higher of COD value resulting under all values of transmembrane pressure for concentration more than 50 vol.% was found to be because to presence of more impurities. Additionally, at higher transmembrane pressure causes to throughout membrane more amount of impurities which attributed to increase the final COD as reported earlier [23].

The effects of interaction between the POME concentration and transmembrane pressure to produce on lower value of COD were shown in Fig. 4(c). At lower POME concentration (10 vol.%), the changing in transmembrane pressure was not affected on COD value due to lower of impurities and organic molecules. The lower COD at lower POME concentration than 50 vol.% under transmembrane pressure of 0.5 kPa was observed. However, the interaction between the POME concentration and transmembrane pressure showed affecting at higher values.

3.3.3 Effects of Transmembrane pressure and pH on COD value

Effects of pH and transmembrane pressure on COD value were investigated under several varying values as shown in Fig.5. The lower COD values of POME at higher of 11.0 and lower of 0.5 kPa for pH and transmembrane pressure respectively were observed. At lower pH of 3.0 and all transmembrane pressure range, the higher COD values were produced. Influence of pH on COD value refers to changing the properties of impurities in POME and membrane surfaces, which at lower pH of solution, the attraction force between the impurities and surface of membrane was increased. This result leads to easily attach of impurities to membrane surface and then pass through the pores which attributed to increasing of COD value. However, the influence of transmembrane pressure also showed the COD value increases as the pressure was increased. This results because under higher transmembrane pressure leads to more impurities pass through the pore membrane compare to under lower pressure which presence of small amount of impurities as reported in previous study (Zinatizadeh and Mohamed, 2007). The pH of solution shows as the main

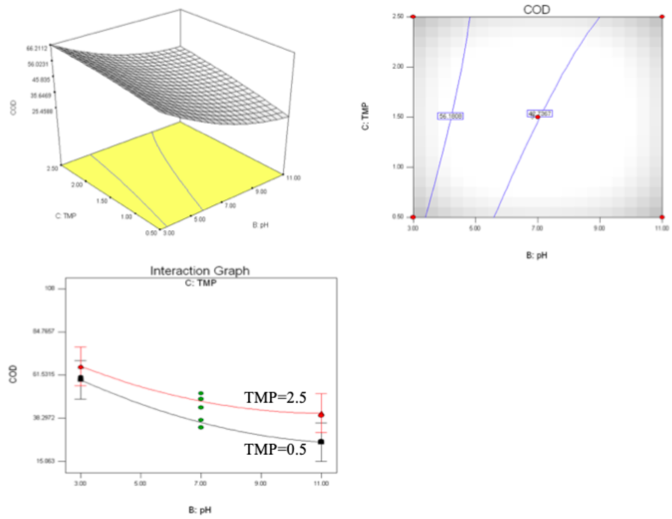

Figure 5. The effect of the Transmembrane pressure and pH on COD value of POME treatment (a) 3-D, (b) contour, (c) interaction

factor was affected on the performance of POME treatment process which reported for other treatment process earlier (Ba-abbad et al., 2013, 2012).

3.4 Model validation of POME optimization conditions

To check of Box Behken model validation is very important to produce more stable model for POME treatment process. The main important term use to investigate the validity of the predicted model is desirability function (Box et al., 2005). The maximum value of the desirability function (D=1.000 or D≈1.000) was selected to optimize of POME treatment process. To determine factors to produce lower value of COD within the experiment process, the optimization criteria of RSM had five options: none, maximum, minimum, target and within range as shown in Table 4.

The ANOVA analysis of this model, all factors of POME concentration, pH and Transmembrane pressure were significant which each factor contributed affecting within their range option. The minimum value of COD according to the factor as POME concentration =28.30 vol. %, pH =10.75 and Transmembrane pressure= 0.69 kPa was to be 24.137 mg/l as shown in Fig.6.

To investigate the model validity predicted by Box Behken design, a triplicate of experimental runs under optimum conditions of all factors was achieved. The average COD results was 25.763 mg/l was obtained which in good agreed with the predicted by model. Accordingly, the optimization results (comparison between the predicted and experimental results) showed more effective and reliable to apply the Box Behken design for reduces of COD value which was attributed to a good interaction between the selected factors with their ranges.

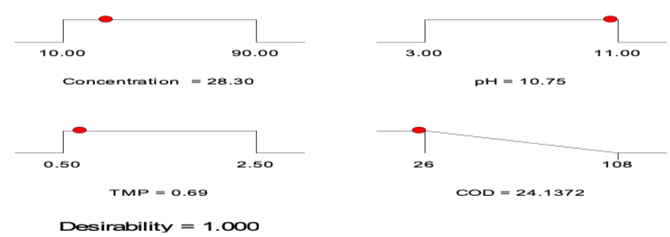

Figure 6. Predicted COD, as obtained from the RSM based on Box Behken design under optimal conditions

4. CONCLUSIONS

In this study, the POME treatment process using reverse osmosis membrane was optimized by Appling the response surface method (RSM) based on the Box Behken design. Effects of POME treatment process factors with their interactions were estimated by ANOVA. Good coefficients of R^2 as 0.9692, R^2_{adj} as 0.9297 of the predicted model were obtained. Optimum conditions for lower value of response COD were POME concentration =28.30 vol.%, pH = 10.75 and Transmembrane pressure= 0.69 kPa. The predicted value of COD under optimal condition was 25.763 mg/l which showed in good agreement with the predicted by model as 24.137 mg/l. This result evidences to support the validity of the model was created by Box Behken design which also showed as suitable way for optimizing the conditions POME treatment process in future.

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Table 4. Optimization details of POME treatment process

term	target	Lower limit	Upper limit	Lower weight	Upper weight
POME conc. (vol.%)	In the range	10	90	1	1
pH	In the range	3	11	1	1
Transmembrane pressure (kPa)	In the range	0.5	2.5	1	1
COD(mg/l)	Minimum	26	108	1	1

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