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Application of Response Surface Method in Reverse Osmosis Membrane to Optimize BOD, COD and Colour Removal from Palm Oil Mill Effluent

Muhammad Said[#], Muneer M. Ba-Abbad[*], Siti Rozaimah Sheikh Abdullah[*], Abdul Wahab Mohammad[*]

[#] *Department of chemistry, Sriwijaya University, Indralaya 30662, Sumatera Selatan, Indonesia*
E-mail: msaidusman@unsri.ac.id

[*] *Department of Chemical and Process Engineering, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia*
E-mail: muneer711@gmail.com; rozaimah@ukm.edu.my; drawm@ukm.edu.my

***Abstract*— Palm oil mill effluent (POME) is typically non-biodegradable and has high concentration of organic matter that represented as COD, BOD and Colour values. The correlation of concentration and pH of POME, and Trans membrane pressure (TMP) of Reverse Osmosis (RO) membrane was optimized by response surface method using a second order polynomial model with central composite design (CCD) which is a part model of response surface method (RSM) in Design-Expert® software. The main limits that influenced the parameters removal i.e. concentration of POME, pH of solution and transmembrane pressure were empirically determined at laboratory level and successfully optimized using RSM. The best conditions were determined from 3D response surface and 2D contour graphs i.e. 10.05% of POME concentration at pH 3.0 and TMP 0.50 kPa to yield the last values of COD, BOD and Colour i.e. 24.1372 mg/L, 24.33 mg/L and 11.76 PtCo, respectively. The results show that the response surface method effective to reduce the number of experiment.**

***Keywords*— POME; RO membrane; RSM; CCD; pollutant removal**

INTRODUCTION

Palm oil mill effluent (POME) is a wastewater produced from the palm oil mill industry. The extraction of crude palm oil from the fruit requires a huge amount of water. It is estimated to produce 1 tons of crude palm oil, is required 5-7.5 tons of water. Unfortunately, 50% of the water will end up as POME. This wastewater, if not handled properly, will contaminate the environment [1]. In Malaysia, the common methods used to handle POME are integrated anaerobic and aerobic ponds. This method requires large area and long residence times [2]. To solve the above problems, the researchers tried to find a variety of new methods including biological [2-4] and physical-chemical [5-9]. The detail information about POME treatment is explained by Wu et al. [10]. In recent years, membrane technology has been applied for POME tertiary treatment to improve the effluents quality.

The use of membrane in treating POME has been a very interesting issue among researchers. Membrane filtration has the capability to produce clear water in a relatively short amount of time, small area and energy consumption. The membrane system has been shown to be able to significantly reduce the BOD, COD, and, TSS to acceptable levels set by regulatory agency [11-13].

In carrying out an experiment containing many variables, the most commonly used technique is to change one variable within a specified time while the other variables are constant. This technique is not practical and cannot describe the interactions among variables [14]. Therefore, it is required a tool to optimize the response of the overall variables simultaneously in one time. Response Surface Methodology (RSM) was one of famous tool used in collecting data and mathematical modelling that can be used to determine the effect of several independent variables on the response. RSM is very useful to increase the accuracy of existing design processes [15].

The aims of the present work are to test Reverse Osmosis (RO) membrane ability to remove the important variable in POME. The experiments were planned by using the RSM. Three optimization processes will do i.e. determination of mathematical model coefficients, prediction of response and model validation. In order to analyse the process, three independent variables, viz. concentration of POME, pH of solution and trans membrane pressure and three dependent variables, viz. BOD, COD and Colour were studied.

MATERIALS AND METHODS

A. Materials

Raw POME was collected from a local palm oil mill in Selangor, Malaysia. No chemical was added to raw POME. The samples were stored in cold room at 4oC. Before used, raw POME was pre-treated using Adsorption and Ultrafiltration membrane to reduce the suspended solids. The characterisation of the feed sample and permeate was performed (BOD, COD, TSS, and colour) and analysed using a DR/2010 portable data logging spectrophotometer (HACH, USA). The compositions of raw and pre-treated POME are summarized in Table 1.

TABLE I
COMPOSITION OF RAW AND PRETREATED POME

Parameter	Raw POME	Pretreated POME
BOD (mg/L)	482	174.67
COD (mg/L)	2100	244.67
TSS (mg/L)	23300	7.00
Colour (PtCo)	7067	1263.33

B. Methods

The three stages of POME treatment were Adsorption with Montmorillonite, Filtration with UF membrane and lastly RO membrane. The 10 L of raw POME was fed to adsorption column using dosing pump. The flow rate keep constant at 2 ml/min. POME fed at the top and flows downward along gravity, and exits at the bottom of the column. This process takes approximately 2 hours. POME, most of them has been absorbed by montmorillonite, will be poured into the UF membrane for further treatment. The ultrafiltration pretreatment were carried out in cross flow unit with a hollow fiber membrane. The feed solution entered the module at one side while the permeate exits at the other side and retentate was recycled to the feed reservoir. Permeate from the UF membrane also simultaneously serves as feed for the RO membrane.

On the RO membrane, optimization process is done with the assistance of Response Surface Method (RSM). In the stage of RO membrane, the feed was pumped through a spiral wound (RE2012-LPF, CSM filter) and recycled back to the UF feed reservoir. Sequential backwashes were automatically operated. The permeate flux was collected and measured gravimetrically with an electronic balance. Permeate were analyzed to determine the concentration of parameters. The detail experiment setup is shown in Fig. 1.

C. Statistical design of experiments

In this study, the central composite design (CCD) was used to design the experiments (DOE) using Design Expert software version 6.0. The independent variables are notated as X and the dependent variables as Y. Determination of the DOE aims to cut the number of experiments and get the best response (Y) as the result of interaction of all the factors (X) involved. In this study, the responses were concentration of BOD, COD and Colour while the factors were concentration of POME, pH of solution and Trans membrane pressure.

After conducting the experiment, the coefficients of the polynomial model were determined using the following equation:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1,i<j}^{k-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j \quad (1)$$

where, βo is the constant coefficient, βi is the linear coefficients, βii is the quadratic coefficients, and βij is the interaction coefficients. Three dimensional plots and two dimensional contour plots were obtained based on the effect of the interaction of the two factors. From these plots, the optimum region can be identified. The experimental and results data are shown in Table 2.

TABLE II
DATA OF EXPERIMENT AND RESULTS OF RSM

Run No.	Conc. of POME (%)	pH solution	TMP (kPa)	Response		
				BOD (mg/L)	COD (mg/L)	Colour (PtCo)
1	90.00	11.00	2.50	53.67	74	32
2	90.00	11.00	0.50	52	65.67	31.33
3	-17.27	7.00	1.50	24.33	33	8
4	10.00	11.00	0.50	29.33	30	16.33
5	50.00	7.00	1.50	34.33	48.67	25
6	50.00	13.73	1.50	36.67	40	22.67
7	90.00	3.00	2.50	57	108	34.33
8	10.00	11.00	2.50	29.33	33.33	12
9	10.00	3.00	2.50	27.67	36.67	14.67
10	90.00	3.00	0.50	52.67	96	30
11	50.00	7.00	3.18	34.67	60.67	29.33
12	50.00	7.00	1.50	32.33	52.67	27.33
13	10.00	3.00	0.50	24.33	32	10
14	50.00	7.00	1.50	32.67	44	29.33
15	50.00	7.00	1.50	32	47.33	25.33
16	117.27	7.00	1.50	66.67	107	34.67
17	50.00	0.27	1.50	36.67	60.67	23.67
18	50.00	7.00	1.50	32.67	44.07	26.67
19	50.00	7.00	-0.18	32	36.67	25
20	50.00	7.00	1.50	33.67	51.67	25.33

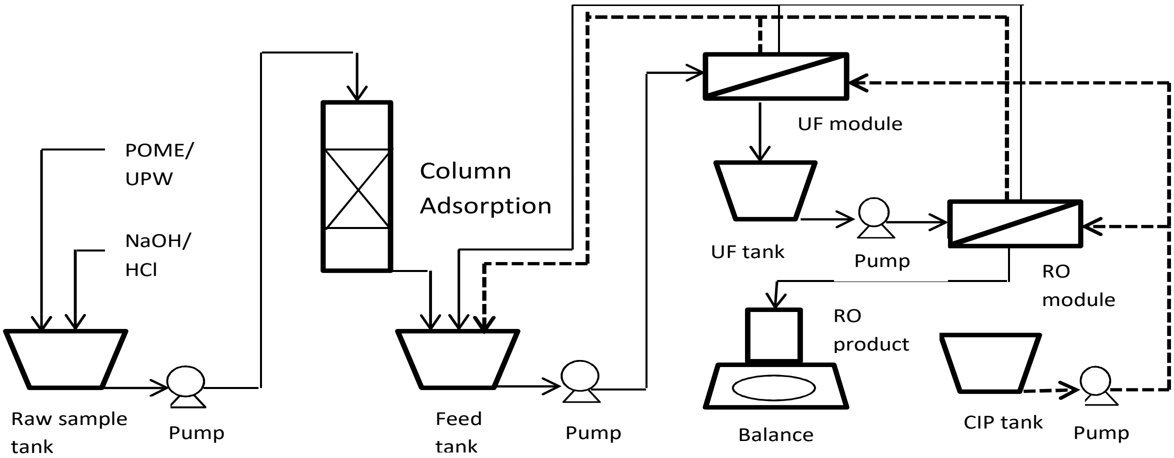


Fig.1. Detail experiment setup

RESULTS AND DISCUSSIONS

A. Model Fitting of CCD Design

The effects of all the factors on the responses of COD, BOD, and Colour were investigated using the quadratic polynomial model. The final models of optimization were estimated based on the experimental results of the CCD design with the respective coefficients as given below:

COD = 27.02461 + 0.36089* Conc. - 0.55800* pH + 1.90104* TMP + 5.19211E-003*Conc.^{2} + 0.084672*pH^2 + 0.76609*TMP^2 - 0.046086*Conc.*pH +0.038531*Conc.*TMP - 0.15656*pH*TMP

BOD = 24.71650 + 0.065167*Conc. - 0.75892*pH + 0.28596*TMP + 3.02637E-003*Conc.^{2} + 0.10752 * pH^2 + 0.54121*TMP^2 - 8.32812E-003*Conc.*pH + 8.31250E - 003*Conc.*TMP - 0.18750*pH*TMP

Colour = 15551 + 0.35167*Conc. + 2.03524*pH + 98596*TMP - 1.29252E-003*Conc.^{2} - 088704 *pH^2 - 6.82306E-003*TMP^2 - 64063E-003*Conc.*pH + 014562*Conc.*TMP - 0.39563*pH*TMP

The predicted models that were created by the CCD design involved all the coefficients which were shown as a quadratic regression for all responses. All responses of COD, BOD, Colour using the model at each point of the experiment are listed in Table 2 as a comparison between the theoretical (predicted) and the experimental results. The analysis of variance (ANOVA) results of the CCD model is given in Tables 3,4 and 5 for each response individually. The degree of significance of the model and all the factors (X1, X2, and X3) are presented according to the P-value, where a value that is less than 0.050 is considered to be significant, and any other value that is greater than 0.050 is not significant.

TABLE III
ANOVA RESULTS FOR QUADRATIC MODEL OF COD

Source	Sum of squares	Degree of freedom	Mean square	F-value	P-value
Model	10872.70	9	1208.08	54.74	< 0.0001
X_1	8272.66	1	8272.66	374.87	< 0.0001*
X_2	798.59	1	798.59	36.19	0.0001*
X_3	345.52	1	345.52	15.66	0.0027*
X^2_1	994.56	1	994.56	45.07	< 0.0001
X^2_2	26.45	1	26.45	1.20	0.2993
X^2_3	8.46	1	8.46	0.38	0.5497
X_{12}	434.98	1	434.98	19.71	0.0013
X_{13}	19.00	1	19.00	0.86	0.3753
X_{23}	3.14	1	3.14	0.14	0.7140
Residual	220.68	10	22.07		
Lack of fit	153.09	5	30.62	2.26	0.953**
Pure error	67.59	5	13.52		
Total	11093.38	19			

*Significant at < 0.05% level; ** Not significant, R^2= 0.9801, R^2_{adj} =0.9622, Std. Dev.= 4.70, Mean=55.10, C.V= 8.53, Adeq Precision=24.961

All models of COD, BOD and Colour were most significant which the p-value much lower than 0.05 (0.0001).The significant factor is the concentration (X1) on all of responses then the TMP (X3) in COD, BOD as well as the pH (X2) only affected in COD. Other important terms are the accuracy and variability of the models, which can be evaluated according to the R-Squared (R2) value which is between 0 to 1. If the value closer to1 means a better prediction of the response [16, 17]. The R2 of the CCD models showed a higher value, which were 0.9801, 0.9936 and 0.9745 for COD, BOD, and Colour, respectively.

TABLE IV
ANOVA RESULTS FOR QUADRATIC MODEL OF BOD

Source	Sum of squares	Degree of freedom	Mean square	F-value	P-value
Model	2659.67	9	295.52	171.42	< 0.0001
X_1	2265.26	1	2265.26	1314.02	< 0.0001*
X_2	0.52	1	0.52	0.03	0.5956**
X_3	14.01	1	14.01	8.12	0.0172*
X^2_1	337.90	1	337.90	196.01	< 0.0001
X^2_2	42.65	1	42.65	24.74	0.0006
X^2_3	4.22	1	4.22	2.45	0.1487
X_{12}	14.20	1	14.20	8.24	0.0167
X_{13}	0.88	1	0.88	0.51	0.4902
X_{23}	4.50	1	4.50	2.61	0.1372
Residual	17.24	10	1.72		
Lack of fit	13.37	5	2.67	3.46	0.0997**
Pure error	3.87	5	0.77		
Total	2676.91	19			

*Significant at < 0.05% level; ** Not significant, R^2=0.9936, R^2_{adj}=0.9878, Std. Dev.=1.31, Mean=37.73, C.V= 3.48, Adeq Precision=47.488.

On the other hand, the Adj R-Squared (R2adj) coefficient was also found to be 0.9622, 0.9878 and 0.9515 for COD, BOD, and Colour, respectively which was close to the R2 value for each response. These values indicated a good correlation among the factors of the process using the CCD design.

TABLE V
ANOVA RESULTS FOR QUADRATIC MODEL OF COLOUR

Source	Sum of squares	Degree of freedom	Mean square	F-value	P-value
Model	1167.56	9	129.73	42.38	0.0001
X_1	1045.88	1	1045.88	341.66	< 0.0001*
X_2	0.070	1	0.070	0.023	0.08828**
X_3	11.67	1	11.67	3.81	0.0795**
X^2_1	61.63	1	61.63	20.13	0.0012
X^2_2	29.03	1	29.03	9.48	0.0117
X^2_3	6.709 E-004	1	6.709 E-004	2.192 E-004	0.9885
X_{12}	2.71	1	2.71	0.89	0.3686
X_{13}	2.71	1	2.71	0.89	0.3686
X_{23}	20.03	1	20.03	6.54	0.0285
Residual	30.61	10	3.06		
Lack of fit	16.90	5	3.38	1.23	0.4122**
Pure error	13.71	5	2.74		
Total	1198.17	19			

*Significant at < 0.05% level; **Not significant, R^2=0.9745, R^2_{adj} =0.9515, Std. Dev.=1.75, Mean=24.15, C.V=7.24, Adeq Precision=23.792

Additionally, adequate precision is a term used for evaluating the predicted range of responses on the associated error, where a value that is greater than 4 is required to support the fitness of the model which 24.961, 47.488 and 23.792 respectively for COD, BOD, and Colour.

B. Adequacy Check of the Model

All the plots for investigating the optimization of COD, BOD, Colour responses using the CCD design are given in Fig. 2. The studentized residuals with normal probability show that all points were close to the line for COD, BOD and Colour, indicating there were no obvious problems with the normality of the design. The general effect of the plots between the studentized residuals and the predicted COD, BOD and Colour were the random scattering of all the points rather than a funnel-shaped pattern, which confirms that the response had an original observation of variance and that there was no problem with the response variable [18].

The values of the studentized residuals of COD, BOD, Colour responses were almost at intervals of between -3.5 to +3.5, and the observed responses value were not considered for any value beyond these values. These models had a studentized residual value that was lower than ±3.5, which gives a good fitting of the models to the response surface. The outlier of the experiment runs clearly showed that all the points in the range of the outlier had a good distribution for the CCD design. The real value of the COD, BOD, and Colour from the experimental runs was nearly the same as the value predicted by the model, which was evaluated by approximating the terms of R^2 and R^2_{adj}.

C. Response Surface Plotting and Optimization of COD, BOD and Colour responses

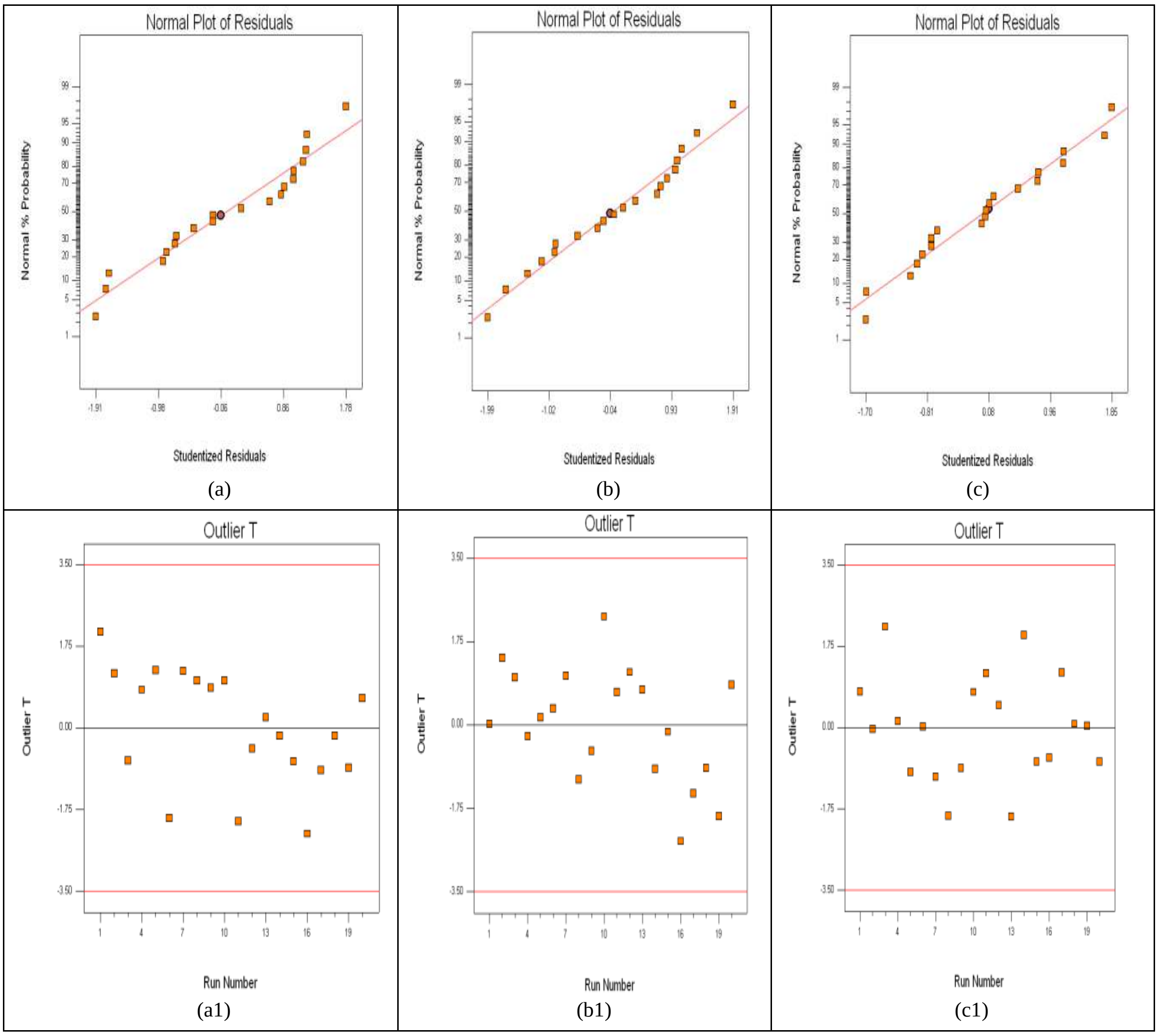
The effects of the concentration, pH and the TMP on the COD, BOD and Colour were evaluated by the RSM based on a CCD design. The 3D response surfaces graphs were used to illustrate the effects of the interaction between the two factors of the concentration, pH and the TMP, on performance of COD, BOD and Colour as shown in Fig.3. It can be observed that the efficiency of parameter removal was determined by the low value of COD, BOD and Colour with a lower concentration as below 30% and pH of 3 where the optimum value of COD, BOD and Colour were 31.66 mg/L, 24.51mg/L and 13.64 PtCo, respectively. It can be observed that the parameter value decreased slightly as the pH increased from 3 to 11. The main reason for this behaviour was the pH changing the character of the impurities.

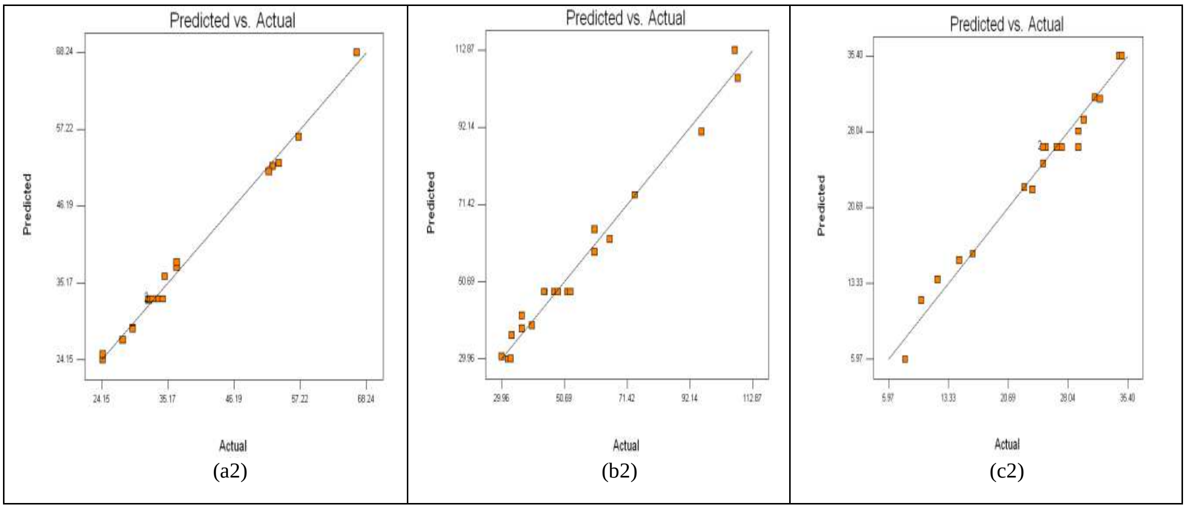
The higher pH means the charge of the impurities will be equal to the charge on the surface of the membrane. The similarity of these charges will be made into a more hydrophilic nature of the membrane so that the impurities do not stick to the surface and is trapped to the bulk solution. In contrast, at the lower pH, the POME has the same charge to membrane hence it increases the attraction force between POME and membrane. The particle will easily attach to membrane surface and then pass through the pores [19].

The effect of concentration and pH on COD, BOD and Colour was observed as given in Fig.3a, 3b, 3c, respectively. The lowest value of the COD, BOD and Colour removal were obtained

The effect of TMP and concentration on COD, BOD and Colour was shown in Fig.4. It is clearly shown that both variables have higher effect on Colour. It is also observed according to the p-value of interaction from Table 5. The table shows the effect of concentration on the COD, BOD and Colour removal. It was found that the lowest value was occurred at concentration lower than 30% and TMP lower than 1.5 kPa. Similar to the previous phenomenon, the parameter values were found to be increase as the concentration increased. It is not surprising when the concentration increased significantly, more solid particles contained in the solution. This phenomenon is probably due to the smaller concentration of POME as indicator of the presence of impurities. In addition, the increase in Trans membrane pressure makes bigger chance of the small particles in the solution to pass through the membrane pores. Wu explains this phenomenon as being due to the absorption of macromolecules on the membrane surface to form a thick cake layer. The cake layer not only retained the inorganic but also the organic molecule. Therefore, the parameter value after filtration will significant decreased [20].

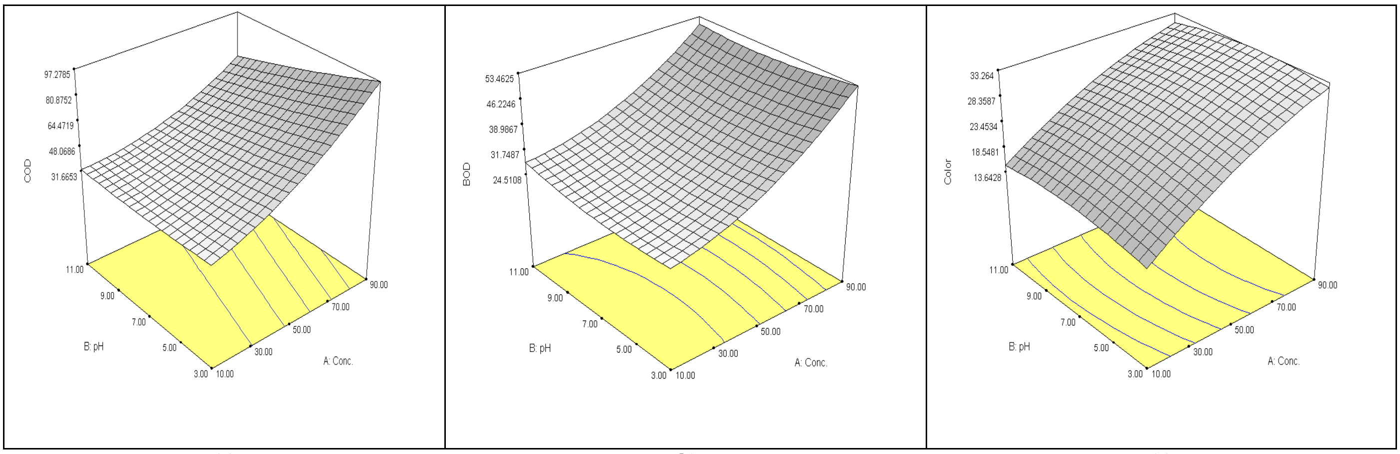
COD BOD Colour





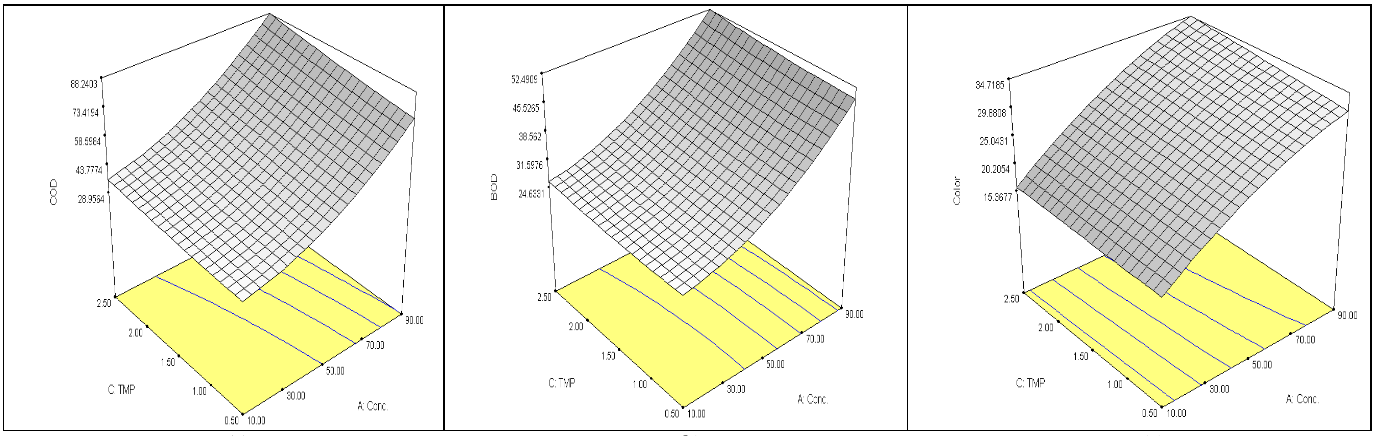
(a2) (b2) (c2)

Fig.2. All diagnostic plots of optimization of COD, BOD, and Colour using CCD design:
Normality (a, b, c); Outlier T (a1, b1, c1); Actual and predicted (a2, b2, c2).



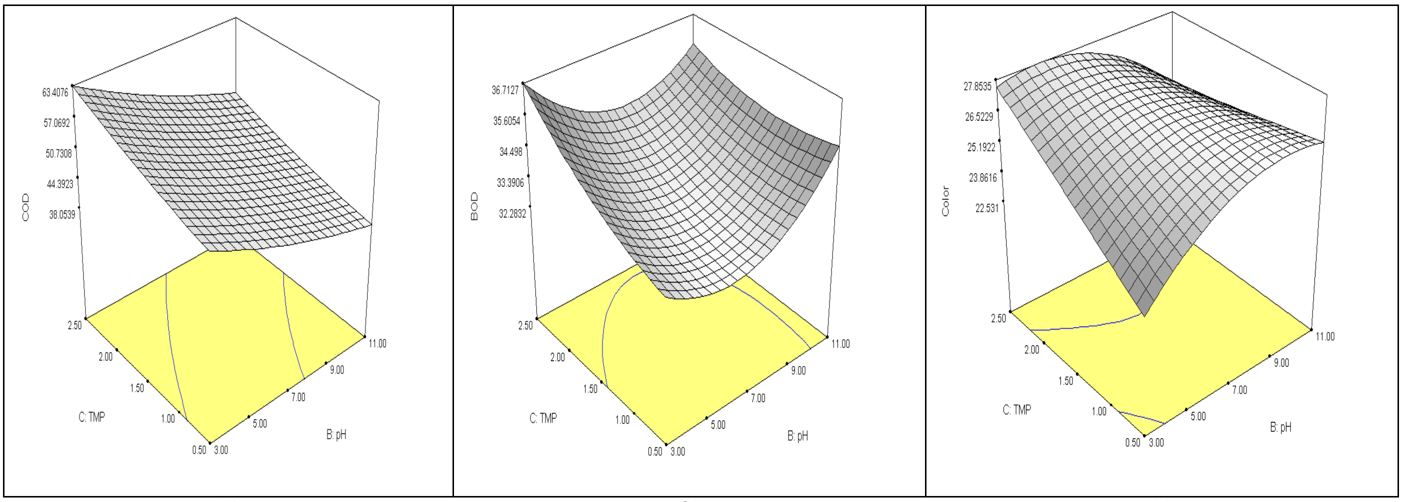
(a) (b) (c)

Fig.3. The parameter removal as the effect of Concentration and pH on (a) COD, (b) BOD and (c) Colour



(a) (b) (c)

Fig.4. The parameter removal as the effect of Concentration and TMP on (a) COD, (b) BOD and (c) Colour



(a) (b) (c)

Fig.5. The parameter removal as the effect of pH and TMP on (a) COD, (b) BOD and (c) Colour

Finally, effects of the interaction between the pH and TMP on the COD, BOD and Colour were observed differentially for each response as shown in Fig. 5a, 5b, 5c, respectively. By increasing of TMP from 0.5 to 2.5, the COD value was increased to higher value. Increasing the pressure means increasing the concentration polarization and thus increasing also the number of collisions between particles. Collisions between particles will force the particles to enter the pores of the membrane. Since the size of the particles was larger than the pore size of the membrane, this causes the deposition of particles

on the membrane surface and ultimately forms a cake layer. This phenomenon indicates that the accumulation of particles on the surface and in the pores of the membrane had reached the highest stage.

In the same time, by the effects of interaction between the TMP and pH on the COD, it is found that the decreasing of COD value reached when the TMP and pH increased from 0.5 to 2.5 and 3.0 to 11.0 respectively. This effect of TMP and pH on the COD value was considered as liner. On the other hand, effects of pH and TMP on the BOD show as polynomials which at middle of pH value, lower values of BOD were obtained Fig. 5b. The liner effects of TMP on BOD value when increasing the TMP, BOD values was increased which almost at same linear effect by pH and TMP on the COD. However, different behaviour of pH effects on BOD value which at lower pH at 30.0 to 7.0, the BOD values deceased and then increased from 7.0 to 11.0. The same effects of pH on the COD and BOD on the other process performance was reported [21].

The effects of the pH and TMP on the Colour show also different compared to COD and BOD as given in Fig. 5c. The liner effects of TMP on Colour were observed which increasing the TMP leads to Colour increased at all TMP values. However, by increasing the pH from 3.0 to 7.0, the Colour was increased and then constant at lower value of TMP (0.5). In addition, at higher value of TMP (2.5) the value of Colour was decreased clearly which confirm the effects of interaction between the pH and TMP (X_{23}) and the p-value was found to be lower than 0.05 as 0.0285 in Table 5.

D. Model validation of optimization conditions

For the optimization of all the factors to produce a lower COD, BOD and Colour, the in range and the minimum options were selected for all the main factors (concentration, pH and TMP) and response (COD, BOD and Colour), respectively. The predicted conditions for all the factors for a lower response are illustrated in Fig. 6.

Based on desirability option, the simulated figures showed that the best value of COD, BOD and Colour were 29.974 mg/L, 24.33 mg/L and 11.76 PtCo, respectively. The best values were obtained at concentration of 10.05%, pH of 3.0 and TMP of 0.50 kPa. By applying the best values, additional experiment was done. The experimental results were much closer to the predicted by the model. It proves the CCD design is one of the great tool to get the best working conditions and this was attributed to the good interaction between the all selected factors.

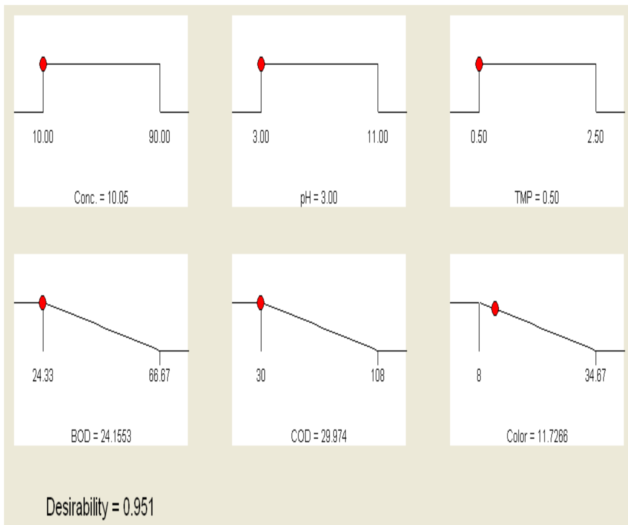


Fig.6. Predicted COD, BOD and Colour as obtained from the RSM based on CCD design under optimal conditions.

Conclusion

To optimize the process of reverse osmosis membrane treatment, is applied the response surface method (RSM). Factors that affect the removal of parameters are POME concentration, solution pH and Trans membrane pressure. Overall, the study showed the optimum removal of BOD, COD and colour. Based on the response surface and contour plots can be seen that the optimum of parameter removal will be achieved when the concentration of POME was 10.05%, pH of POME was 3.0 and the TMP was 0:50 kPa. The final values of COD, BOD and Colour were 29.974 mg/L, 24.33 mg/L and 11.76 PtCo, respectively.

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