Desalination and Water Treatment
www.deswater.com
doi: 10.1080/19443994.2014.919607

55 (2015) 615–623
July

Taylor & Francis
Taylor & Francis Group

Application of response surface methodology (RSM) to optimize operating conditions during ultrafiltration of oil-in-water emulsion

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Received 7 October 2013; Accepted 26 April 2014

ABSTRACT

In this research, the capability of ultrafiltration hollow fiber polyvinylidene fluoride membrane by polyaluminum chloride in pretreated oil-in-water emulsion was studied. Central composite design and response surface method were applied to optimize the operating variables: transmembrane pressure (TMP) and velocity. Quadratic models developed for the three responses (permeate flux (PF), turbidity removal, and chemical oxygen demand (COD) removal) indicated that the optimum PF of 50 L/m^2 h, turbidity removal of 79%, and COD removal of 77% would be achieved after pretreatment at a TMP of 1 bar and velocity of 3 m/s. The simulated values obtained from the statistical model were in agreement with the experimental results.

Keywords: Ultrafiltration; Oil-in-water emulsion; PAC; RSM; Optimization

1. Introduction

Wastewaters containing an emulsion of oil in water are produced in many industries, including the oil production, petrochemical, metal, and food industries. These wastewaters are a complex mixture of water, oil, and additives, such as emulsifiers, corrosion inhibitors, antifoaming agents, and biocides. To reuse this wastewater or to return it to natural waterways, this wastewater must be treated [1].

Membrane processes are considered one of the techniques for the treatment of oily wastewaters. Experience shows that these techniques are successful in terms of the quality of produced permeate, lower cost, easy operation, and in some cases, the ability to reduce contaminants below existing pollution limits [2,3].

Even though membrane processes such as microfiltration and ultrafiltration (UF) are effective, they still suffer from fouling and its consequences [4].

In order to achieve higher separation efficiencies, several techniques have been integrated with membrane processes, which are chemical and physical pretreatment methods such as coagulation, adsorption, and oxidation. Integrating membrane with a coagulation process will increase the performance of both coagulation and membrane processes. Pretreatment with coagulants reduces organic and colloidal matter in oily wastewater. Organic content and colloidal matter play an important role in the fouling phenomenon of membrane in oily wastewater filtration [5–7].

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In this integrated process (coagulation + membrane), several parameters such as coagulant dosage, pH of coagulation, and membrane operating conditions, e.g. pressure and flow velocity, play a major role. Optimization of these parameters could result in the best permeate quality and quantity, at an acceptable total cost.

Conventional multi-factor optimization by varying one factor and fixing the others is time- consuming. Furthermore, if the interaction among variables is ignored, reaching optimum conditions is difficult [8].

A statistical method, response surface methodology (RSM), has been proposed as a solution to consider both the influences of individual factors and their interactive influences [8,9]. RSM is an empirical statistical modeling technique for designing experiments, evaluating the effects of several factors, building models, and searching optimum conditions for desirable response, with a limited number of planned experiments [8].

In the present study, the statistical design of experiments (DOE) and RSM are applied to optimize the process conditions of oily wastewater treatment by hollow fiber polyvinylidene fluoride (PVDF) membrane.

RSM, coupled with central composite design (CCD), is selected to study simultaneously the effects of a two process variable, transmembrane pressure (TMP) and velocity, on three responses, permeate flux (PF), turbidity removal, and chemical oxygen demand (COD) removal. The optimum value of the variables is obtained in order to maximize PF, turbidity removal, and COD removal. The experimental results are compared with the simulated values obtained from the proposed models.

2. Materials and methods

2.1. Chemicals and materials

Polyaluminum chloride (PAC) was used as a coagulant in this work. PAC was obtained from Youbang Co., China. HCl and NaOH, supplied by Merck, Germany, were used for pH adjustments.

2.2. Analytical methods

pH and turbidity were analyzed using a pH meter (WTW Co., Germany) and a turbidity meter (Hach Co., USA), respectively. The oil concentrations were determined using a UV spectrophotometer (Hach Co., USA). COD of the wastewater was analyzed according to the standard method [10].

2.3. Preparation of model solution

Oil-in-water emulsion was prepared from a commercial cutting fluid (Behran Oil Co., Iran) used in machining processes. Cutting fluid was added to tap water at a concentration of 0.5 v%. Cutting oil was chosen as the base for making the emulsion as it emulsifies easily in water and remains stable for a long time [11]. Table 1 shows the characteristics of model solution.

2.4. Coagulant dosing and pH selection

Step 1: Optimum dosage of coagulant: varying doses (500–700 mg/L) of the coagulant (PAC) are added to the model solution at pH values between 7.5 and 9.5. The experiments were carried out in a standard jar test, with six paddle stirrers (Phipps & Bird, USA). The best dosage was selected with respect to turbidity and COD removal.

Step 2: The optimum dose of PAC and pH obtained from Step 1 was used for pretreatment of oily wastewater followed by UF.

2.5. Experimental set-up and operation

The membrane used in the experiments was a UF hollow fiber PVDF membrane (Parsian Pooya Polymer Co., Iran) with a cut-off of 100 kg/mol, internal diameter of 0.5 mm, external diameter of 0.8 mm, and surface area of 1.5 m^2. The flow in this membrane was from outside to inside.

Fig. 1 shows the experimental set-up. The model solution in feed tank 2 was pumped to a hollow fiber membrane module. The required pressure—TMP: 1–2.8 bar and velocity: 1–4.2 m/s—were achieved by passing part of the flow to the feed tank. In order to minimize the error caused by feed concentration variation, both permeate and concentrate return to feed tank 2. Each filtration in the designed experimental plant was performed for 30 min. Operating conditions as well as the weight of permeate were recorded during the experimental time.

Table 1
Characteristics of model solution

Parameters	Units	Values
pH	N/A	8.5
Turbidity	NTU	3,200
COD	mg/L	10,200
Oil content	mg/L	2,700

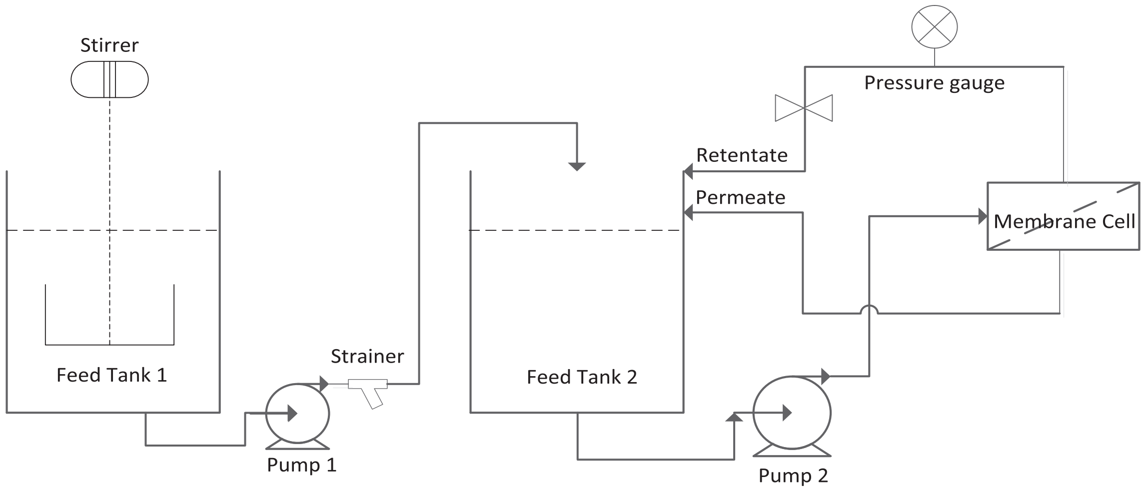

Fig. 1. Schematic diagram of the experimental setup.

2.6. Design of experiments

DOE was used to optimize process variables in this study. Various methods are available in performing DOE. One is RSM, a statistical method that uses quantitative data from appropriate experiments to determine regression model equations and operating conditions [12]. RSM is also a collection of mathematical and statistical techniques for modeling and analysis of problems in which a response of interest is influenced by several variables [13]. This method is used for fitting a model and helps to optimize the effective variables with a minimum number of experiments, as well as to analyze the interaction among the variables [14]. Analysis of variance (ANOVA) for the model was also carried out to establish its statistical significance.

Design-Expert® 7 software was used for the statistical DOE and data analysis. In this study, CCD and RSM were applied to optimize the two most important operating variables: TMP and velocity. According to the factorial portion, this gives a full factorial design with all parts of the factors at two levels (high, +1 and low, –1). The center point (coded level 0) is the center point between the high and low levels, and is duplicated thrice. The axial points for all but one factor are set at level 0 and one factor is set at the outer value based on an α value of 1.414. The processing parameters involved in the study are shown in Table 2. The generated experimental plan, using Design-Expert® 7 software, is shown in Table 3.

In order to obtain the optimum TMP and velocity, the effects were studied on three dependent parameters, i.e. PF, turbidity removal, and COD removal.

The empirical model for predicting optimal conditions can be expressed according to Eq. (1):

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_{12} AB + \beta_{11} A^2 + \beta_{22} B^2 \quad (1)$$

where Y is the predicted response, and A and B are the coded forms of TMP and velocity, respectively. The term β_0 is the offset term, β_1 and β_2 are the linear terms, β_{11} and β_{22} are the quadratic terms, and $\beta_{12}$is the interaction term. The above model was used in the present work and tested with ANOVA.

The ANOVA result was used for graphical analyses of the data to obtain the interaction between the responses and the variables [8,15]. Three-dimensional plots were obtained based on the effect of the two

Table 2
Experimental independent variables

Variables	Factor code	Level and range (code)				
		−1.414	−1	0	+1	+1.414
TMP (bar)	*A*	0.7	1.0	1.7	2.5	2.8
Velocity (m/s)	*B*	1.0	1.5	2.6	3.7	4.2

Table 3
CCD for the study of two experimental valuables in code units

Run no.	Experimental design		Results		
	A: TMP (code)	*B*: Velocity (code)	PF (L/m^2 h)	Turbidity R (%)	COD R (%)
1	0.7 (–1.414)	2.6 (0)	40	82	78
2	1.7 (0)	1.0 (–1.414)	32	85	82
3	1.7 (0)	2.6 (0)	48	74	79
4	2.5 (+1)	3.7 (+1)	64	72	68
5	2.8 (+1.414)	2.6 (0)	52	70	69
6	2.5 (+1)	1.5 (–1)	42	68	78
7	1.7 (0)	2.6 (0)	50	73	81
8	1.7 (0)	2.6 (0)	47	75	78
9	1.7 (0)	2.6 (0)	49	77	80
10	1.7 (0)	4.2 (+1.414)	68	69	68
11	1.0 (–1)	3.7 (+1)	58	80	73
12	1.0 (–1)	1.5 (–1)	33	88	82
13	1.7 (0)	2.6 (0)	49	76	79

Note: Permeate flux (PF).

factors and their levels. In addition, the simultaneous interaction of the two factors on the responses was studied from these plots.

3. Results and discussion

3.1. Determination of optimum conditions of coagulation

Pretreated oily wastewater with low turbidity and COD values is the main objective to be achieved through the coagulation process. RSM was used for optimization of the coagulation process [16]. A 2^2 full-factorial CCD was chosen to explain the effect and interaction of two factors: PAC dosage and pH (code factors *a* and *b*). Table 4 shows the models in terms of coded factors and also illustrates other statistical parameters. Results demonstrate that the correlation coefficient R^2 values for turbidity removal and COD removals are 0.964 and 0.943, respectively, indicating an acceptable fitting of the quadratic models to experimental data. The values of the adjusted R^2 for these two models are 0.928 and 0.864, which could confirm the accuracy of the quadratic models. In addition, two models are highly significant, implied by the high F-test values (27.08 and 11.9) with low probability values (Prob > F). The lack-of-fit (LOF) F-test depicts the variation of the data around the fitted model. A model can be considered reproducible if its coefficient of variance (CV), the ratio of the standard error of predicted value to the mean value of the observed response is not greater than 10% [8]. In this work, the CV values of two models are 2.86% and 2.71%, respectively. Adequate precision (AP) compares the range of the predicted values at the design points to the average prediction error. In this work, the AP ratios of two models (12.56–9.684) are greater than 4, indicating adequate model discrimination [8].

With regard to the aforementioned ANOVA results, the optimum values of selected variables were obtained by solving the equations. The turbidity of 240 NTU and COD value 873 mg/L can be obtained at the optimum values of coagulant dosage and pH at 617 mg/L and 8.2 mg/L, respectively.

Table 4
ANOVA results for response parameters

Response	The model in terms of code factors	R^2	R^2 adjusted	F-value	Prob > F	Coefficient of variance, CV (%)	F-value for lack of fit	Adequate precision, AP
Turbidity removal	$92.47 + 1.22a + b + 0.25ab - 15.67a^2 - 3.67b^2 + 1.25a^2b$	0.964	0.928	27.08	0.0004	2.86	0.5	12.56
COD removal	$87.49 + 7a - 5b + 0.25ab - 7.93a^2 - 3.93b^2 + 6.75a^2b - 4.75ab^2$	0.943	0.864	11.9	0.007	2.71	1.41	9.684

3.2. Statistical analysis of UF process

The relationship between the two variables (TMP and velocity) and the three important process responses (PF, turbidity removal, and COD removal) for the UF process was analyzed using RSM. Table 3 shows the values of the three responses against each experiment conducted as per CCD. The CCD was used to develop correlation between the two variables (factors A and B) to three responses ($Y1$, $Y2$, and $Y3$). Experimental runs at the center point were used to check reproducibility and to estimate an experimental error. PF ($Y1$) varied from 32 to 68 L/m^2 h, and turbidity removal ($Y2$) was in the range of 68–88%, whereas COD removal ($Y3$) was in the range of 68–82%. All three response results were reproducible within an experimental error of (±5%). ANOVA for 2^2 full CCD designs of the three responses ($Y1$, $Y2$, and $Y3$) is represented in Table 5.

3.2.1. Response surface modeling of PF

The models for PF in terms of coded factors and actual factors are described by Eqs. (2a) and (2b), respectively.

$$Y1\left(L/m^2h\right) = 48.64 + 3.95A + 12.35B - 0.67AB - 0.91A^2 + 1.07B^2 \quad (2a)$$

$$\text{Permeate flux}\left(L/m^2h\right) = 7.54 + 13.04\text{TMP} + 8.07V - 0.82\text{TMP}.V - 1.61\text{TMP}^2 + 0.88V^2 \quad (2b)$$

where TMP is the transmembrane pressure (bar) and V is flow velocity (m/s). Eq. (2b) is subjected to 0.7 bar < TMP < 2.8 bar and 1.0 m/s < V < 4.2 m/s.

The larger the magnitude of F-value and correspondingly smaller the "Prob > F" value, the more significant is the corresponding coefficient [9].

The model F value of 167.50 implies that the model is significant at a 95% confidence level. For the model term to be significant at this confidence level, the calculated probability should be lower than 0.05 ("Prob > F" less than 0.05).

Reliability of model and how good model values is fitted to experimental data is shown by the correlation coefficient R^2, which is 0.9917. Diagnostic plots such as predicted vs. actual values help to judge the model's satisfactoriness. The predicted vs. actual values plot of PF is represented in Fig. 2. This plot indicates an adequate agreement between real data and those obtained from the model.

The adjusted R^2 value is particularly useful when comparing models with different number of terms. This comparison is, however, performed in the background when model reduction is taking place [8,17].

The value of the adjusted R^2 for the model is 0.9858, which could also confirm the accuracy of the quadratic models. The LOF F-test describes the variation of the data around the fitted model. "Lack of fit F value" of 1.85 implies that the lack of fit was not significant relative to the pure error, due to noise.

AP compares the range of the predicted values at the design points to the average prediction error. An AP ratio greater than 4 indicates adequate model discrimination [8,17]. In this model, the value is well above 4.

A model can be considered to be reproducible if its CV, the ratio of the standard error of predicted value to the mean value of the observed response, is less than 10% [8]. In this model, the CV value is 2.62, suggesting good precision and reliability of the model.

Fig. 3 shows a three-dimensional response surface plot for the interaction of TMP and velocity on PF. Based on Darcy's law, increasing TMP increases the PF [18,19].

The effects of velocity on PF in a range of 1.5–4.2 m/s is represented in Fig. 3. It demonstrates that increasing velocity increases the PF. Increasing velocity promotes turbulence, and thus can reduce aggregation

Table 5
Statistical parameters obtained from the ANOVA for the models

Variable	Turbidity R (%)	COD R (%)	PF (L/m^2 h)
R^2	0.977	0.9725	0.9917
R^2 adjusted	0.945	0.9450	0.9858
F-value	30.37	35.39	167.50
Prob > F	0.0008	0.0002	<0.0001
Coefficient of variance, CV (%)	1.90	1.59	2.62
F value for lack of fit	0.2	1.44	1.85
Adequate precision, AP	17.593	17.269	42.025

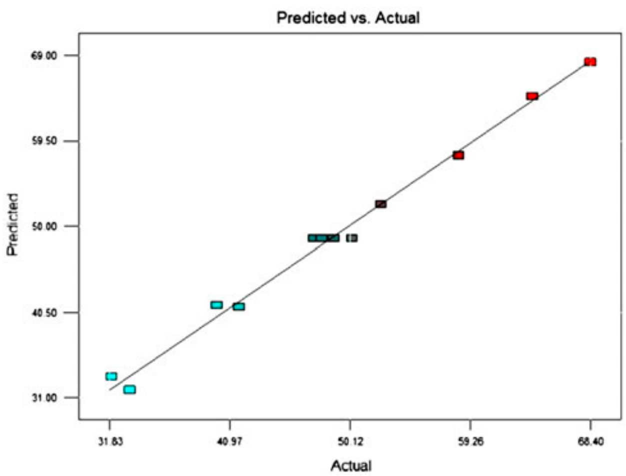

Fig. 2. Predicted vs. actual values plot for permeates flux.

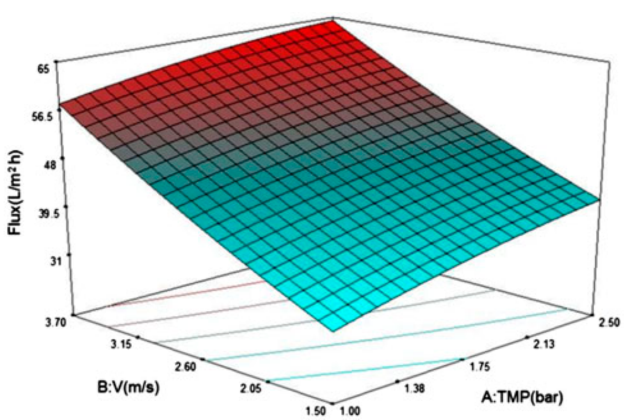

Fig. 3. Design-Expert® plot. three-dimensional surface graph of permeate flux showing the effect of TMP and velocity.

of oil concentration in the gel layer; as a result, this weakens the effect of concentration polarization and increases PF [20].

3.2.2. Response surface modeling of turbidity removal

The models for turbidity removal in terms of coded factors and actual factors are presented by Eqs. (3a) and (3b), respectively:

$$Y2 = 75.0 - 4.24A - 5.66B + 3.0AB + 0.63A^2 + 1.13B^2 + 4.66A^2B - 2.76AB^2 \tag{3a}$$

$$\text{Turbidity } (R\%) = 100.52 + 28.95\text{TMP} - 20.94V - 6.90\text{TMP}.V - 18.46\text{TMP}^2 + 6.25V^2 + 7.53\text{TMP}^2V - 3.04\text{TMP}.V^2 \tag{3b}$$

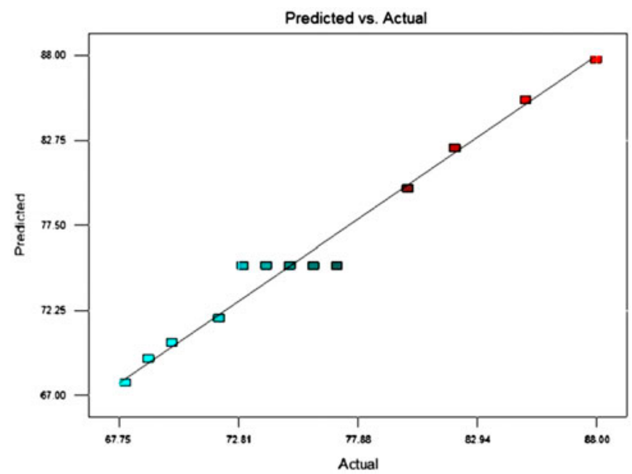

Fig. 4. Predicted vs. actual values plot for turbidity removal.

where TMP is a transmembrane pressure (bar) and V is flow velocity (m/s). Eq. (3b) is subjected to 0.7 bar < TMP < 2.8 bar and 1.0 m/s < V < 4.2 m/s.

The model F value of 30.37 in Table 4 implies that the model is significant. The value of "Prob > F" in Table 4 for the model is less than 0.05, which also indicates that the model is significant. This is desirable as it indicates that the terms in the model have a significant effect on the response.

The correlation coefficient R^2 value is 0.977, indicating a satisfactory fitting of a model to the experimental data. The predicted vs. actual turbidity removal is plotted in Fig. 4.

The AP ratio for the model is greater than 4, indicating adequate model discrimination.

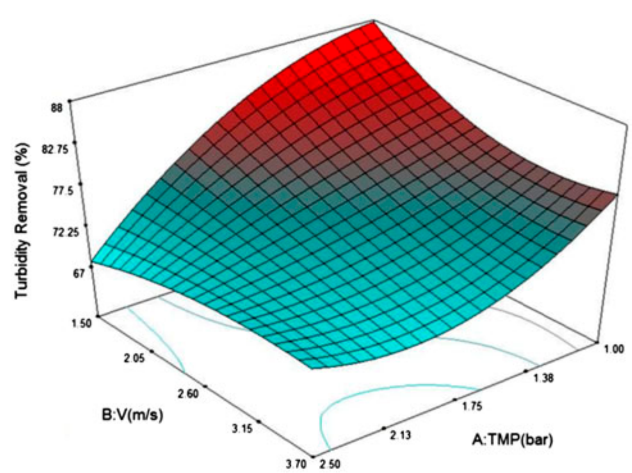

Fig. 5. Design-Expert® plot. three-dimensional surface graph of turbidity removal showing the effect of TMP and velocity.

Moreover, the CV value of the model is 1.90, showing that the model is reproducible.

The three-dimensional surface plot demonstrates the effect of process variables on turbidity removal, depicted in Fig. 5. Clearly, turbidity removal increases with a decrease in TMP, because by decreasing TMP, the PF decreases. Therefore, oil droplets cannot pass through the membrane pores and so turbidity removal increases. In addition, turbidity removal decreased with the increase in velocity, since turbulence sweep and high shear rate deposited particles away from the membrane surface; consequently, the fouling layer on the membrane surface thins. Thus, more organic matter can pass through the membrane and turbidity removal decreases, as displayed in Fig. 5 [21].

3.2.3. Response surface modeling of COD removal

The models for COD removal in terms of coded factors and actual factors are presented by Eqs. (4a) and (4b), respectively:

$$Y3 = 79.4 - 2.72A - 4.95B - 0.25AB - 2.7A^2 - 1.95B^2 + 0.2A^2B \quad (4a)$$

$$\text{COD}\,(R\%) = 67.90 + 16.90\text{TMP} + 5.40V - 1.43\text{TMP}.V - 5.64\text{TMP}^2 - 1.61V^2 + 0.32\text{TMP}^2.V \quad (4b)$$

The model F value of 35.39 in Table 4 implies that the model is significant at a 95% confidence level. The value of "Prob > F" in Table 4 for the model is less than 0.05. The value for "Prob > F" is 0.0002, showing that the model is valid in the present work.

The predicted vs. actual values plot of COD removal is illustrated in Fig. 6. The obtained correlation coefficient R^2 is 0.9725.

The AP ratio for the model is 17.269, indicating adequate model discrimination. The CV value of the model is 1.59, implying that the model is reproducible.

The interaction between TMP and velocity on COD removal is shown in the three-dimensional response surface plots in Fig. 7. Increase in TMP and velocity decreases the COD removal efficiency. This can be due to the fact that oil and grease droplets can pass through the membrane pores by increased TMP and velocity and, thus, COD removal is reduced [21].

Results demonstrate that the correlation coefficient R^2 values for PF, turbidity removal, and COD removal are 0.991, 0.977, and 0.972, respectively, indicating an acceptable fitting of the quadratic models to experimental data. In other words, the quadratic equation developed in this study shows the presence of a high correlation between observed and predicated values.

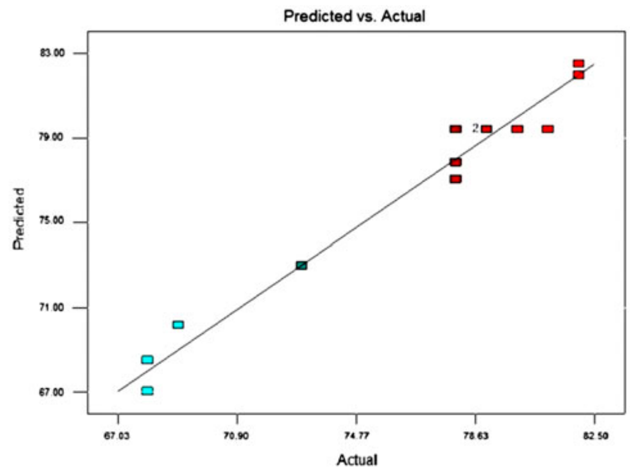

Fig. 6. Predicted vs. actual values plot for COD removal.

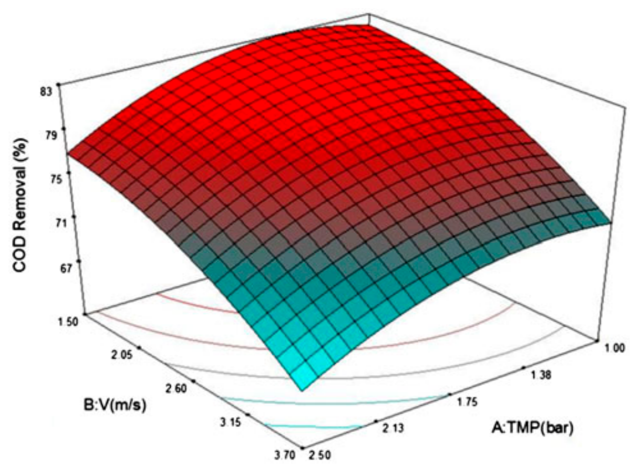

Fig. 7. Design-Expert® plot. three-dimensional surface graph of COD removal showing the effect of TMP and velocity.

Table 6
Optimization criteria at desired goal

Criteria	Goal	Low limit	Upper limit
TMP (bar)	In the range	1	2.5
Velocity) m/s)	In the range	1.5	3.7
PF (L/m^2 h)	Maximize	32	68.4
Turbidity R (%)	Maximize	68	88
COD R (%)	Maximize	68	82

Table 7
Optimum condition generated by DOE for oil-in-water emulsion treatment

Solution	TMP (bar)	Velocity (m/s)	PF (L/m^2 h)	Turbidity *R* (%)	COD *R* (%)	Total desirability
1	1.0	3.16	50.66	78.83	76.63	0.555
2	1.0	3.17	50.84	78.85	76.54	0.555
3	1.0	3.14	50.44	78.84	76.74	0.555
4	1.0	3.09	49.75	78.86	77.05	0.555

Table 8
Verification experiments under optimum condition

Run no.	PF (L/m^2 h)			Turbidity *R* (%)			COD *R* (%)		
	Experimental	Predicted	Error	Experimental	Predicted	Error	Experimental	Predicted	Error
1	51	50.66	0.34	78	78.83	−0.83	75	76.63	−1.63
2	50	50.66	−0.66	79	78.83	0.17	76	76.63	−0.63
3	49	50.66	−1.66	80	78.83	1.17	78	76.63	1.37
Mean error			−0.66			0.17			−0.29

3.2.4. Optimization of operating conditions using RSM

In this section, the main goal was to maximize PF, turbidity removal, and COD removal. Table 6 illustrates the optimization criteria used for obtaining the three responses.

In RSM, desirability functions are used simultaneously to optimize a series of quadratic models [22].

Total desirability is defined as a geometric mean of individual desirability:

$$D = (d_1 \times d_2 \times \ldots d_k)^{1/k} \quad (5)$$

where d_i is the ith desirability, $i = 1, 2, \ldots, k$ and D is the total desirability. Desirability lies between 0 and 1 [23].

The four optimum solutions and total desirability demonstrated in Table 7 were obtained by Design Expert® 7 software. With respect to the total desirability, these four solutions are not very different; consequently, Solution 1 was selected.

Table 8 shows the results of the three repeated experiments. Additional triple experiments were carried out at the optimum condition (Solution 1), in order to check the accuracy of the optimum condition generated by the software. The experimental values of the three responses were compared with the values predicted by the software. The mean error was –0.66 for PF, 0.17 for turbidity removal, and –0.29 for COD removal. This shows that there was good agreement between the experimental and predicted values.

4. Conclusion

The application of statistical DOE to study UF hollow-fiber PVDF membrane in the treatment of oil-in-water emulsion has been successfully demonstrated in the present work. A CCD coupled with RSM aided in obtaining the optimum value of the process variables studied. The optimum conditions for TMP and velocity are 1 bar and 3 m/s, respectively, where 79% of turbidity removal, COD removal of 77%, and PF of 50 L/m^2 h can be obtained after pretreatment. The experimental data and model predictions agreed well.

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