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Application of response surface methodology in pes/speek blend NF membrane for dyeing solution treatment

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Abstract. In this study, response surface methodology (RSM) was performed in NF membrane process to evaluate the separation efficiency of membrane in the removal of salt and reactive dye by varying different variables such as pressure, temperature, pH, dye concentration and salt concentration. The significant level of both the main effects and the interaction were observed by analysis of variance (ANOVA) approach. Based on the statistical analysis, the results have provided valuable information on the relationship between these variables and the performances of membrane. The rejection of salt was found to be greatly influenced by pressure, pH and salt concentration whereas the dye rejection was relatively constant in between 96.22 and 99.43% regardless of the changes in the variables. The water flux on the other hand was found to be affected by the pressure and salt concentration. It is also found that the model predictions were in good agreement with the experimental data, indicating the validity of these models in predicting membrane performances prior to the real filtration process.

Keywords: nanofiltration; membrane; response surface methodology; salt rejection; dye removal.

1. Introduction

The textile industry is a worldwide water-pollution source. Considering the volume discharged and effluent composition, the wastewater generated from dyeing and finishing operations is rated as one of the most polluting among all industrial sectors. The large quantity of wastewater generated from textile manufacturing industry has raised the environmental concerns all over the world about the elimination of colour and salt from the wastewater. It is generally known that the biodegrability of dyes is poor and the conventional treatment methods are not very efficient in removing them. There is typically about 10-50% of reactive dye still remains in waste stream due to their incomplete exhaustion during dyeing process. In some areas, soil salination is another environmental problem created by textile wastewater resulting from the high concentration of salt used for enhancing the dye uptake by the fabric during dyeing process. Therefore, dyeing contributes essentially all of the salt and colour in effluent from textile operations. To overcome these problems, nanofiltration (NF) membrane process has played a main role. There are a number of studies regarding the use of NF in the textile wastewater treatment which can be found in the literature (Lau and Ismail 2009a, Ismail and Lau 2009, Petrinic, *et al.* 2007, Shu, *et al.* 2005, Schafer, *et al.* 2003, Bes-Pia, *et al.* 2003, Akbari, *et al.* 2002, Tang and Chen 2002, Van der Bruggen, *et al.* 2001, Chen, *et al.* 1997). The researchers had investigated the effects of operating conditions and/or feed conditions on separation

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efficiency to find out the most important factors influencing the process. Since there are a large number of variables involved in separation, it is an urgent need for researcher to design experiments, building models, evaluating the effects of variables and searching optimum conditions of variables to predict targeted responses. Due to this, design of experiments (DOE) is often used to meet the objectives because it takes less time and resources than univariate procedures and able to provide large quantities of information in a minimum number of experiments.

The objective of this work is to study the separation efficiency of the self made NF membrane under different conditions of synthetic dyeing solutions by varying the pressure, temperature, pH, dye concentration and salt concentration. The salt rejection, dye rejection and water flux were analyzed using response surface methodology (RSM) in order to describe the individual and interactive effects of these variables, and to build a mathematical model for the response as a function of variables involved to find the experimental conditions in which the treatment process was efficient.

2. Experimental

2.1. Material

In this experiment, Reactive Black 5 (RB 5) from Sigma and sodium sulphate (Na_2SO_4) from Merck were used to prepare synthetic dyeing solutions at different compositions. Both HCl (0.1 N) and NaOH (0.1 N) aqueous solutions from Merck were used to modify the pH of dyeing solution prepared. All the materials were used as purchased without further purification. Self-made NF membrane - PES/SPEEK 4 blend NF was used for all experiments due to its excellent performance in removing solute with reasonable level of water flux. The blend NF membrane was fabricated with the addition of 4 wt% SPEEK in the dope. The resultant membrane has an average pore radius of 0.79 nm, surface porosity of 2.5% and effective charge density of –21.02 mol/m^3. The detail of the membrane fabrication process and the characterization of the membrane properties can be found in previous work (Lau and Ismail 2009b).

2.2. Experimental design by response surface method (RSM)

For the determination of the optimal levels of five input variables, namely pressure, temperature, pH, salt concentration and dye concentration, the response surface approach by using a set of experimental design was performed. For these five factors, this design was made up of a half fractional DOE in order to reduce the amount of experimentation necessary to obtain almost as much usable information without running a full factorial design. The range and level of the experimental input variables are presented in Table 1. In this study, the range of solute concentration used is based on the previous works (Körbahti and Rauf 2009, Mo, *et al.* 2008, Alinsafi, *et al.* 2005, Sunget, *et al.* 2004, Szpyrkowicz, *et al.* 2001, US Environmental Protection Agency 1996). Although these values cannot be modified by the experimenter (when effluent discharged directly from textile industry), they still play important role affecting the response. Due to this, it is necessary to include these variables in the preliminary study so that one could understand fundamentally the influences of solute concentration on NF performance prior to carry out pilot plant test using real textile effluent. The experimental design which was statistically designed using Design-Expert® version 6.0.8 is shown in Table 2.

Table 1 Design factors and their levels

Control factors	Code	Unit	Factor levels	
			Low (–)	High (+)
Pressure	A	bar	4	8
Temperature	B	ºC	28	50
pH	C	–	4	11
Dye concentration	D	ppm	100	400
Salt concentration	E	ppm	1000	6000

Table 2 Experimental design and responses for NF performances in dye-salt-water aqueous solution

Run	Factor					Response		
	A	B	C	D	E	Y_1	Y_2	Y_3
	Pressure (bar)	Temperature (ºC)	pH –	C_{dye} (ppm)	C_{salt} (ppm)	R_{salt} (%)	R_{dye} (%)	Flux ($\times 10^{-7}$m/s)
1	4	50	11	100	6000	82.11	97.66	3.56
2	8	50	11	400	6000	89.93	96.22	5.82
3	8	28	11	400	1000	91.20	98.31	8.96
4	8	28	4	100	1000	80.29	99.13	9.22
5	8	50	4	100	6000	71.42	97.70	6.30
6	8	50	4	400	1000	81.45	99.43	9.07
7	4	28	11	400	6000	87.01	97.50	3.18
8	8	28	4	400	6000	70.82	97.00	5.57
9	4	28	4	400	1000	73.22	99.37	5.04
10	4	50	11	400	1000	90.50	97.81	4.82
11	6	39	7.5	250	3500	87.00	98.08	6.48
12	4	28	4	100	6000	57.12	97.02	2.19
13	4	50	4	100	1000	72.24	99.33	5.19
14	8	50	11	100	1000	94.50	98.40	9.46
15	8	28	11	100	6000	84.60	96.48	5.97
16	4	50	4	400	6000	62.56	97.97	2.23
17	4	28	11	100	1000	93.06	98.91	4.60
18	4	39	7.5	250	3500	83.88	98.51	3.84
19	8	39	7.5	250	3500	87.70	97.11	8.21
20	6	28	7.5	250	3500	87.10	97.32	6.33
21	6	50	7.5	250	3500	87.01	97.03	6.45
22	6	39	4	250	3500	84.10	97.66	5.66
23	6	39	11	250	3500	90.89	98.56	5.71
24	6	39	7.5	100	3500	86.05	98.17	6.73
25	6	39	7.5	400	3500	87.93	97.06	5.53
26	6	39	7.5	250	1000	93.18	99.39	7.44
27	6	39	7.5	250	6000	82.79	97.84	5.63
28	6	39	7.5	250	3500	87.30	97.98	6.33
29	6	39	7.5	250	3500	87.49	97.76	6.38

2.3. Experimental permeation system

The solute separation performance of membranes was conducted on a laboratory-scale circulating filtration unit, as shown in Fig. 1. The feed was permeated through the shell-side of the hollow fibers, and permeate was collected from the lumen side. The desired trans-membrane pressure (TMP) was controlled by adjusting the back-pressure regulator. Full circulation mode was used, whereby the retentate and permeate were recycled to the feed storage. The composition of synthetic dyeing solutions used for the membrane filtration experiments was prepared according to Table 2. Solutions were prepared from reagent-grade chemicals in distilled water. The feed pH was adjusted in the range from 4 to 11 by HCl or by NaOH aqueous solutions. The operation temperature of the solutions was maintained at desired value using multi-purpose immersion coiled heater (Model 830-S1, Protech Electronic).

2.4. Measurement of flux and rejection

2.4.1. Water flux

Each membrane was firstly subjected to the pure water flux test in order to achieve a steady state permeate flux before being used for testing. The membrane water flux was measured using the following equation:

$$J_v = \frac{Q}{At} \tag{1}$$

where J_v is water flux (m.s^{-1}), Q permeate volume (m^3), A effective membrane area (m^2) and t is time to obtain the quantity of Q (s). Permeate of each sample collected is based on 1 hr duration.

2.4.2. Rejection of salt

Portable conductivity meter (EC300, YSI Inc) was used to measure the conductivity values of the sample and the percentage of salt rejection was calculated using a calibration curve prepared

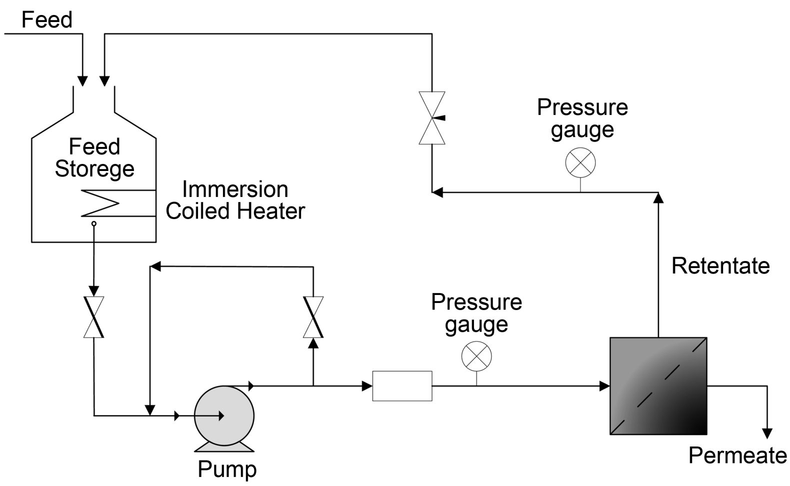

Fig. 1 Schematic lab-scale cross-flow filtration system

previously. The removal efficiency can be defined by Eq. (2)

$$R_{salt}(\%) = \left(1-\frac{C_p}{C_f}\right)\times 100 \tag{2}$$

where C_p and C_f represent the concentration of permeate (ppm) and concentration of feed (ppm), respectively.

2.4.3. Rejection of dye

To assess the extent of dye rejection, the changes in the absorbance of colour samples (RB 5) was read at wavelength of 592 nm as specified by manufacturer using DR 4000 Hach spectrophotometer based on the equation as follows.

$$R_{dye}(\%) = \left(1-\frac{Abs_p}{Abs_f}\right)\times 100 \tag{3}$$

where Abs_p and Abs_f are the absorbance of permeate (Abs) and absorbance of feed (Abs), respectively.

2.5. Modeling process

Fig. 2 shows the scheme of modeling procedure followed in this study based on RSM in Design-Expert® version 6.0.8.

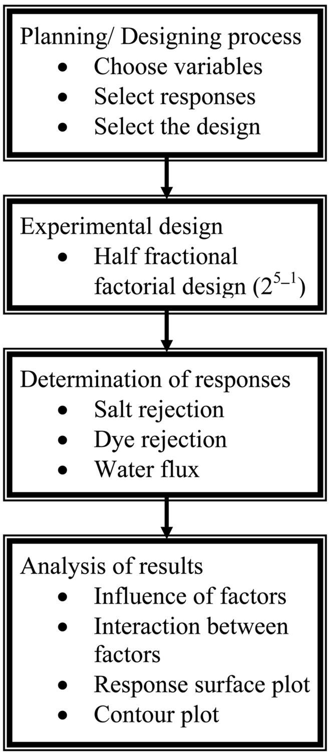

Fig. 2 Scheme of the procedure used in the modeling process

3. Results and discussion

In the first stage of study, the effect of various variables on the removal of salt and dye as well as membrane water flux was investigated using RSM according to half fractional factorial design. The results obtained from the different experimental sets are presented in Table 2. In the second stage, comparison between the results of experiments and model response was conducted to determine the suitability and validity of response surface approach in evaluating the separation efficiency of membrane during dyeing filtration process.

Table 3 shows the ANOVA results of the model of solute rejection and water flux in PES/SPEEK blend NF membrane. The model F-value obtained (22.07, 9.28 and 115.21) from each source implied the respective model was significant for salt rejection, dye rejection and water flux. A P value lower than 0.01% (or 0.0001) indicates that the respective model is considered to be statistically significant (Montgomery 1991). Also, as can be seen in Table 3, the "lack of fit F-value" of 369.95 (salt rejection), 12.71 (dye rejection) and 20.41 (water flux) implies that the lack of fit phenomenon is not important relatively to pure error, indicating the suggested model is well fitted to the observed salt and dye rejection as well as water flux. Though statistical analysis had been performed using RSM, no precise conclusion could be made on the effect of process variables on the dye rejection. It is because the values of R^2 and R^2_{adj} were far away from the aptness of model which usually has the value greater than 0.75 in most of the cases. The low value of least

Table 3 ANOVA results of the model of solute rejection and water flux in PES/SPEEK blend NF membrane

Source	Sum of squares	Degree of freedom	Mean square	F-value	Prob>F	Remarks
[a]Salt rejection (%)						
Model	1827.96	4	459.99	22.07	<0.0001	Significant
Residual	496.87	24	20.7			
Lack of fit	496.75	22	22.58	369.95	0.0027	Not significant
Pure error	0.12	2	0.061			
[b]Dye rejection (%)						
Model	14.54	5	2.91	9.28	<0.0001	Significant
Residual	7.20	23	0.31			
Lack of fit	7.15	21	0.34	12.71	0.0754	Not significant
Pure error	0.054	2	0.027			
[c]Water flux (m/s)						
Model	9.96×10^{-13}	8	1.24×10^{-13}	115.21	<0.0001	Significant
Residual	2.16×10^{-14}	20	1.08×10^{-15}			
Lack of fit	2.15×10^{-14}	18	1.19×10^{-15}	20.47	0.0475	Not significant
Pure error	1.17×10^{-16}	2	5.83×10^{-17}			

The selected factorial models for salt rejection, dye rejection and water flux are surface reduced 2FI model, surface linear model and surface reduced quadratic model, respectively.
[a]$R^2 = 0.7863$, $R^2_{adj} = 0.7507$; adequate precision = 16.59.
[b]$R^2 = 0.6687$, $R^2_{adj} = 0.5967$; adequate precision = 11.30.
[c]$R^2 = 0.9788$, $R^2_{adj} = 0.9703$; adequate precision = 39.83.

squares regression can be attributed to the excellent dye compound separation (96.22-99.43%) achieved by PES/SPEEK NF membrane as there was <3.5% difference between the actual and predicted results. On the other hand, as can be seen from Table 3, the value of R^2 (0.79 and 0.98) evaluated for salt rejection and water flux was in reasonable agreement with the R^2_{adj} (0.75 and 0.97), indicating the model is adequate for the observed salt rejection and water flux.

Fig. 3 shows the actual and predicted salt rejection and water flux. The actual values are the measured response data for particular run and the predicted values are the results generated using the approximating functions. It is found that the adequate precision of salt rejection and water flux which measured the signal to noise ratio was greater than 4, reaching the ratio of 16.59 and 39.83, respectively. This indicates the model is adequate to be used to navigate the design space. Eqs. (4) and (5) represent the approximating function of salt rejection and water flux in terms of coded factors for the surface response.

$$Y_1 = 83.26 + 2.79(A) + 8.37(C) - 4.52(E) - 1.96(AC) \tag{4}$$

$$\begin{aligned} Y_3 = {} & 6.31 \times 10^{-7} + 1.89 \times 10^{-7}(A) - 1.67 \times 10^{-8}(D) - 1.30 \times 10^{-7}(E) \\ & - 6.07 \times 10^{-8}(C^2) - 2.85 \times 10^{-8}(AE) - 2.09 \times 10^{-8}(BD) \end{aligned} \tag{5}$$

where Y_1 and Y_3 are defined as membrane salt rejection and membrane water flux, respectively and A, B, C, D and E represent pressure, temperature, pH, dye concentration and salt concentration, respectively.

Fig. 4 shows the response surface graphs of the effect of pressure and pH on the salt rejection. Other factors such as dye concentration, salt concentration and operating temperature were kept constant at 250 ppm, 3500 ppm and 39°C, respectively. The salt rejection was found to increase with pressure and to decrease with feed pH. In pressure driven membrane process, pressure is the most important parameter in enhancing the efficiency of salt removal. An increase in the pressure leads to

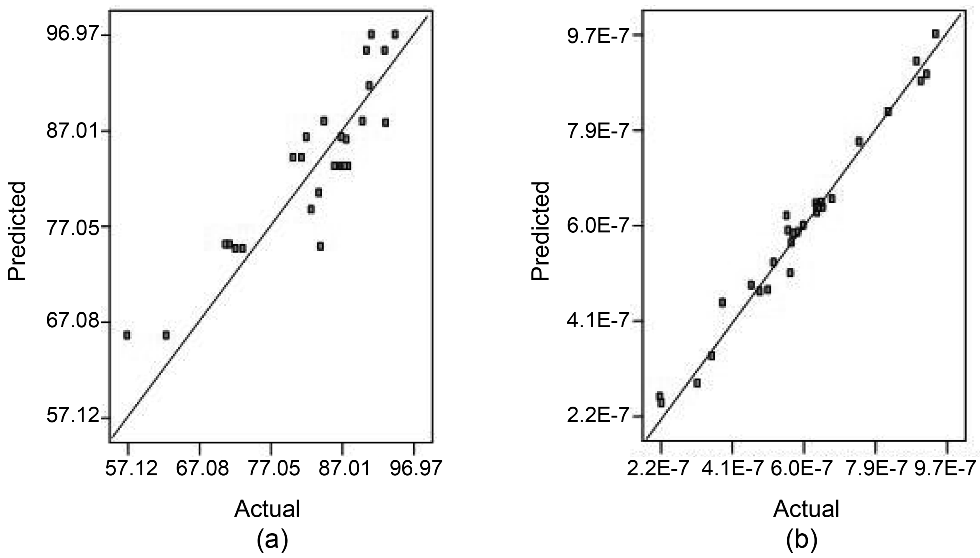

Fig. 3 The actual and predicted plot for (a) salt rejection and (b) water flux

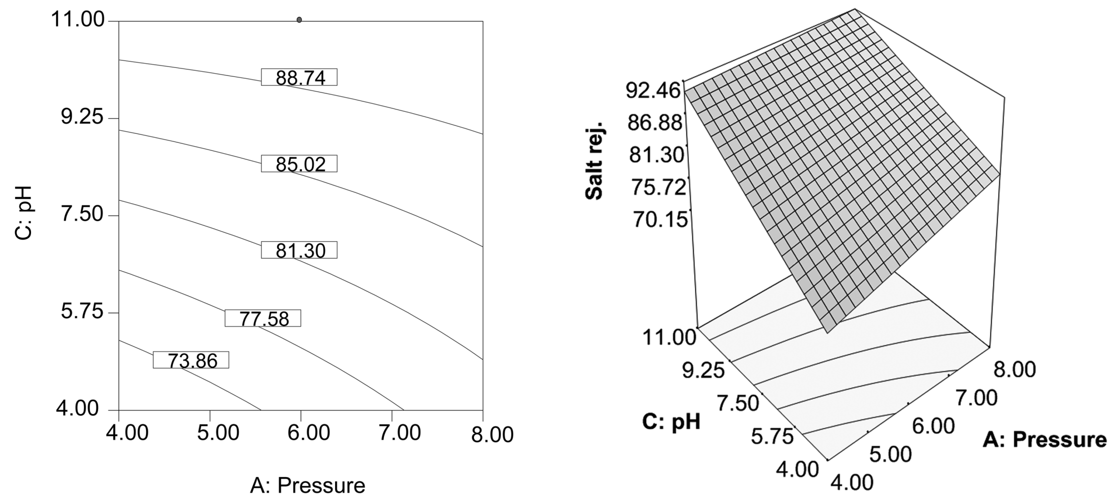

Fig. 4 A contour plot and 3D surface showing the salt rejection as a function of pressure and pH. (Experimental conditions: $C_{dye} = 250$ ppm, $C_{salt} = 3500$ ppm and $T = 39^oC$)

a higher water permeation and hence to a greater salt rejection. It is because the increased water permeation would dilute the concentration of salt in permeate resulting in higher salt rejection. In the case of electrolyte solutions, the separation mechanism is remarkably related to steric and electrostatic partitioning effects between the membrane and external solutions. The negative charge on membrane surface (sulfonic acid groups, $-SO_3H$) resulting from the addition of SPEEK is the factor determining the efficiency of salt rejection. Teixeira, *et al.* (2005) reported that membrane negative charge increases with increasing pH by determining membrane zeta potential along the surface and through the pores. The increased negative charge with the pH thus has direct effect on the salt rejection where the higher the feed pH the greater the rate of rejection (Ismail and Lau 2009). The effect of pH on salt rejection was also greatly pronounced by the significant term in Eq. (4). In addition to pressure and pH, the membrane separation performance can also be influenced by solute concentration (Mulder 1996). In general, increasing salt concentration (high ionic strength) would produce a stronger shielding effect and consequently result in a decrease in membrane repulsion forces against anions, leading to a lower retention. Therefore, based on the model as suggested by RSM in Eq. (4), a decline in the rejection with increasing concentration of salt could be attributed to the electrostatic effect between the membrane surface and the sodium sulfate. As a conclusion, it can be generally said that the salt rejection increases with increasing pressure and feed pH and decreases with increasing solute concentration.

Fig. 5 shows the contour plot of membrane dye rejection as a function of salt concentration and pressure. Clearly, the dye rejection was found to remain constant (97.4-98.5%) by varying both the salt concentration and operating pressure. There were no significant changes in the rejection rate, indicating the tiny influences of these factors on the dye rejection. The results were in good agreement with the previous results reported in which dye rejection of NF is independent of the operating conditions and feed properties (He, *et al.* 2008, Petrinic, *et al.* 2007, Akbari, *et al.* 2002, Jiraratananon, *et al.* 2000). According to Tang and Chen (2002), rejection of RB 5 remained constantly with increasing dye concentration from 92 to 1583 ppm at feed pressure of 5 bar. With the presence of NaCl in the dyeing solution, Jiraratananon, *et al.* (2000) also observed that the dye

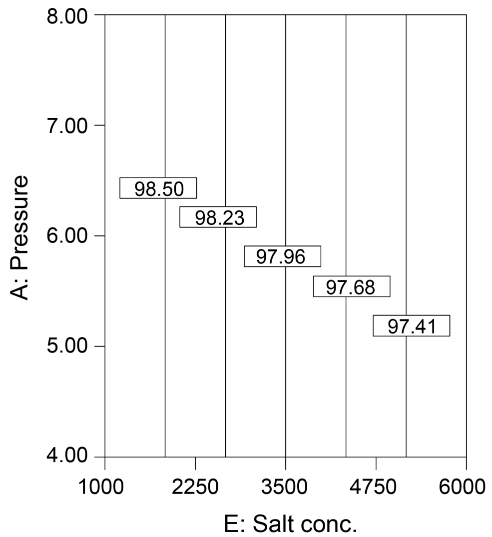

Fig. 5 A contour plot of membrane dye rejection as a function of salt concentration and pressure. (Experimental conditions: C_{dye} = 250 ppm, feed pH = 7.5 and T = 39°C)

rejection of NF membrane remained unchanged. Further, He, *et al.* (2008) experienced that rejection of dye was insensitive to pH as variation in feed pH only resulted in the changes of surface charge properties which in turn affecting the rejection rate of electrolytes. In the same study, the authors also reported that no strong temperature effects are inherently present in the charge repulsion and sieving effect to affect the solute rejection in NF. The description was in line with the observation where the dye rejection decreased slighly (from 99.6-99%) with increasing temperature from 15-40°C. In addition to this, Jiraratananon, *et al.* (2000) and Ledakowicz, *et al.* (1998) found that temperature can be excluded in membrane evaluation as they contributes a little or none to the increase in dye rejection. Based on these descriptions, it could be said that dye separation mechanism in NF process is dominated by the difference between the size of pore radius and the MW of the dye compounds, regardless of process variables. It is thus believed that NF membrane is able to achieve promising results in dye separation if the pore radius of membrane used is controlled at sub-nanometer range, which is typically smaller than the MW of dye compounds.

Fig. 6 illustrates the response surface for the membrane water flux with respect to the salt concentration and operating pressure. It is found that the water flux increased as the operating pressure increased and decreased as the salt concentration increased. The changes in water flux with pressure and salt concentration were also described in Eq. (5). In NF, high concentration of salt causes a large osmotic pressure difference, which leads to lower fluxes. Although this is not a fouling effect (reversible), the result in terms of water fluxes is the same as for membrane fouling. The decrease in water flux due to osmotic pressure build up can be generally expressed by the phenomenological equation as introduced by Spiegler and Kedem (1966).

$$J = L_p(\Delta P - \sigma\Delta\pi) \tag{6}$$

where J is water flux (m.s^{-1}), L_p water permeability coefficient (m.s^{-1}.bar^{-1}), ΔP trans-membrane pressure (bar), σ reflection coefficient and $\Delta\pi$ is osmotic pressure (bar) due to the concentration

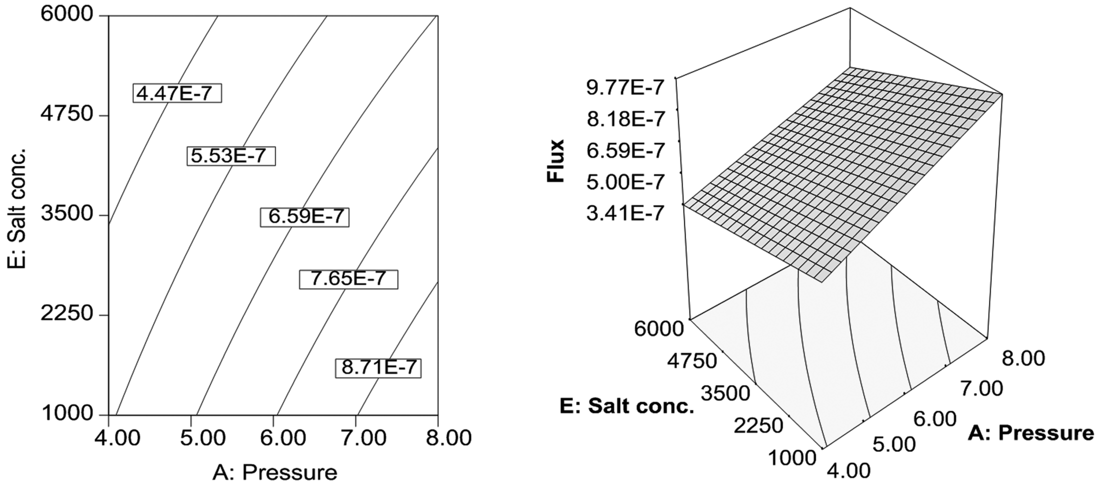

Fig. 6 A contour plot and 3D surface showing the membrane water flux as a function of salt concentration and pressure. (Experimental conditions: C_{dye} = 250 ppm, feed pH = 7.5 and T = 39°C)

difference across the membrane. Since the use of high concentration of salt is inevitable in textile industry during dyeing process, therefore it is necessary to pressurize the membrane at higher operating pressure in order to obtain greater amount of permeate flux. The operating pressure, however, should be controlled properly so that it falls within the range of recommended operating conditions of particular NF membrane. As reported by Bandini, *et al.* (2005), parameters such as pH and dye concentration did play a role in influencing water flux, however, in this study the influences of these two are not contributing in a significant way to flux decline in comparison to the pressure and salt concentration effects. The explanation is found to be in line with the one reported by He, *et al.* (2008) and Gomes, *et al.* (2005). Small concentration of reactive dye in the synthetic dyeing solution would not result in a significant increase in osmotic pressure compared to salt concentration. By employing Eq. (7), Gomes, *et al*. (2005) elucidated that influence of osmotic pressure of reactive dye was minor in affecting the water flux.

$$\pi = 0.6241C - 0.074C^2 + 0.0030C^3 \tag{7}$$

where π is the osmotic pressure value (bar) and C is the dye concentration (g/l). The dye concentration however should be taken into consideration in a long term of operation as it is generally believed that water flux tends to decrease with the time due to the formation of undesirable dye cake layer on membrane surface, which resisting the permeation of water molecules through membrane.

On the other hand, in practical treatment process, it is impossible for one to manipulate the solute concentrations in textile effluent since it is discharged from the textile industry and their concentrations vary depending on the operation. Therefore, we decided to vary only the values of pressure, medium temperature and pH to find out the optimum performance of membrane. The concentrations of both salt and dye compound were selected at maximum level. By employing RSM (point prediction), it is experienced that the NF membrane could achieve 87.94% salt rejection, 96.49% dye rejection and 6.03×10^{-7} m.s^{-1} water flux under operating conditions of 8 bar, pH 11 and

Table 4 Comparison between the experimental values and model response values obtained

	Responses		
	R_{salt} (%)	R_{dye} (%)	Flux ($m.s^{-1}$)
Experimental value	87.03	97.11	6.27×10^{-7}
Model response	87.94	96.49	6.03×10^{-7}
Error (%)	1.05	0.64	3.83

ambient temperature. An additional experiment was also conducted to verify the validity of the model developed, as shown in Table 4. As can be seen, there is small difference between the experimental values and model response values obtained. This therefore confirmed that RSM could be effectively used to predict the separation performance of membrane in dyeing wastewater prior to carry out the separation process.

4. Conclusions

In this study, the performance of NF membrane for the treatment of synthetic dyeing solutions was investigated using RSM by varying control variables such as pressure, temperature, pH, dye concentration and salt concentration. The results obtained from RSM fully explain the NF performance as a function of these variables in terms of water flux and rejections of salt and dye. In conclusion,

(a) Salt rejection was influenced by pressure, pH and salt concentration where the rejection rate increased with increasing both pressure and pH but decreased with increasing salt concentration.

(b) Dye rejection was relatively constant in between 96.22 and 99.43% regardless of the process variables. This indicates these variables have tiny influence on the rejection of dye. In principle, the retention of reactive dye is less affected by membrane surface charge properties as it is mainly decided by membrane pores (sieving effect).

(c) Water flux increased with increasing pressure whereas decreased with increasing salt concentration. Other process variables, however, have negligible effects on water flux.

(d) The models obtained by RSM are able to predict the behavior of membrane performances both in water flux and solute rejection due to the small deviation (%) between the experimental values and model response values.

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