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Optimization of vacuum membrane distillation parameters for water desalination using Box–Behnken design

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ABSTRACT

Water desalination via vacuum membrane distillation (VMD) requires a proper process parametric study to determine optimal performance characteristics. In this study, response surface methodology, involving Box–Behnken design matrix for most important operating parameters: feed temperature, vacuum pressure, feed flow rate, and feed concentration, have been employed. Consequently, 27 experiments were conducted to construct a quadratic model. Optimal experimental conditions for water desalination via VMD were obtained using analysis of variance, feed temperature ($T = 55^{\circ}$C), vacuum pressure ($P_v = 10$ mbar), feed flow rate ($Q = 38.63$ mL/s), and feed concentration ($C = 100$ g/L). Under these conditions the measured permeate flux 17.96 kg/m^2 h was found to be the highest value in this study confirming the validity of the applied optimization procedure. Regression analysis showed agreement of the experimental data with the second-order polynomial model with determination coefficient (R^2) value of 0.9880.

Keywords: Membrane distillation; Desalination; RSM method; Optimization; ANOVA

1. Introduction

Membrane distillation (MD) is an emerging thermally driven process mainly suited for applications in which water is the major component present in the feed solution to be treated. It combines use of both thermal distillation and membrane process, and differs from other membrane technologies in which driving force for desalination is the difference between vapor pressures of water across the membrane, rather than total pressure difference [1,2]. Generally, MD process is characterized by different embodiments designed to impose a vapor pressure difference between the two membrane sides in order to drive vapor across the membrane. Lowering the vapor pressure at the permeate side can be accomplished in different ways: (a) direct contact MD (DCMD), (b) air gap MD (AGMD), (c) sweeping gas MD (SGMD), and (d) vacuum MD (VMD) [3,4].

VMD is a membrane operation where microporous hydrophobic membranes are used for removing water vapor and volatile compounds from aqueous solutions [5]. Being the membrane hydrophobic, the liquid cannot permeate through the pores and they are blocked at one side of the membrane. By applying vacuum at the other side of the membrane, partial pressure

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difference is created across the membrane and both the water vapor and volatile species start to permeate through the membrane pores [6].

This configuration combines two advantages: a very low-conductive heat loss and a reduced mass transfer resistance by removing air from its pores through deaeration or by applying continuous vacuum in the permeate side lower than equilibrium vapor pressure. This process allows to reach higher partial pressure gradients and thus higher fluxes, in comparison with other MD configurations [7]. It is to be mentioned that VMD is the least studied one, only about 8% among the published references [8]. However, its corresponding application research has obtained excellent achievements, exhibiting its promising application prospects. Recently, VMD has become an active area of research by many [5,9–12]. Some studies have focused on the ethanol/water separation [7,13]. Other researchers studied the use of VMD in removal of trace gases and VOCs from water [14–16]. Some attempts have been also made for concentration of ginseng extracts aqueous solutions [17]. Banat et al. [18] and Criscuoli et al. [19] used VMD for treatment of dye solutions. Desalination from water is one of the main applications of VMD and has been studied by many authors [20–23]. Also, Criscuoli et al. compared different lab-made flat module designs in terms of trans-membrane fluxes, energy consumption, and evaporation efficiency for DCMD and VMD experiments. According to the obtained results, VMD performs better than DCMD in terms of permeate flux, energy consumption permeate flow rate ratio, and evaporation efficiency [24,25].

To the best of our knowledge, until now, there is no published report on desalination of water by VMD using response surface methodology (RSM). All studies on VMD were carried out by applying the conventional method of experimentation (i.e. one parameter is varied, while the others are maintained constant). Such approach of experimentation ignores the coupling and interaction effects between operating parameters. Coupling and interaction effects between operating parameters and between different permeating species through the membrane and its pores occur also in VMD process. Studies on this subject as well as on optimization of different VMD systems are needed in order to increase VMD performance.

Design of Experiments (DoE) and RSM, which is a collection of mathematical and statistical techniques useful for process modeling and optimization, can be used to model and optimize VMD process as well as to study interaction effects between VMD operating parameters. The statistical methods of experimentation are useful to understand the interaction effects between factors and to reduce the total number of experimental runs. Recently, RSM has been applied in DCMD [1,26–28] and AGMD [1]. Experimental design and RSM were applied recently by Khayet et al. [27] for optimization of asymmetric flat sheet membranes fabricated by the phase inversion method for DCMD. Also, Mohammadi and Kazemi [29] applied Taguchi method for optimization of phenol removal form phenolic wastewater by VMD. The effects of feed temperature, downstream pressure, feed pH, and feed concentration on the distillate flux and distillate quality were studied.

In this study, statistical experimental design and RSM are applied to model and optimize VMD process. Although, in this case, it is easy to predict the outcome of experiments since there is no significant interaction effect between chosen variables, but the main goal of this study was the development of a precise and reliable model to predict the overall behavior of the system.

2. Experimental

Experiments were carried out using a flat sheet commercial polytetrafluoroethylene (PTFE) and flat microfiltration (MF) membrane from Sartorius Co. A cross flow membrane module made from Teflon was used in the experiments (Fig. 1).

Effective area of the membrane in the module was 7.89 cm^2. To omit the effect of poor flow distribution within the module and reduce the heat transfer phenomena to the outside from the module walls, the membrane cell was designed in such a small dimension. Pore size, porosity, and thickness of PTFE membrane (Sartorius Co) were 0.2 μm, 80%, and 73 μm, respectively. The schematic representation of VMD setup is shown in Fig. 2.

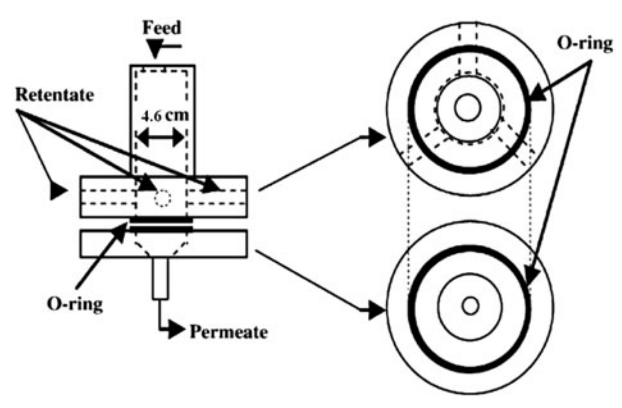

Fig. 1. Membrane module.

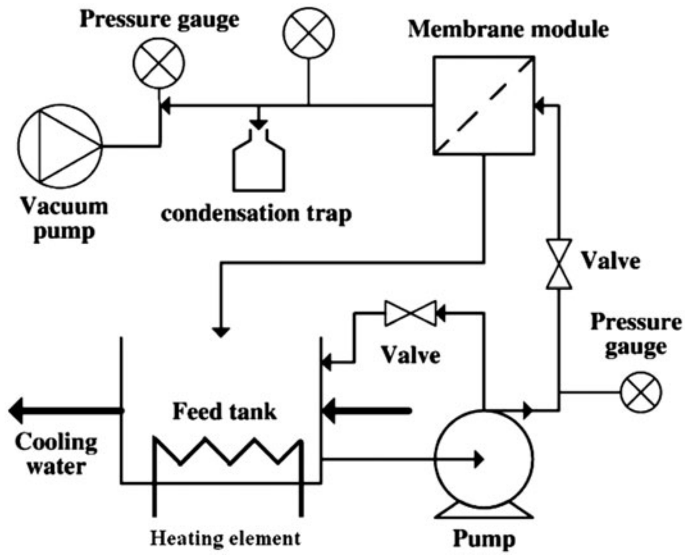

Fig. 2. Schematic diagram of VMD setup.

The feed was continuously fed to the membrane module from a feed tank, sufficiently large, to keep the concentration nearly constant. The membrane flux was measured by collecting the permeate in a condensation trap. Feed composition and temperature were considered as constant values within the module. One important consideration in the setup was that feed pump was not able to flow the small required flow rates in this research, so the excess flow was bypassed. The bypass flow had a significant influence on feed temperature. Because of bypass flow, the pump heats the feed and it is needed to cool the feed to control its temperature, so the feed tank was equipped with a cooling water coil. Electrical conductivity and total dissolved solids (TDS) of the MD permeates were measured using a conduct meter (CRISON GLP 32).

In all experiments, the MD permeates with electrical conductivity within a range of 1.00–3.20 μS/cm and TDS within a range of 0.06–2.10 ppm are produced and analyzed.

2.1. Experimental design

For desalination by VMD, four important operating parameters, such as feed temperature, vacuum pressure, feed flow rate, and feed concentration, were chosen as the independent variables and designated as X_1, X_2, X_3, and X_4, respectively. The low, middle, and high levels of each variable appointed as −1, 0, and 1, respectively, are presented in Table 1.

The statistical design and data analysis were accomplished by Design-Expert 8.0.7.1 software (trial version). The number of experiments is optimized by Box–Behnken statistical design in order to verify the interactions between the major operating variables and their influences on the permeate flux. Consequently, the number of experiments required to investigate four parameters at three levels was 27. The center point in the design was repeated three times for estimation of errors and curvature. The central values chosen for this experimental design were $T = 40$℃, $P_v = 50$ mbar, $Q = 37.5$ mL/s, and $C = 200$ g/L in uncoded form. The results from this limited number of experiments acquired a statistical model. Experimental points for Box–Behnken statistical design are shown in Table 2.

The response of the experiments was measured in terms of permeate flux which is defined by Eq. (1).

$$J = \frac{W}{S \times t} \tag{1}$$

where J is the permeate flux (kg/m^2 h), W is the quantity of permeate (kg), S is the effective membrane area (m^2), and t is the sampling time (h).

The regression analysis was performed to estimate the response function and the permeate flux could be predicted by the quadratic model as shown in Eq. (2).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_i X_i^2 + \sum_{i<j}^{k} \beta_{ij} X_i X_j + \ldots + e \tag{2}$$

where i is the linear coefficient, j is the quadratic coefficient, β is the regression coefficient, k is the number of factors studied and optimized in the experiment, e

Table 1
Experimental design levels of chosen variables

	Factors	Level		
Variable, unit	X	Low (−1)	Middle (0)	High (+1)
Feed temperature, T (℃)	X_1	25	40.0	55
Vacuum pressure, P_v (mbar)	X_2	10	50.0	90
Feed flow rate, Q (mL/s)	X_3	15	37.5	60
Feed concentration, C (g/L)	X_4	100	200.0	300

Table 2
The Box–Behnken design matrix for coded variables

Std. order	Run	T (℃)	P_v (mbar)	Q (mL/s)	C (g/L)	Flux (kg/m^{2}h)
1	18	25	10	37.5	200	9.8612
2	12	55	10	37.5	200	15.4195
3	2	25	90	37.5	200	1.8861
4	1	55	90	37.5	200	7.4445
5	17	40	50	15.0	100	8.9396
6	25	40	50	60.0	100	9.4103
7	15	40	50	15.0	300	4.7718
8	26	40	50	60.0	300	5.2425
9	23	25	50	37.5	100	9.7088
10	16	55	50	37.5	100	15.2672
11	6	25	50	37.5	300	5.5410
12	27	55	50	37.5	300	11.0994
13	7	40	10	15.0	200	9.0920
14	5	40	90	15.0	200	1.1170
15	13	40	10	60.0	200	9.5627
16	20	40	90	60.0	200	1.5876
17	11	25	50	15.0	200	4.9097
18	14	55	50	15.0	200	10.4681
19	24	25	50	60.0	200	5.3804
20	4	55	50	60.0	200	10.9388
21	22	40	10	37.5	100	14.2154
22	9	40	90	37.5	100	4.2945
23	8	40	10	37.5	300	9.3990
24	19	40	90	37.5	300	2.0726
25	21	40	50	37.5	200	8.1540
26	3	40	50	37.5	200	8.8061
27	10	40	50	37.5	200	8.9300

is the random error, and Y is the predicted response. The statistical analysis of the results was carried out by analysis of variance (ANOVA). It evaluates the model and its parameters, along with the determination of the individual and interactive influences of the factors on the permeate flux by discovering the coefficients of Eq. (2). Statistical significance was verified by the F-test in the program. Model terms were selected or rejected based on the probability value with 95% confidence level. Eventually, response surfaces and contour plots were generated in order to visualize the individual and the interactive effects of the independent variables.

3. Results and discussion

The process variables of VMD for desalination were examined using RSM with Box–Behnken statistical design. RSM is a collection of statistical and mathematical techniques and is useful for developing, improving, and optimizing of processes. This method can be used to evaluate the relative significance of several affecting factors even in the presence of complex interactions. RSM has various advantages compared with conventional methods, including reduction in the number of experiments, provision of rapid and reliable experimental data, consideration of the effects and interactions between factors, minimizing experimental time, and cost consumption [30].

The experiments were verified using statistical analysis and a modified quadratic model was selected, as suggested by the software. The regression model equation for permeate flux is expressed as Eq. (3) and Eq. (4) in terms of actual factors and coded factors, respectively.

$$\begin{aligned} Y_{act} = {} & 15.02163 - 0.29745 \times T - 0.048015 \times P_v + 0.30014 \\ & \times Q - 0.042359 \times C + 2.08333 \times \text{E-8} \times T \times P_v \\ & + 2.96296\text{E-8} \times T \times Q - 1.66667\text{E-9} \times T \times C \\ & - 2.77778\text{E-9} \times P_v \times Q + 1.62154\text{E-4} \times P_v \times C \\ & - 3.94746\text{E-19} \times Q \times C + 6.03406\text{E-3} \times T^2 \\ & - 8.68070\text{E-4} \times P_v^2 - 3.86239\text{E-3} \times Q^2 \\ & + 3.62344\text{E-5} \times C^2 \end{aligned} \tag{3}$$

$$\begin{aligned} Y_{cod} = {} & 8.63 + 2.78 \times X_1 - 4.10 \times X_2 + 0.24 \times X_3 - 1.98 \\ & \times X_4 + 1.250\text{E-5} \times X_1 \times X_2 + 1.000\text{E-5} \times X_1 \times X_3 \\ & - 2.500\text{E-6} \times X_1 \times X_4 - 2.500\text{E-6} \times X_2 \times X_3 \\ & + 0.65 \times X_2 \times X_4 + 1.36 \times X_1^2 - 1.39 \times X_2^2 - 1.96 \\ & \times X_3^2 + 0.36 \times X_4^2 \end{aligned} \tag{4}$$

where Y is the permeate flux, X_1 is the feed temperature, X_2 is the vacuum pressure, X_3 is the feed flow rate, and X_4 is the feed concentration. The values of the responses determined by means of the regression equations were compared with the obtained experimental data and the results are presented in Fig. 3. As observed, the models show good predictions of the experimental data. Therefore, based on the statistical tests and data comparison the models can be considered adequate for VMD simulation and optimization.

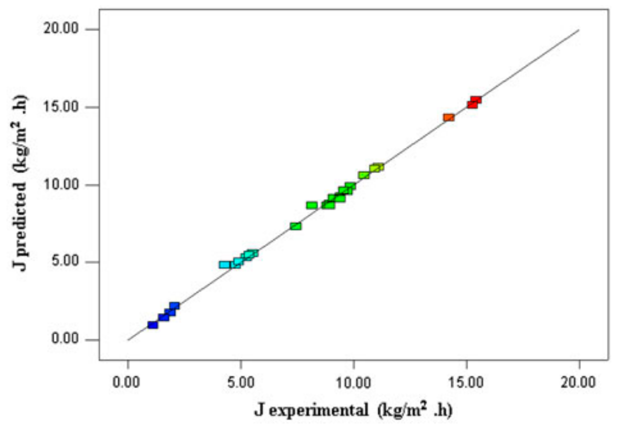

Fig. 3. Scatter diagram of predicted response versus actual response for the desalination from water via VMD.

ANOVA was used to check the significance and fitness of the model. As shown in Table 3, the model F-value of 328.11 implies that the model is significant. There is only 0.01% chance that this large model

Table 3
ANOVA results of quadratic model for water desalination via VMD

Source	Coefficient estimate	Sum of squares	Df	Mean square	F value	Prob > F	Remark
Model		401.46	14	28.67	328.1059	<0.0001	Significant
Intercept	8.63						
X_1	2.78	92.68	1	92.68	1,060.48	<0.0001	
X_2	−4.10	201.28	1	201.28	2,303.11	<0.0001	
X_3	0.24	0.66	1	0.66	7.60	0.0174	
X_4	−1.98	46.84	1	46.84	535.99	<0.0001	
$X_1 \times X_2$	1.25E-05	6.25E-10	1	6.25E-10	7.15E-09	0.9999	
$X_1 \times X_3$	1.00E-05	4.00E-10	1	4.00E-10	4.58E-09	0.9999	
$X_1 \times X_4$	−2.50E-06	2.50E-11	1	2.50E-11	2.86E-10	1.0000	
$X_2 \times X_3$	−2.50E-06	2.50E-11	1	2.50E-11	2.86E-10	1.0000	
$X_2 \times X_4$	0.65	1.68	1	1.68	19.25	0.0009	
$X_3 \times X_4$	0	0	1	0	0	1.0000	
X_1^2	1.36	9.83	1	9.83	112.48	<0.0001	
X_2^2	−1.39	10.28	1	10.28	117.71	<0.0001	
X_3^2	−1.96	20.39	1	20.39	233.31	<0.0001	
X_4^2	0.36	0.70	1	0.70	8.01	0.0152	
Residual		1.04	12	0.08			
Lack of fit		0.70	10	0.07	0.40	0.8664	Not significant
Pure error		0.34	2	0.17			
Cor total		402.51	26				

F-value occurs due to noise. Values of Prob > F less than 0.05 indicate that the model terms are significant. The values greater than 0.1 indicate that the model terms are not significant. As seen in Table 3, the linear and quadratic coefficients are found to be more significant than the interacting coefficients. ANOVA study suggests that vacuum pressure (X_2) ($p < 0.0001$, $F = 2{,}303.11$, SS = 201.28) has the most significant effect on permeate flux followed by feed temperature (X_1) ($p < 0.0001$, $F = 1060.48$, SS = 92.68) and feed concentration (X_4) ($p < 0.0001$, $F = 535.99$, SS = 46.84). Feed flow rate (X_3) ($p = 0.0174$) has comparatively negligible effect on the response. There is no significant interaction between parameters since the p value for the most of interacting coefficients is roughly 1 except between feed concentration and vacuum pressure ($X_2 \times X_4$) ($p = 0.0009$) which there is some weak interaction effect. This is due to the fact that vacuum pressure is the most significant factor on the permeate flux, therefore by decreasing it the permeate flux increases significantly, and as the setup works in a batch mode by increasing the permeate flux more water passes through the membrane and this causes the feed concentration in the feed tank.

The lack of fit F-value of 0.4 implies that the lack of fit is not significant relative to the pure error. There is 86.64% chance that this large lack of fit F-value occurs due to noise.

Predicted R^2 is a measure of how accurate the model predicts a response value. The adjusted R^2 and predicted R^2 should be within approximately 0.20 of each other, to be in reasonable agreement. If they are not, there may be a problem with either the data or the model. In this case, the predicted R^2 of 0.9880 is in reasonable agreement with the adjusted R^2 of 0.9944. Adequate precision is a measure of the range in predicted response relative to its associated error, in other words a signal-to-noise ratio. Its desired value is 4 or

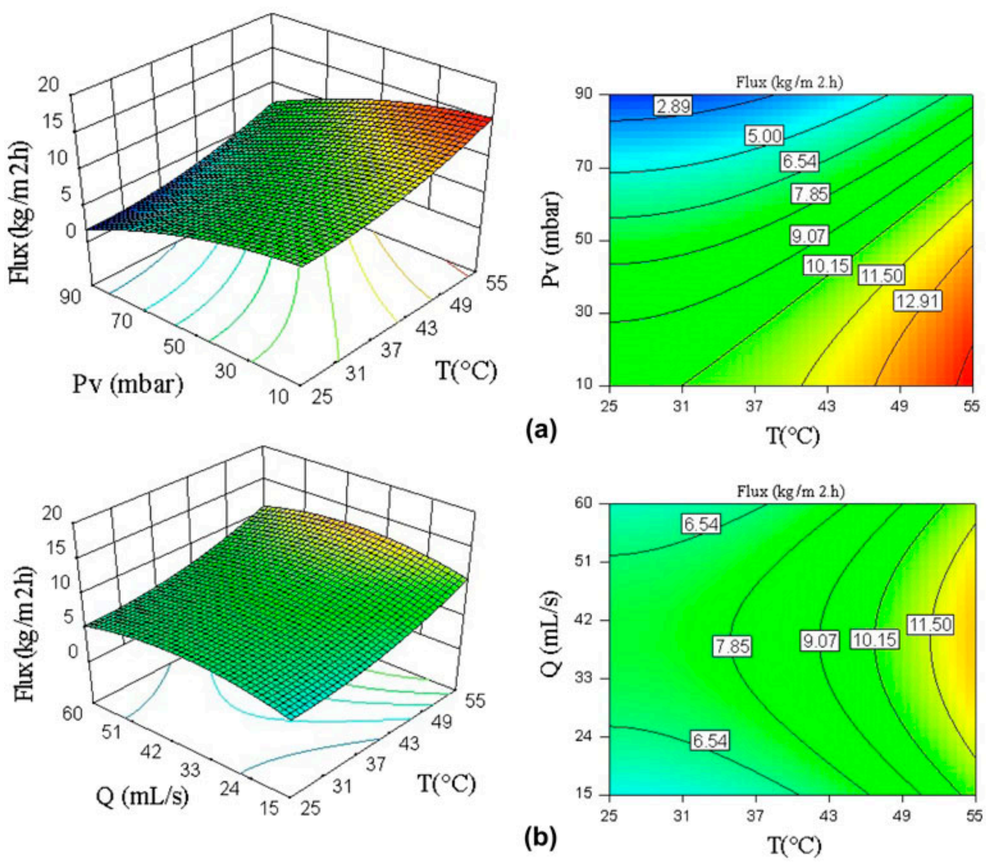

Fig. 4. Response surface plots and a contour-line plot of predicted VMD flux (a) as a function of vacuum pressure (P_v) and feed temperature (T) at salt concentration $C = 200$ g/L and feed flow rate $Q = 37.5$ mL/s, (b) as a function of Q and T at $P_v = 50$ mbar and $C = 200$ g/L, (c) as a function of C and T at $P_v = 50$ mbar and $Q = 37.5$ mL/s, (d) as a function of Q and P_v at $T = 40$°C and $C = 200$ g/L, (e) as a function of C and P_v at $T = 40$°C and $Q = 37.5$ mL/s, (f) as a function of C and Q at $T = 40$°C and $P_v = 50$ mbar.

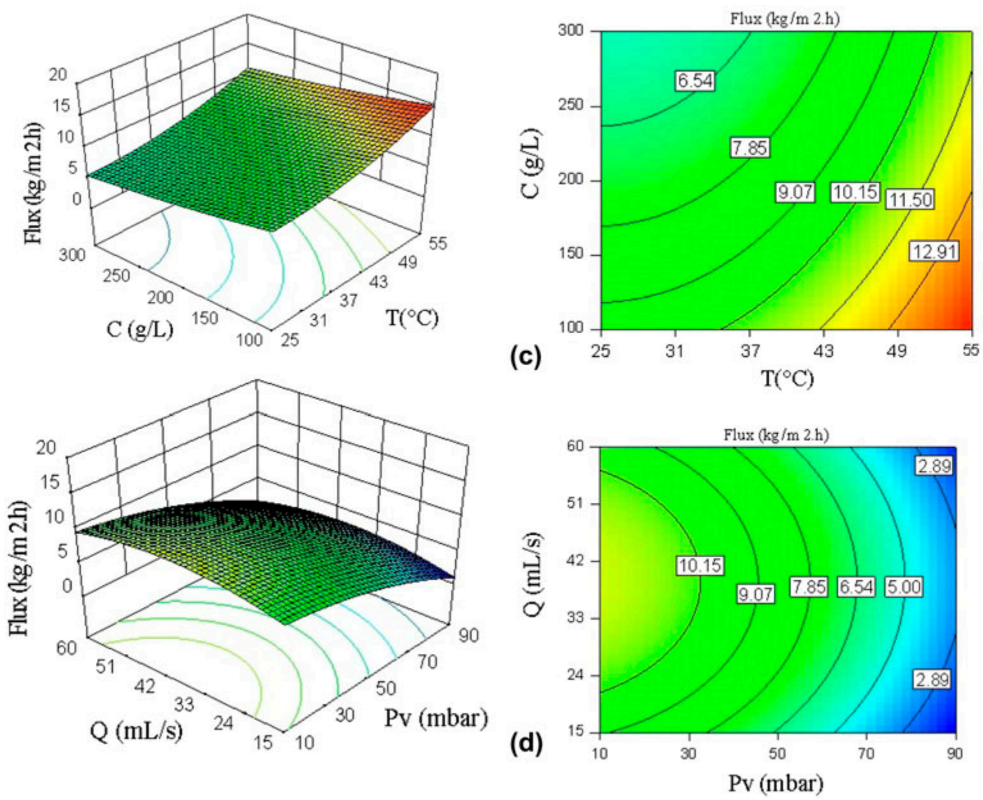

Fig. 4. (*Continued*).

more [31,32]. The ratio of 65.889 indicates an adequate signal. The coefficient of variation for this model is the error expressed as a percentage of the mean. The effects of the VMD operating parameters on the permeate flux are plotted in Fig. 4 in 3-D and 2-D contour plots.

As expected, increasing feed temperature leads to an "exponential" increase of the VMD permeate flux indicating the exponential variation of the feed vapor pressure as the driving force of transmembrane vapor pressure [3].

On the other hand, reduction of downstream pressure increases the VMD permeate flux through the membrane. This is due to the fact that the driving force in MD process, in general, and in VMD, is a vapor pressure difference across both sides of the membrane pores. Therefore, working at lower downstream pressure usually results in higher transmembrane permeate flux [22].

Moreover, the VMD permeate flux reduction with increasing feed concentration was also observed, as expected. This can be attributed to the fact that addition of NaCl reduces water activity in the feed. Since water vapor pressure is the driving force of MD process, and it relates to water activity, reduction of VMD permeate flux with further increasing feed concentration can be due to the driving force reduction of the process. At high-salt concentrations, an additional boundary layer develops next to the membrane interface, parallel to the temperature boundary layer. This concentration boundary layer, together with the temperature boundary layer further reduces the driving force for evaporation [4].

Enhanced turbulent cross flow reduces both boundary layers and improves VMD performance (Fig. 4(b), (d), and (f)). Increasing VMD permeate flux with feed flow rate (Reynolds number) indicates importance of the polarization effects in the system. In other words, increasing VMD permeate flux with feed flow rate is due to the reduction of temperature and concentration boundary layers thicknesses. At higher feed flow rates (more than 38 mL/s), permeate flux decreases. This is due to the fact that a greater feed flow rate means a lower residence time and as a result the feed solution spends less time in contact with the membrane surface [23,33]. Also, with increasing feed flow rate, due to the reduction of residence time of the feed in the feed tank, feed temperature in the module may be lower than that expected, because the feed remains in the feed tank for a shorter time to be reheated at the operating temperature.

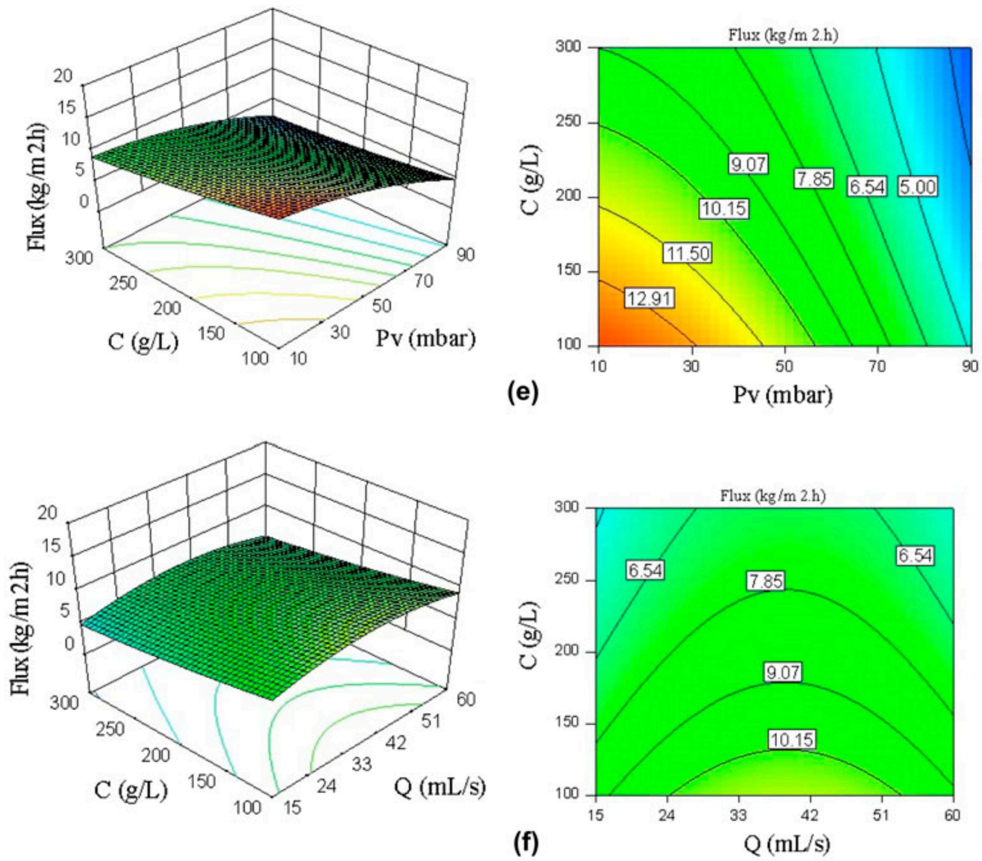

Fig. 4. (*Continued*).

To confirm the model adequacy for predicting the maximum response results, four experiments using these optimum operation conditions were performed in Table 4. The obtained actual values and its associated predicted values from the experiments were compared for further residual and percentage error analysis. The percentage error between actual and predicted values of the responses was calculated based on Eq. (5).

$$\% \text{ Error} = \frac{\text{Residual}}{\text{Actual value}} \times 100 \tag{5}$$

where the residual can be determined from the difference between actual and predicted values, actual values are the experimental values obtained in this study. The results presented in Table 5 have demonstrated that the percentage error implied by the developed empirical model is considerably small for the response. The percentage error between the actual and predicted values is well within the value of 3%, suggesting that the model adequacy is reasonable within 97% of the prediction interval [34]. The good agreement between the predicted and experimental results verifies the validity of the model and confirms the existence of the optimal point.

Table 4
Optimum values of the factors (process parameters) for maximum response results

Factors	Optimum value
Y (flux, kg/m^{2}h)	18.46
X_1 (Feed temperature, ℃)	55.00
X_2 (Vacuum pressure, mbar)	10.00
X_3 (Feed flow rate, mL/s)	38.63
X_4 (Feed concentration, g/L)	100.00

Table 5
Predicted and experimental values for the responses at the optimum condition

Response	Flux (kg/m^{2}h)
Predicted	18.46
Experimental	17.96
Residual	0.50
% error	2.78

4. Conclusion

In this work, RSM by Box–Behnken statistical model was used to examine the effects of four operating parameters, (feed temperature (T = 25–55˚C), vacuum pressure (P_v = 10–90 mbar), feed flow rate (Q = 15–60 mL/s), and feed concentration (C = 100–300 g/L)), on desalination from water via VMD process. This study showed that RSM is a suitable approach to optimize the operating conditions for achieving maximum water desalination via VMD process. It was shown that the predicted R^2 value of 0.9880 is in reasonable agreement with the adjusted R^2 value of 0.9944. The model indicated that feed concentration of 100 g/L, feed temperature of 55˚C, vacuum pressure of 10 mbar, and feed flow rate of 38.63 mL/s are optimum conditions for obtaining the maximum VMD permeate flux.

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