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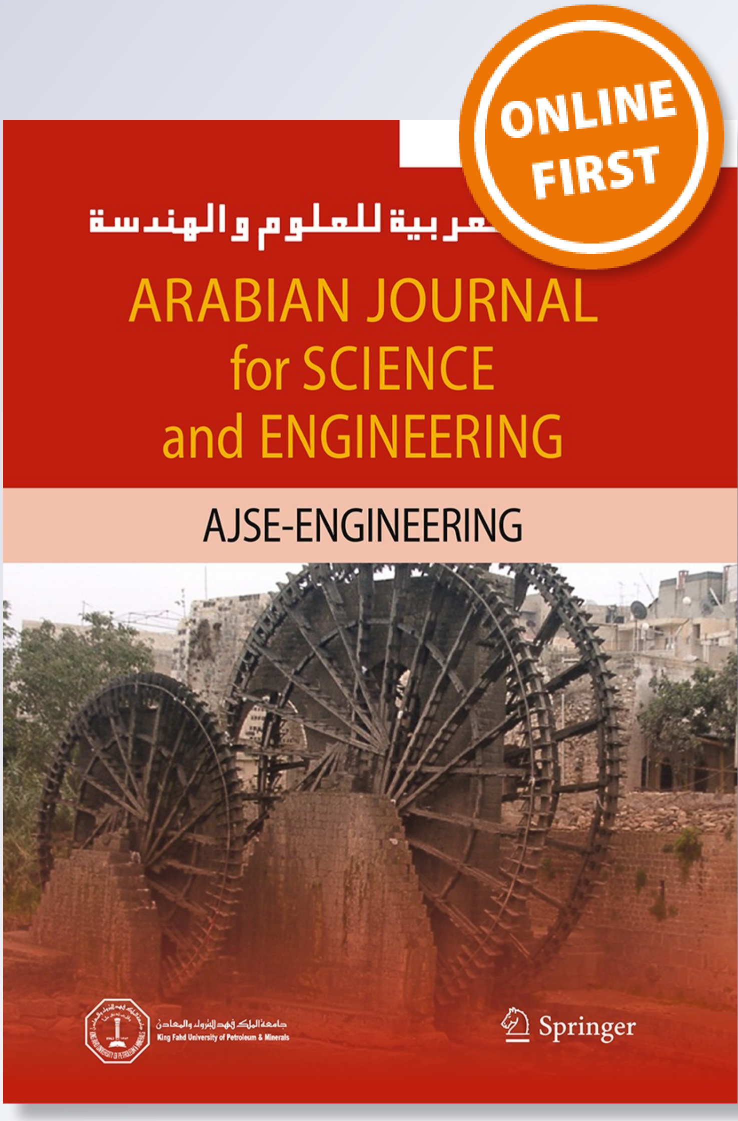

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RESEARCH ARTICLE - CHEMICAL ENGINEERING

Optimum Operating Parameters for Hollow Fiber Membranes in Direct Contact Membrane Distillation

Khalid T. Rashid[1] · **Sunarti Binti Abdul Rahman**[1] · **Qusay F. Alsalhy**[2]

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Abstract The aim of this study is the optimization of the operating conditions of PVDF-co-HFP hollow fiber membrane for direct contact membrane distillation applications. The influence of the operation parameters, such as the feed temperature (40–80 °C), the feed flow rate (0.3–0.6 L/min), and the PVP content (0–9 %), as well as the feed concentration increased from 3.5 to 5.0 wt%, and their interactions on the PVDF-co-HFP hollow fiber membrane permeate flux have been investigated. The optimum operating parameters have been specified using second-order FULL FACTORIAL and Taguchi optimization techniques to find optimum values for operation parameters in the DCMD process in order to obtain a good value of permeate flux. The results showed that PVDF-co-HFP membrane has the best performance at 21 (kg/m^2 h) when a hot feed temperature of 80°C with 0.6 L/min flow rate, 3.5 wt% NaCl feed concentration and 9 wt% PVP content in the casting solution were used. The PVP % content and inlet temperature had a significant impact on the permeate flux, while feed flow rate and feed concentration have less influence.

Keywords Optimization · Permeate flux · Hollow fiber membrane · Operating conditions · Factorial method

Abbreviations

ANOVA	Analysis of variance
Cm	Distillation coefficient
CMDC	Continuous membrane distillation crystallization
DCMD	Direct contact membrane distillation
DMAc	Dimethylacetamide
EE	Thermal efficiency
MD	Membrane distillation
PVDF	Poly (vinylidene fluoride
PVDF-co-HFP	Poly (vinylidene fluoride-co-hexafluoropropylene)
PVP	Polyvinylpyrrolidone
SWGMD	Sweeping gas membrane distillation

1 Introduction

Water desalination is a very significant challenge to obtain fresh, pure water. Seawater desalination means the removal of salt and other undesirable particles from salty water. The feed seawater is heated to raise the slope vapor pressure along the two parts of the membrane, as it is the driving force, then the feed stream molecules which are close to the membrane evaporate, and just vapor transfers through the pores of the hydrophobic membrane to condense in the permeate zone [1–3].

Previous studies suggested that there are several difficulties to be addressed in order to develop a viable DCMD operation for water treatment applications, and one of these difficulties is how to obtain an optimal operating condition

✉ Sunarti Binti Abdul Rahman
sunartirahman@gmail.com

✉ Qusay F. Alsalhy
qusay_alsalhy@yahoo.com; 80006@uotechnology.edu.iq

[1] Faculty of Chemical and Natural Recourses, University Malaysia Pahang, Lebuhraya Tun Razak, 26300 Kuantan, Pahang, Malaysia

[2] Membrane Technology Research Unit, Chemical Engineering Department, University of Technology, Baghdad, Iraq

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for the process. Choosing appropriate range of the operating conditions is critical to determining the operation of the DCMD processes with the formula that gives higher flux with minimal losses. It was concluded that more than 95 % of the energy required in the DCMD system is consumed by the heater for heating of the feed stream, so the feed temperature is one of the important operating parameters.

Broadly, in MD process an optimization dilemma is represented in maximization or minimization of some parameters (such as operating conditions). Chen et al. [4] studied the optimization of operating conditions for a continuous membrane distillation process. The study was based on the effect of three levels for each of the four parameters, with nine experiments. He assessed the feed flow rate, inlet temperature, and permeate-side flow rate and found inlet temperature as the major parameter that affects the permeate flux.

Song et al. [5] studied the optimization of spinning conditions and the effect of these conditions on the polyvinylidene fluoride (PVDF) hollow fiber membranes characterization. They found that the diameters of hollow fibers indicated significant dependence on PVDF concentration and the non-solvent content in the dope solution. They also suggested that the content of non-solvent content (i.e., glycerol) in the dope solution has the most considerable impact on both membrane distillation coefficient Cm and thermal efficiency EE, while the external coagulant composition is less effective.

Khayet et al. [6] applied a new design to optimize the sweeping gas membrane distillation operation (SWGMD), and he studied the membrane distillation flux influenced by the operating conditions, the temperature of liquid, gas temperature, liquid and gas flow rate with their interactions. Using Monte Carlo technique, the optimum operating parameters have been specified, liquid inlet temperature found to be 71.6, 17.3 °C was the gas inlet temperature, water pumping velocity of 0.16 m/s and gas flow rate of 36 L/min. The optimal distillate flux of water under these conditions was 2.789×10^{-3}(kg/m^2 s).

In order to achieve a near-zero salt discharge by changing the flow rates and operating temperatures on the feed and permeate sides together, Chen et al. [7] studied the continuous membrane distillation crystallization operation for saturated brine feed solution and also optimized the operating conditions using an orthogonal fractional factorial technique, he reported that the experiment design specified that the flow rates on the feed and permeate sides are the essential parameters controlling the CMDC performance, while the temperatures on either the feed or permeate sides are not major parameters.

To optimize the design of an operating condition of DCMD process, it is indispensable to identify which parameters have the greatest influence. So, the experiments were carried out using Taguchi experimental design. Taguchi's tactic complements two important fields; first, he obviously determines a group of orthogonal arrays, each of which is possible to utilize for many experimental cases, and second he develops a standard mode for analysis of outcome. The collection of standard experiments design methods and analysis method in the Taguchi approach gives consistency and reproducibility seldom found in another statistical technique. Analysis of experiments using Taguchi method gives allowing for several influences of parameters to be simultaneously specified, effectively and efficiently [8,9]. Using this technique, it can be dramatically minimize the time required for experimental realization. This is substantial in investigating the impacts of multiple parameters on performance as well as the influence of one factor to determine which parameter has more effect and which one has less effect [10]. Consequently, via using Taguchi method, the optimum level for each parameter is specified. With (Taguchi) method, the outcomes of the experiments are resolved, and by investigating the main impacts of each parameter, the overall trends of the affecting factors can be described. The characteristics can be controlled, such that a minimum or a maximum value of a particular parameter achieves the preferred outcome [11].

From the published literatures, it was found that there are no studies available using optimization approach to relate the operating conditions of MD process with the weight percent of the PVP as additive in the casting solution. Therefore, in this work the optimization of operating conditions in addition to the percentage of PVP content in the dope solution on the basis of Taguchi approach was discussed.

This study is aimed at optimizing various operating parameters such as feed temperature (40−80 °C), the feed flow rate (0.3–0.6 L/min), and PVP content (0–9 %) as well as the feed concentration increased from 3.5 to 5.0 wt%. Feed concentration for four hydrophobic membranes made of PVDF-co-HFP with various concentrations of PVP added was utilized for desalination of seawater by DCMD configuration using second-order FULL FACTORIAL and Taguchi optimization techniques. Meanwhile, the interaction effects between operating parameters were also studied. The performance evaluation was carried out considering the effect of different operating parameters including feed temperature, PVP % content, feed flow rate and feed concentration. DCMD runs were conducted to evaluate permeate flux. Permeate flux is known as the mass or volume (kg or L) of the water permeate collection per the membrane efficient area (m^2) per operating time (h) [12,13].

2 Experimental

2.1 Chemicals

Poly(vinylidene fluoride-co-hexafluoropropylene) PVDF-co-HFP (Mw 400,000) from Sigma-Aldrich Chemical Com-

pany was utilized as the basis membrane material. Dimethylacetamide (DMAc) has been used as a polar solvent (Sigma-Aldrich Chemical). Polyvinylpyrrolidone (PVP) (K30) (30,000 Da) purchased from Aldrich Chemical was used as a support and pore-forming additive.

2.2 Membrane

Dope solutions were prepared by dissolving PVDF-co-HFP and PVP in DMAc using different PVP concentrations (0, 5, 7 and 9 wt%) in a glass flask which keeps sealed during preparation process to avoid the penetration of air into the casting solution. Solution mixing process was carried out under the temperature of 50°C. The mixing process continues for several hours until a homogeneous solution is obtained. PVDF-co-HFP was fabricated as a hollow fiber membrane via phase inversion method. The distilled water (coagulation fluid) was pumped through the inner pipe of the spinneret via precision gear pump, whereas the PVDF-co-HFP/PVP solution flowed through a ring nozzle in the spinneret using a pure nitrogen gas. Finally, the product PVDF-co-HFP hollow fibers were immersed in water bath at 40°C for 24 h to remove the permanent DMAc solvent.

PVDF-co-HFP hollow fibers 15 cm in length were packed into a stainless steel tube, and the two ends of the bundle of fibers were sealed with epoxy resin to format a membrane module. For feed salt solution preparation, sodium chloride NaCl was utilized, with different concentration (3.5, 4.0, 4.5, and 5.0) wt%. The DCMD experimental setup schematic diagram used is illustrated in Fig. 1. The hot streams flowed through the lumen side were (0.3, 0.4, 0.5, and 0.6) L/min, while the cold permeate stream flowed through the shell side was kept at 0.25 L/min. The hot stream and cold permeate flowed concurrently through the module by means of a double-head peristaltic pump (WT600-1F- China) and their temperatures were controlled via water baths. The flow rates were monitored by in-line digital flow meters. The hot feed temperature was changed from 40 to 80°C, whereas the permeate inlet temperature was kept at 20°C. The water flux was determined from the volume gain in the cooled collecting permeate tank and recorded every 20 min. The permeation flux, J (kg/m^2 h), was calculated from the following equation [14, 15]:

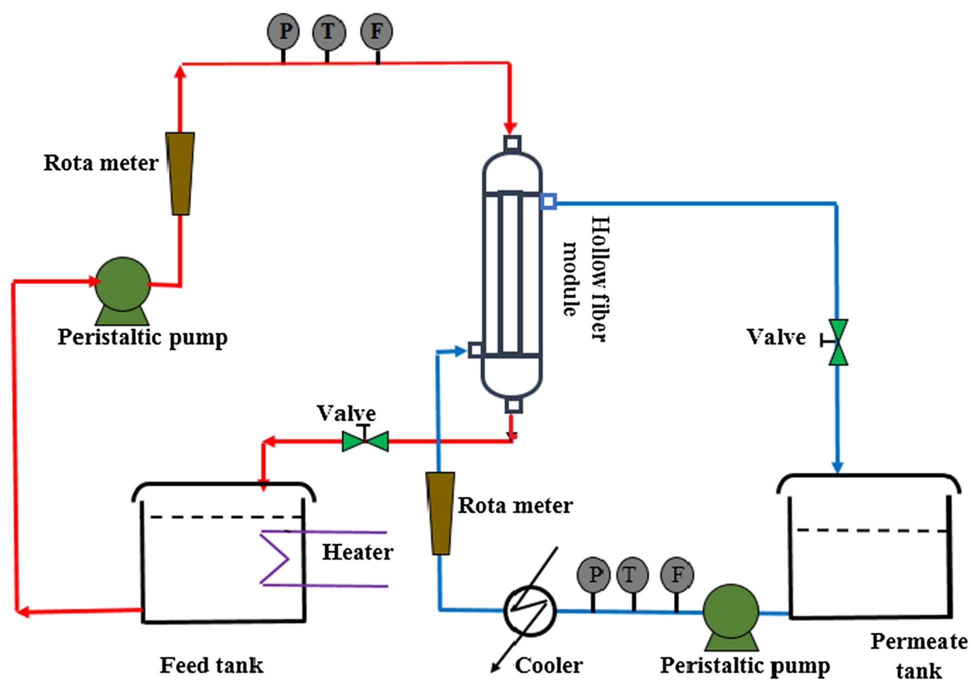


Fig. 1 Schematic DCMD experimental setup

Table 1 The values and code of input variables at various levels

Parameters	Code	Notation	Units	Levels: Low level (−)	High level (+)
Temperature	$X1$	T	°C	40	80
Flow rate	$X2$	F	L/min	0.3	0.6
Concentration	$X3$	C	g/L	35	50
PVP added	$X4$	PVP	wt%	0	9

Table 2 Design matrix for response of experimental values at deferent levels of input parameters

S.No	*X*1	*X*2	*X*3	*X*4	*X*1 Temperature (°C)	*X*2 Feed flow rate (L/min)	*X*3 Feed concentration (g/L)	*X*4 PVP content (wt%)	Y Permeate flux (kg/m^2 h)
1	+	+	+	+	80	0.6	50	9	15
2	−	+	+	+	40	0.6	50	9	5.7
3	+	−	+	+	80	0.3	50	9	14.2
4	−	−	+	+	40	0.3	50	9	5.4
5	+	+	−	+	80	0.6	35	9	21.9
6	−	+	−	+	40	0.6	35	9	6.5
7	+	−	−	+	80	0.3	35	9	13.7
8	−	−	−	+	40	0.3	35	9	5.6
9	+	+	+	−	80	0.6	50	0	4.9
10	−	+	+	−	40	0.6	50	0	0.85
11	+	−	+	−	80	0.3	50	0	4.0
12	−	−	+	−	40	0.3	50	0	0.59
13	+	+	−	−	80	0.6	35	0	5.3
14	−	+	−	−	40	0.6	35	0	1.1
15	+	−	−	−	80	0.3	35	0	4.3
16	−	−	−	−	40	0.3	35	0	0.7

Table 3 ANOVA Analysis of variance for permeate flux rate

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	10	386.111	386.111	38.6111	317.46	0.000
Linear	4	357.308	0.728	0.1819	1.50	0.330
*X*1	1	122.656	0.456	0.4559	3.75	0.111
*X*2	1	3.516	0.000	0.0003	0.00	0.963
*X*3	1	0.856	0.178	0.1776	1.46	0.281
*X*4	1	230.281	0.177	0.1767	1.45	0.282
Interaction	6	28.804	28.804	4.8006	39.47	0.000
*X*1 × *X*2	1	1.051	1.051	1.0506	8.64	0.032
*X*1 × *X*3	1	0.076	0.076	0.0756	0.62	0.466
*X*1 × *X*4	1	26.266	26.266	26.2656	215.96	0.000
*X*2 × *X*3	1	0.226	0.226	0.2256	1.86	0.231
*X*2 × *X*4	1	0.856	0.856	0.8556	7.03	0.045
*X*3 × *X*4	1	0.331	0.331	0.3306	2.72	0.160
Residual Error	5	0.608	0.608	0.1216		
Total	15	386.719				

The F-ratio can be considered of as a indicator of how various the means are relative to the variability within each sample

DF degrees of freedom, *Seq SS* sequential sum of squares, *Adj SS* adjusted sum of squares, *Adj MS* adjusted mean squares, *P* percentage of contribution, *X1* feed temperature, *X2* feed flow rate, *X3* feed concentration, *X4* percentage content of PVP content

$$\mathbf{J = Q/n\pi DL} \tag{1}$$

where *J* is the specific flux of the hollow fiber membrane (kg/m^2 h); *Q* the water flow rate reading (L/h); *n* the number of fibers in the module; *D* the outer diameter of hollow fiber (m); and *L* represents the effective length of hollow fibers (m).

3 Results and Discussion

3.1 Optimization of Operating Parameters:

Table 1 illustrates the experimental design with experimental codes of various input parameters (T, F, C, and PVP) indi-

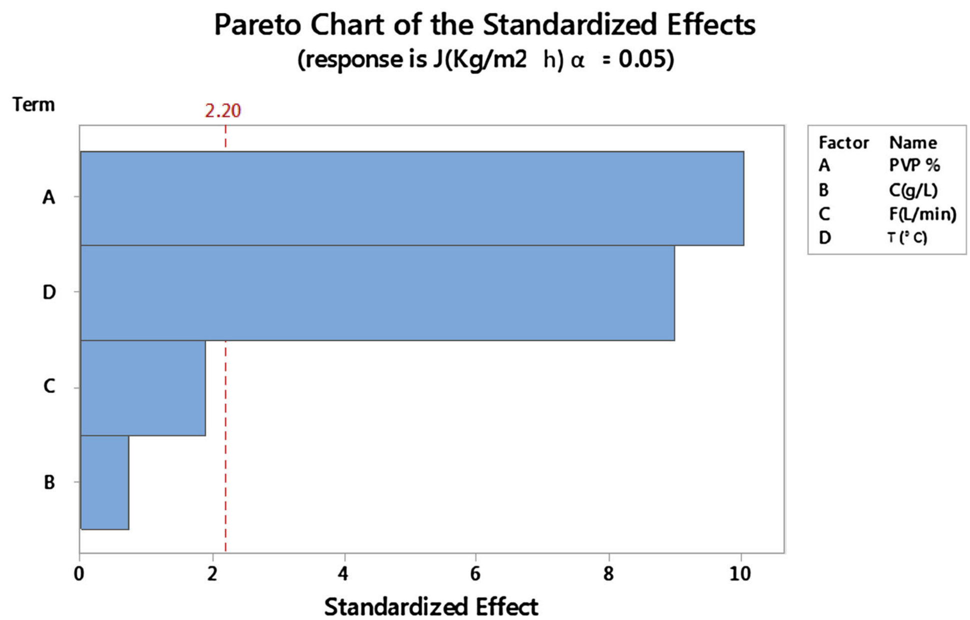


Fig. 2 Pareto chart of the standardized effects for permeate flux

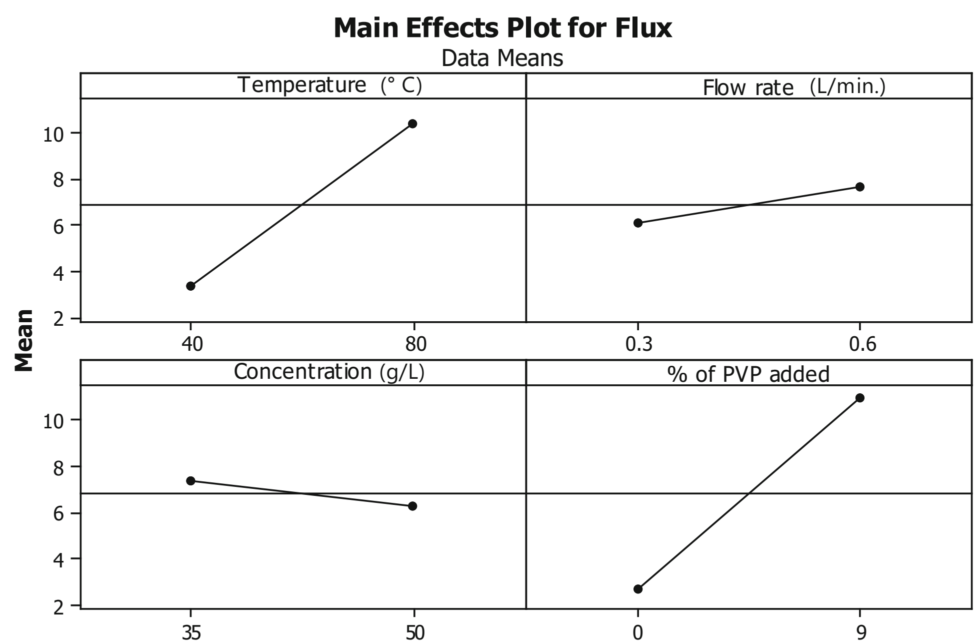


Fig. 3 Main effect plot for permeate flux

cating low and high levels along with their units. Table 2 demonstrates the matrix arrangement where 16 runs have been carried out per the details explained previously. To avoid introducing any methodical error in the optimization method, a random distribution was utilized, using second-order FULL FACTORIAL and Taguchi optimization techniques to find optimum values of operation parameters for the DCMD experimental results.

3.2 Analysis of Variance (ANOVA)

Analysis of variance (ANOVA) is a collection of statistical models technique used to examine the variations between two or more variables; more precisely, it is used to specify the influence of independent variables have on the dependent parameters in a retrogression analysis. Use of ANOVA technique can estimate the optimum collection of process

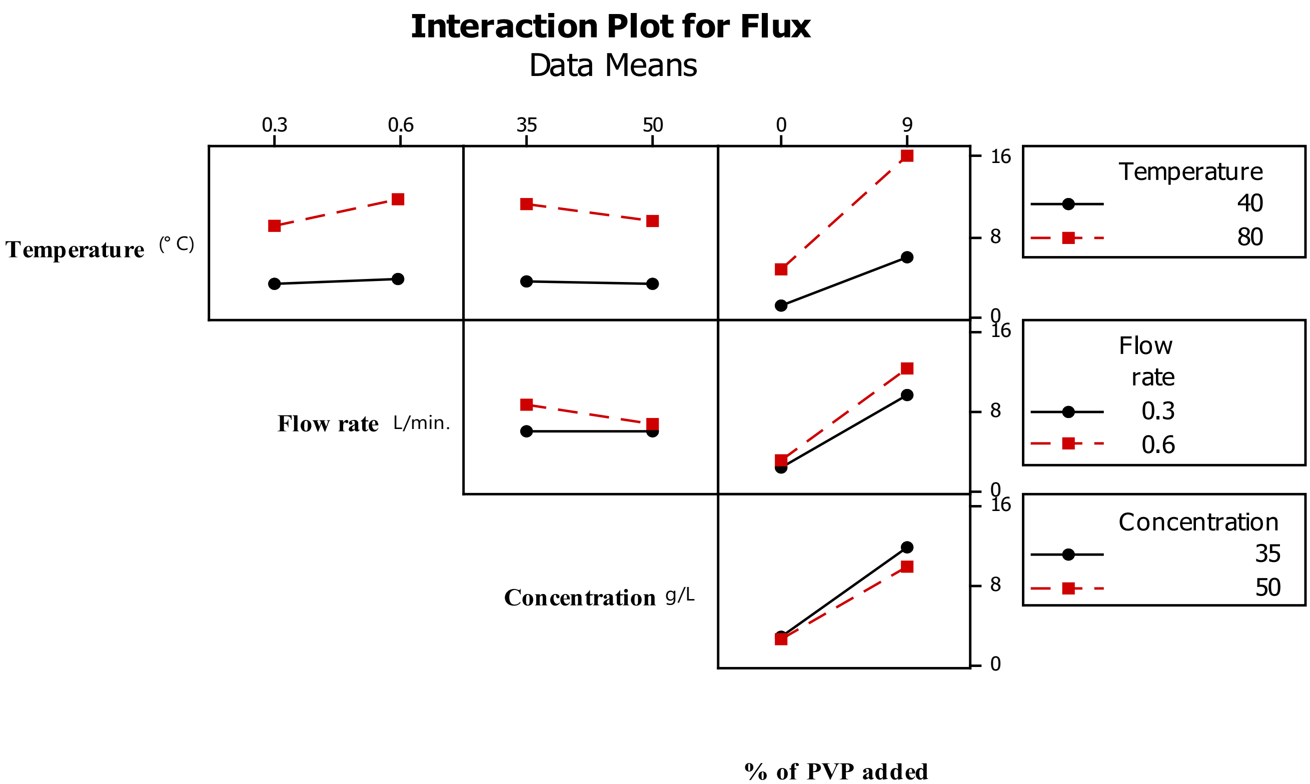


Fig. 4 Interaction plots for PVDF-co-HFP membrane permeate flux

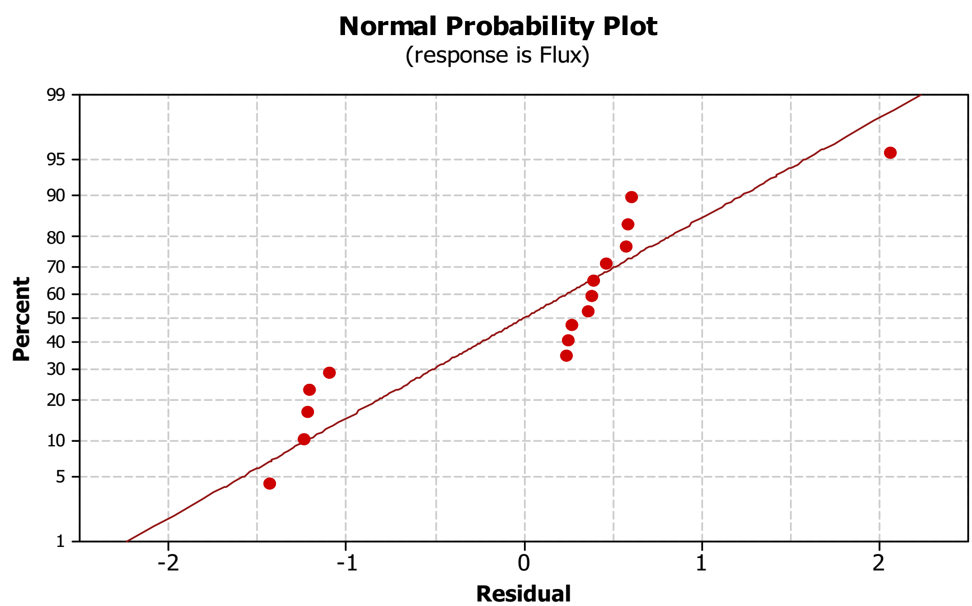


Fig. 5 Normal probability plot of residuals for permeate flux

operating parameters more strictly via testing the relative significance among the parameters. ANOVA was conducted with the help of the software package MINITAB 17 for a level of significance of 5 % to study the participation of all factors. Table 3 indicates the ANOVA results for the membrane permeate flux. In the ANOVA table, there is a *P* value for each independent parameter in the model. When the *P* value is less than 0.05, the parameter can be looked as a statistically highly considerable table [16].

3.3 Evaluating and Analyzing Results

Figure 2 illustrates the Pareto chart of the standardized effects for membrane flux, which refer to the effect of the parameters:

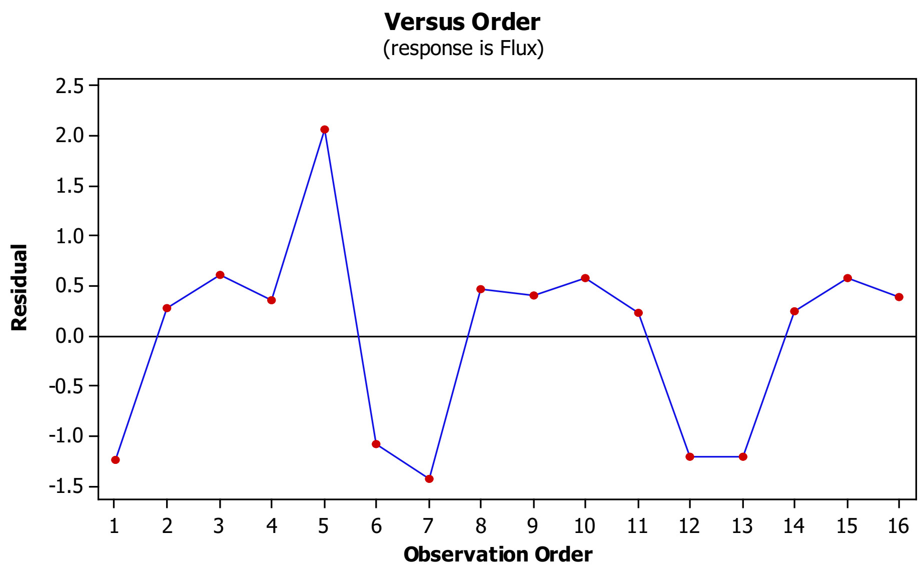


Fig. 6 Plot of residuals with the order of data

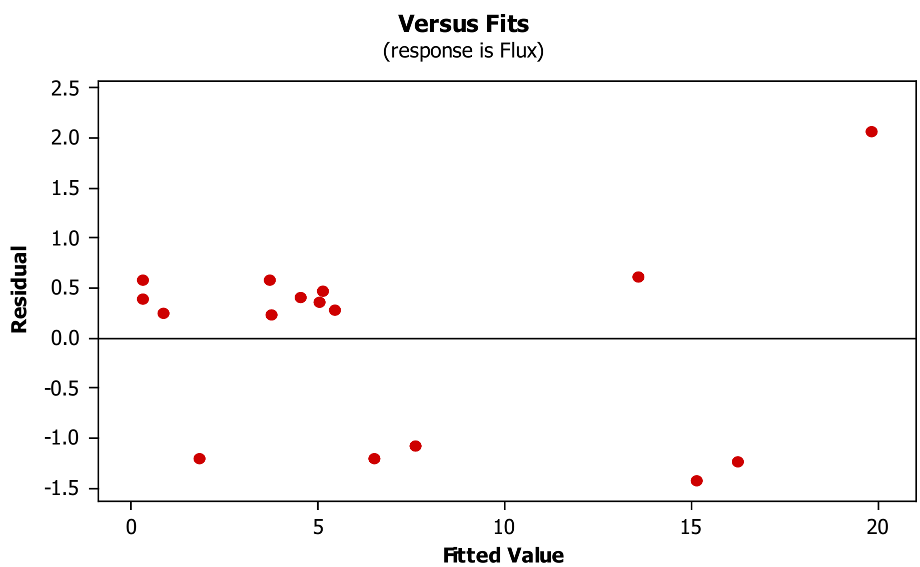


Fig. 7 The residuals with the fitted values plot

PVP added into the dope solution (A), concentration of feed solution (B), the feed flow rate (C) and the feed temperature (D) on the permeate flux (J). It can be concluded that the percentage of PVP added to the dope solution and the feed temperature play an important role in this process.

Figure 3 illustrates the mean effect for permeate flux. It can be concluded that the maximum permeate flux can be achieved when feed temperature is 80 °C, 0.6 L/min for feed flow rate, the feed concentration is 35 gm/L, and the % of PVP added to the dope solution is 9 wt%. This is in line with the findings of Shirazi [17] who concluded that the feed temperature and feed flow rate have a more significant influence on the membrane permeate flux than the other operating conditions.

3.4 Parameters Interactions and Normal Probability Plot

Figure 4 demonstrates the interaction between the parameters. It can be seen that the lines which represent the temperature, flow rate, concentration and PVP content tend to cross one another, so it indicates that there is a considerable interaction effect occurring between parameters. Experiments including the interaction may be required to optimize the DCMD process.

The normal probability plot is a graphical method for evaluating whether or not a data set is almost normally distributed. Data are plotted versus theoretical normal distribution in the way in which the points should take shape on an

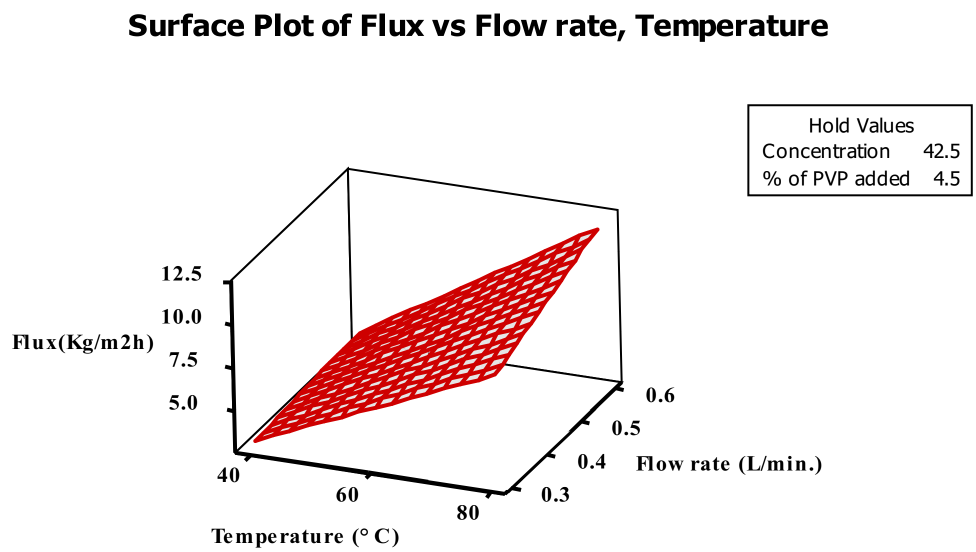


Fig. 8 A three-dimensional response surface plot of the expected permeate flux as a function of feed temperature and feed flow rate at constant fed concentration and PVP added

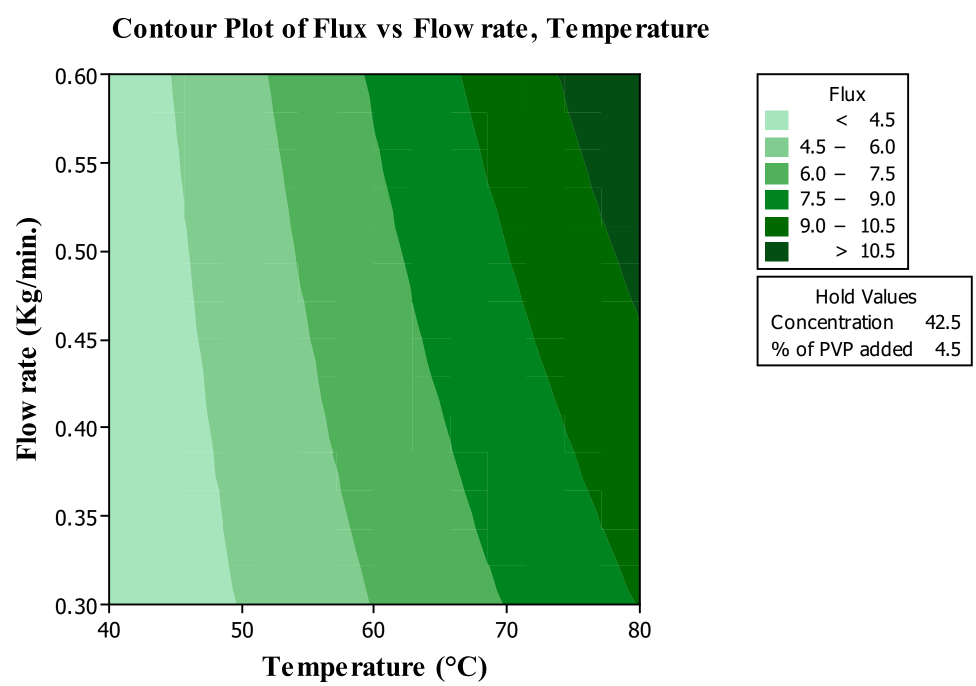


Fig. 9 A contour plot corresponding to the response surface in Fig. 6

approximate straight line. In other words, points that are away from the straight line mean they are away from the normality. Using computer software MINITAB, normal probability charts were created. Normal probability plots are demonstrated in Fig. 5 which represents the difference between the real value and the value estimated from the regression model (regression line), and the desired advantage of this figure is to find out how the approaching of the actual data extent of the suggested model. It can be observed from the plotted data that all data residuals have an almost normal distribution because the overall points form an approximate straight line.

3.5 Analysis Independence and Equivalence of Difference Assumptions

The errors do not rely on each other. The errors are independent of each other. The better method to be free of errors is to run the order of the experimental trials randomly, as illustrated in Fig. 6. The error difference does not allow for uneven levels of factors. In another meaning, the error difference does not alter according to the values of the predicted response, as shown in Fig. 7.

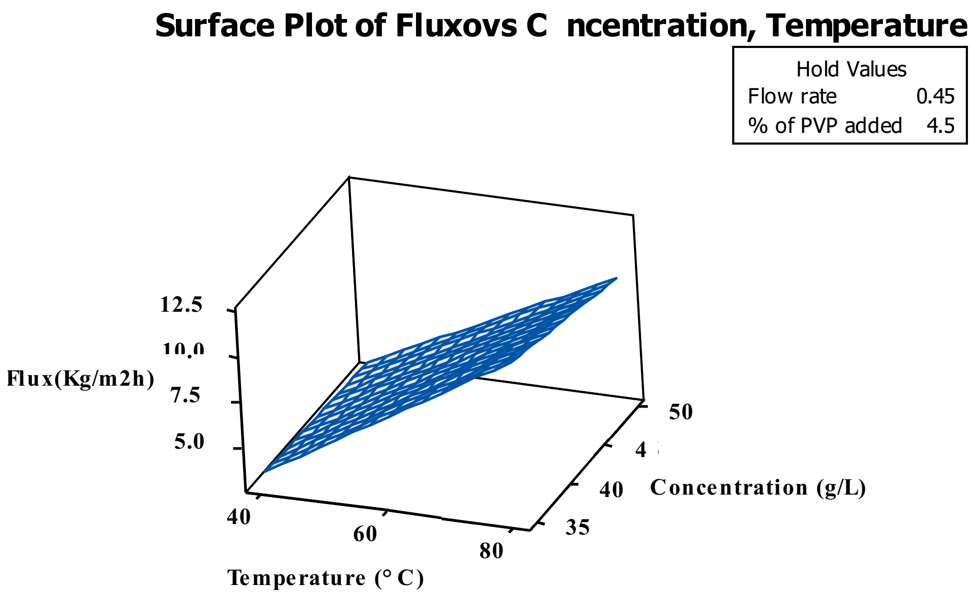


Fig. 10 A three-dimensional response surface plot of the expected permeate flux as a function of feed temperature and feed concentration at constant fed flow rate and PVP added

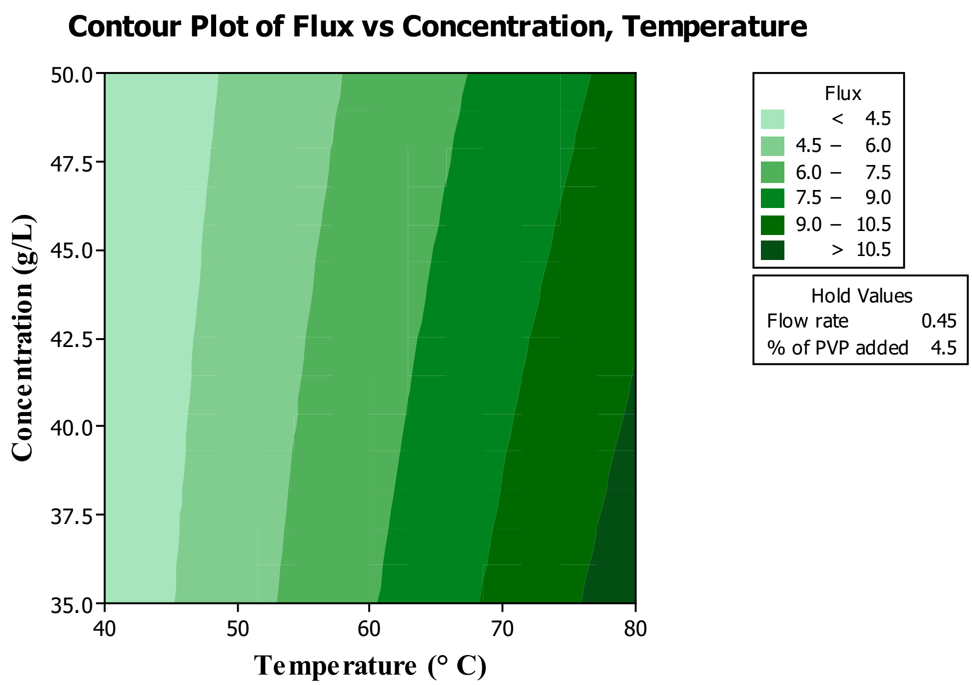


Fig. 11 A contour plot corresponding to the response surface in Fig. 8

Response of membrane permeate flux can be represented as a solid surface in a three-dimensional space, where the response (Flux) is plotted against the limits of two process parameters. The essential goal of the response surface plot is to specify the optimum operating parameters represented by the feed temperature, feed concentration, amount of PVP added and feed flow rate, which result in the maximum permeate flux desired. Via utilization of the MINITAB 17 computer software, the three-dimensional response surface plots created from the fitted model are described in Figs. 8, 10 and 12. The symmetrical three-dimensional contour plots in Figs. 9, 11, and 13 demonstrate the corresponding three-dimensional contour plots. These figures show the magnitude impact of each parameter for operating condition, feed temperature, concentration of feed stream, amount of PVP added and feed flow rate on the PVDF-HFP membrane flux.

3.6 Model Description and Optimization of Membrane Permeate Flux

The operating parameters such as feed temperature ($X1$), feed flow rate ($X2$), feed concentration ($X3$) and PVP % content ($X4$) were optimized Via MINITAB computer software method (*MINITAB* software version 17) in order to reach the

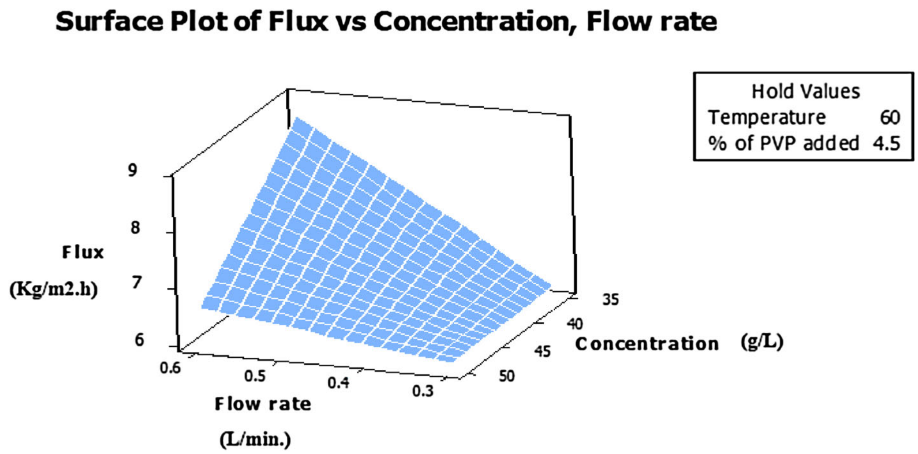


Fig. 12 A three-dimensional response surface plot of the expected permeate flux as a function of feed concentration and feed flow rate at constant fed temperature and PVP added

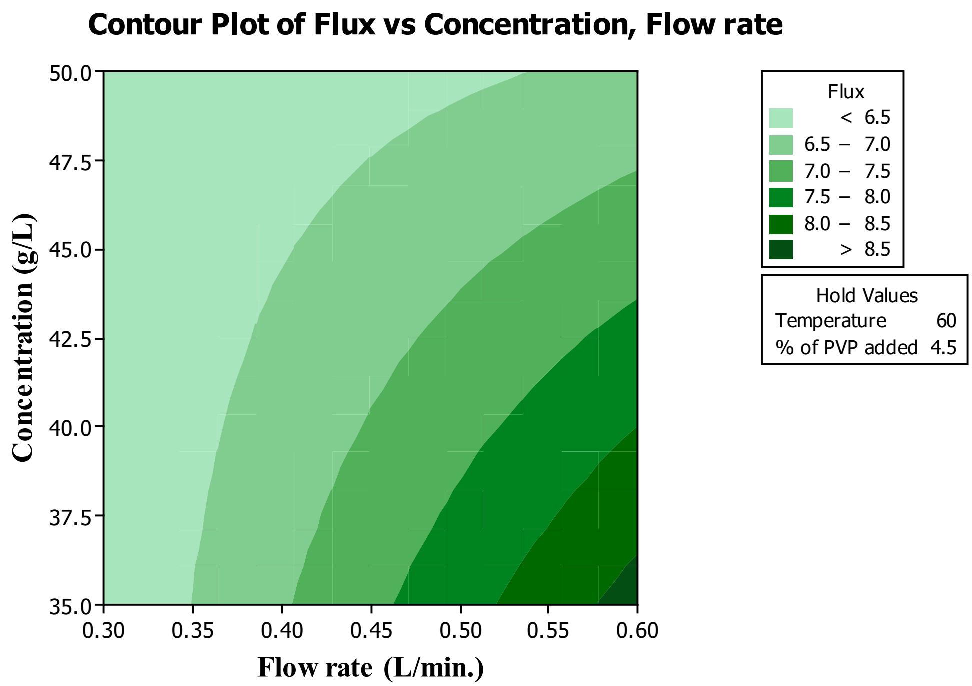


Fig. 13 A contour plot corresponding to the response surface in Fig. 10

optimum values of operating conditions to enhance the permeation flux of the PVDF-co-HFP hollow fiber for direct contact membrane distillation.

The MINITAB technique has several modes to solve the models; one of these modes is linear equation programming, and linear equation programming can be solved using graphical method or algebraic method.

The mathematical form of linear programming is:

$$\text{Max (Min) } Y = A1\,X1 + A2X2 + \cdots + AiXi + \cdots + nXn \quad (2)$$

where Y is the objective function (permeate flux), Xi is the affecting variable, Ai is a numerical coefficient, $i = 1 \ldots n$

Using the MINITAB technique, the following model equation for permeates flux was obtained:

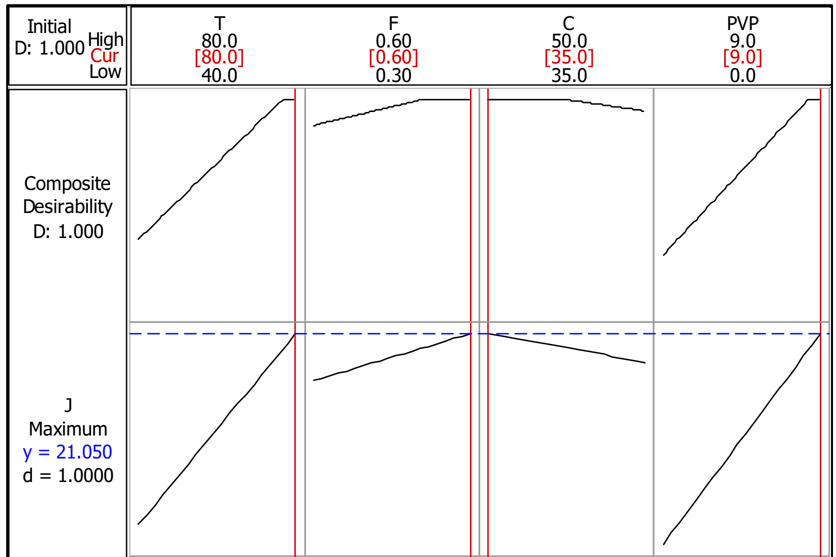
$$Y = -4.03681 + 0.11389X1X2 + 0.01898X1X4 + 0.34259X2X4 \quad (3)$$

This equation with the following coefficient:

$$\text{S} = 1.66335 \quad \text{PRESS} = 141.656$$
$$\text{R-Sq} = 97.56\,\% \quad \text{R-Sq(pred)} = 74.97\,\%$$
$$\text{R-Sq(adj)} = 92.67\,\%$$

Figure 14 clarifies the optimization design for the membranes permeate flux with different levels for four factors (T, F, C and PVP). Via MINITAB17 computer software, the optimization chart is created by using the information in Table 2. The optimization outcome appears in the left column, while the optimum tuning of each parameter is illustrated at the middle of the top row. It can be observed that

Fig. 14 Optimization chart of process parameters for maximum membrane flux



T=Feed temperature (° C), F= Feed flow rate (L/min.), C=Feed concentration (g/L), PVP= wt. % of PVP content, J= Permeate flux (Kg/m^2.h)

the feed concentration and feed flow rate showed small effect on permeate flux, whereas both feed temperature and PVP % content displayed influence dramatically. The parameters impact on permeate flux is clear and have the following arrangement from the most to least effect: PVP % content > feed temperature > feed flow rate > feed concentration. The behavior curve of each factor is clear underneath. The illustration shows that an optimum operation at feed temperature is (80 °C), PVP content (9 wt%), feed salt concentration is (35 g/L) and feed flow rate is (0.6 L/min), which would result in optimal permeate flux about (21 kg/m^2 h).

4 Conclusion

FULL FACTORIAL and Taguchi optimization techniques were used to identify the operating parameters that affect the permeate flux of PVDF-co-HFP hollow fiber membrane in DCMD process. Feed temperature, feed flow rate, PVP content and feed concentration were identified as the four factors whose impact on the water permeate flux was to be evaluated. The experimental design allows studying the effect of four levels for each of the four factors with sixteen experiments. The extent analysis of the results for these experiments specified the feed temperature and PVP % content as the most efficacious factors that affect the water permeate flux. The design also permitted assigning the optimal values of the four parameters based on the sixteen experiments. The optimum parameters were feed temperature (80 °C), PVP content (9 wt%), feed NaCl concentration (35 g/L) and feed flow rate (0.6 L/min). Under these conditions, a maximum permeate flux of (21 kg/m^2 h) was achieved.

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