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MODELING OF PREPARATION CONDITIONS OF PES ULTRAFILTRATION HOLLOW FIBER MEMBRANES USING STATISTICAL REGRESSION TECHNIQUES

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ABSTRACT

Mathematical modeling of the spinning process is crucial for a better understanding of the process variables and process functionality in membrane development. Due to the broad use and key importance of mathematical models in chemical process engineering, experimental design is becoming essential for the rapid development and validation of these empirical models. This work used the design of experiment methodology and aimed to predict the performance of ultrafiltration systems for water treatment by considering the statistical regression technique as an important approach for modeling flux. The utilization of regression modeling was also explored to show the principle elements for predicting flux in the spinning process. In order to investigate how proficient the statistical regression technique is at approximating the predicted value for flux, a real spinning experiment was conducted in this study. In this experiment, 30 samples of data were collected based on a half fractional factorial experiment with design resolution *V*, as well as 4 replications of center points and 10 axial points. The spinning factors that were investigated are the dope extrusion rate, air gap length, coagulation bath temperature, bore fluid ratio, and post-treatment time for predicting the corresponding flux. The regression model obtained shows that there is a correlation between the experimental data and predicted values. The results of the proposed model can be used to give a good prediction of the spinning process during membrane fabrication.

Keywords: modeling, design of experiment methodology, statistical regression technique, membrane separation.

INTRODUCTION

Nowadays, membrane separation has become one of the most effective and demanding techniques used to fulfill the demands in numerous industrial processes based on separation. The ability that these membrane technologies have in separating multicomponent compositions into two or more preferred outputs has allowed them to become a more popular choice, considering their potentials and benefits. The advances made by Loeb and Sourirajan with high-flux asymmetric membranes have led to the further development of membrane separation techniques. Since then, this technology has attracted much attention and support for research (Wiesner and Chellam, 1999). Membrane technologies have been a better option compared to conventional separation processes because they are more economical since they possess low capital investment, low energy consumption, and low operating cost. They are also environmentally friendly and yield superior product quality. Membrane technology has grown to become the most demanded method of separation. Thus, membrane separation technology can be applied to fulfill many of the separation demands of the process industries such as gas purification, water purification, wastewater treatment, hemodialysis, and numerous other types of application.

Among membrane modules, hollow fiber configuration is more favorable for industrial applications mainly due to its high module packing per unit area, thus providing higher productivity in a single membrane unit. Hollow fiber membrane is commonly fabricated via a spinning process which involves the extrusion of polymer solution from an annular spinneret. Inherently, phase inversion between solvent and non-solvent prevails concurrently on the bore side, and shell surfaces of the hollow fiber membranes are strongly influenced by the mass transfer of a strong precipitant like water (Pesek and Koros, 1994). As a result, the fabrication of membranes by phase inversion method could be completed with the assistance of thermodynamics or precipitate kinetic concepts in the polymer solution.

A model description of a system design for spinning parameters using mathematical models would be valuable for the future development of membranes. In addition, the first necessary step for process parameters optimization in the spinning process is to understand the fundamental principles governing the spinning process by developing an explicit mathematical model, which may be of two types: mechanistic and empirical (Box and Draper, 1986). The functional relationship between input-output and in-process spinning parameters, as determined analytically in the spinning process, is called mechanistic modeling. Nevertheless, due to lack of acceptable mechanistic models and information, as well as rare literature availability on the spinning process, empirical models are commonly applied in the spinning process. To simulate such a dynamic process, mathematical modeling based on the systematic design of experiment (DOE) method shows an effective and widely used tool which increases the speed of the development cycle time, improves reliability, reduces process variability, and increases overall product quality (Yong and Hahn, 2007). Although these models mostly do not represent the accurate spinning mechanisms of the phenomena, they are helpful in getting a rooted understanding of the fundamental processes or allowing suggestions for future experiments. In addition, if the number of model variables is not adequate, the model might be unfit for the data. It is because a good model should trade-off between being as simple as possible, but still managing to maintain a good approximation of the observed measurement data (Posada and Buckley, 2004). There are a limited number of models available for the analysis of the spinning process in membrane fabrication. Therefore, it is still difficult to acquire the quantitative prediction of filtration performance with certain different designs and operational settings. In addition, the fabrication of polymer hollow fiber membranes with a consistently high level of performance remains a complex challenge due to the fact that their morphologies, mechanical properties, and performances are influenced by various spinning conditions (Qin *et al*., 2005; Setiawan *et al*., 2011; Khayet *et al*. 2002). The common method used to characterize the resulting membrane and its performance, is to measure the Pure Water Permeability (PWP) flux (Winston and Sirkar, 1992).

Currently, statistical regression techniques have become the chosen method and they are most commonly used to create a model for the spinning process. Regression is a conceptually simple technique for investigating the functional relationship between output and input decision variables of the spinning process and may be useful for the spinning process data description, parameter estimation, and control (Shwehdi *et al*., 1998). Several applications of regression equation-based modeling in spinning processes are reported in the literature. For example, Gorji *et al*. (2012) proposed some regression models to describe polyurethane nanofiber membranes for protective clothing applications. The regression analysis revealed very clearly the performance of nanofiber membranes. Tan *et al*. (2005) claimed that a statistical regression model might make a decent temperature profile prediction in typical spinning operations of mixed-conducting ceramic hollow fiber membranes. Malaisamy *et al*. (2000) have demonstrated that the separation factors of PSF ultrafiltration membranes were in good correlation with the predicted values using a second order polynomial regression model. Also, the potential of statistical regression techniques as a better modeling technique for flux prediction was stated by Lee *et al*. (1995) in that a statistical regression model could give a better prediction in typical spinning operations of PSF hollow fiber membranes. In this respect, by advancing the state-of-the-art technology in this field and the modeling of spinning conditions, statistical regression techniques seem to be the promising alternative for further investigation of the spinning process and related membrane processes. Statistical regression models are superior compared to other modeling techniques in identifying possible causal relationships. In statistical regression, it is possible to determine which variables are most strongly predictive of an outcome. Furthermore, through a stepwise variable selection process, it is possible to eliminate a number of independent variables that are not related to a particular outcome of interest. Based on previous research, it clearly shows that the statistical regression technique has been successfully applied to modeling flux in the spinning process and membrane process respectively. It also shows that this technique usually produces a better prediction result compared to the output attained with the real experiment. The potential of statistical regression technique proves to be a better modeling technique because the regression model presents a higher accuracy rate for predicting flux in the spinning process. Statistical regression models help to produce slightly more precise prediction values compared to the conventional model. Essentially, statistical regression techniques have been proposed to reduce experimental work with maximal information outcomes, resulting in the development of a regression model for analyzing the performance of membranes prepared with different spinning conditions.

Statistical regression is indeed an interesting approach to developing mathematical models. However, statistical regression that involves simultaneously the parameters of dope extrusion rate, air gap length, coagulation bath temperature, bore fluid ratio and post-treatment time has yet to be investigated. Hence, the present study focused on developing a statistical regression model for the prediction of flux in the spinning process based on statistically designed experiments. The experiments were operated using a hollow fiber membrane module under constant flux mode with periodic backwashing and distilled water was used as feed water. The collected spinning parameters and experimental outcomes were used as a training database for the regression model.

EXPERIMENTAL WORK

Materials

The polyethersulfone (PES) pellets, purchased from Solvay Advanced Polymer Company, were used as the main membrane forming materials. 1-methyl-2-pyrrolidone (NMP), purchased from Merck Darmstadt Germany, was used as a solvent to dissolve the polymer without further purification. Polyethylene glycol (PEG 400), purchased from Fluka Milwaukee, was used as an additive to enhance PES membrane properties.

PES hollow fiber membranes preparation

The polymer solution comprising 15.25% PES concentration, 66.43% 1-methyl-2-pyrrolidone (NMP), 14.3% polyethylene glycol (PEG), and 4.02% water was prepared using vigorous mixture. Asymmetric PES hollow fiber membranes were fabricated in spinning equipment (Figure-1) using a spinneret with dimensions of 1100 μm (outer diameter) and 550 μm (inner diameter).

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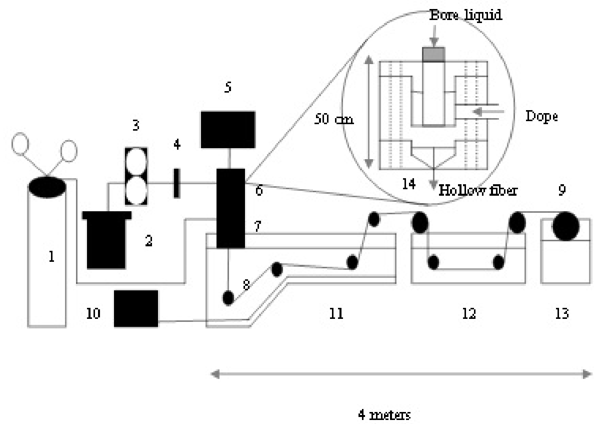

Figure-1. Hollow fiber spinning system: 1) nitrogen cylinder; 2) dope reservoir; 3) gear pump; 4) on-line filter, 7 mm; 5) syringe pump; 6) spinneret; 7) forced convective tube; 8) roller; 9) wind-up drum; 10) refrigeration/heating unit; 11) coagulation bath; 12) washing/treatment bath; 13) wind-up bath; 14) schematic spinneret (Ismail, 1997).

The dope reservoir was connected with a N_2 gas cylinder. The process started when the polymer solution and a bore liquid mixture were simultaneously extruded through a tube-in-orifice. When the polymer solution was extruded through a tube in the spinneret orifice, shear stress was induced within the thin annular of the spinneret at a controlled rate. The fibers passed through an air gap of specific length before entering a coagulation bath with a controlled temperature. Tap water was used as an external coagulant, while the mixtures of pure water and NMP with different ratios were used as the bore fluid. The nascent hollow fibers were taken up by a roller at a free falling velocity and kept stored in a water bath for at least 2 days in order to remove any residual solvent. Then, the membranes were collected and treated subsequently for the removal of residual solvent. In order to minimize the membranes shrinkage during the drying process at ambient conditions, the fibers were submerged in the methanol solution for specific durations. During this process, the water in the membrane pores was gradually replaced with the exchanged reagents, which have a lower surface tension. The membranes were subsequently dried at room temperature before characterization.

Measurement of hollow fiber membranes water flux

In preparation of the modules, the hollow fiber membranes were subsequently potted into bundles consisting of 120 fibers of approximately 22cm in a U shape prior to flux measurement. The water flux experiments were conducted in a cross flow filtration set-up. The water flux rig comprised a pump, feed holding tank, and the ultrafiltration hollow fiber module. The feed was pumped at 1bar transmembrane pressure (TMP) into the shell side of the module and the permeate came out from the lumen of the fiber. For each batch of hollow fibers, a total of 30 bundles of fibers were potted for testing 30 different spinning condition combinations. In this way, a test exhibiting the importance of the replication error in comparison to the model dependent error can be implemented. The flux was calculated by taking the average of three readings, which were collected at regular intervals. These readings consisted of noting the volume of water collected in 5 minutes for each condition. Thus, the average flux data were reported. The specific flux of the membranes was computed using the formula:

$$J = \frac{Q}{\Delta P \times A} = \frac{Q}{n\pi Dl\Delta P} \tag{1}$$

Q = water flux reading (L/h)
ΔP = pressure difference between the feed side and the permeation side of the membrane (atm)
A = effective membrane surface area (m^2)
n = number of fibers in the module
D = outer diameter of the hollow fiber membrane (m)
l = effective length of the hollow fiber membrane (m)

Design of experiment

Statistical regression techniques were applied to investigate their potential usefulness in providing estimates on flux and in identifying the parameters most critical in controlling such flux. An experimental task was done to gather experimental data to examine the spinning conditions that contributed to the flux performance of ultrafiltration hollow fiber membranes. It covered the whole process involved in hollow fiber membranes fabrication. The work was related to the development of a mathematical model for flux to describe the relationship between the independent spinning process variables (spinning conditions) and dependent variable (flux) in the spinning process. A half fractional factorial experiment with design resolution *V*, as well as 4 replications of center points, and 10 axial points integrated with the regression technique were used in developing the flux model in relation to the primary spinning decision variables which were dope extrusion rate (DER), air gap length (AGL), coagulation bath temperature (CBT), bore fluid ratio (BFR), and post-treatment time (PT). Two levels of each factor were selected for the experiment, the units and notations are given in Table-1.

These spinning conditions were chosen based on typical operating conditions of the system recommended for this operation and available literature. Center points experiment has two important roles in order to check the reproducibility and stability of results. First, it allows the experimenter to obtain an estimate of the experimental error. Second, if the sample mean is used to estimate the effect of a factor in the experiment, then, the center points permit the experimenter to obtain a more precise estimate of the effect. In this study, the order of the experiment has been generated randomly because Analysis of Variance (ANOVA) requires the observations or errors to be independently distributed random variables. The runs were conducted in a randomized manner to prevent systematic bias. By properly randomizing the experiment, the effects of extraneous factors or confounding variables that might be present were averaged out. A confidence level of 95%

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(α = 0.05) was used throughout the analyses of the experimental results and the Fisher's F-test verified the statistical significance of the model.

Table-1. Spinning condition values for real spinning process.

Spinning	Units	Level in coded form		
variables		-1	0	1
DER, (A)	cm^3/min	2	4	6
AGL, (B)	cm	0	1	2
CBT, (C)	°C	18	24	30
BFR, (D)	NMP/H_2O, wt.%	0/100	35/65	70/30
PT, (E)	h	2	4	6

A total of 30 experiments were performed as illustrated in Table-2. Design Expert 6.0.5 software was utilized for analyzing the data.

RESULTS AND DISCUSSION

Development of the flux regression model

All the conditions were tested in the real ultrafiltration process to display the actual value of flux. The outcomes of the experiment are presented in Table-2.

Table-2. Experimental results for spinning experiments.

		Values of spinning conditions					Response
Std. order	Run order	A	B	C	D	E	Flux (L/m^2.h)
1	4	2	0	18	0/100	6	35.76
2	13	6	0	18	0/100	2	4.07
3	7	2	2	18	0/100	2	10.31
4	5	6	2	18	0/100	6	5.25
5	19	2	0	30	0/100	2	26.37
6	16	6	0	30	0/100	6	28
7	1	2	2	30	0/100	6	40.42
8	8	6	2	30	0/100	2	4.05
9	2	2	0	18	70/30	2	0.26
10	11	6	0	18	70/30	6	2.68
11	12	2	2	18	70/30	6	31.17
12	3	6	2	18	70/30	2	21.36
13	18	2	0	30	70/30	6	7.63
14	14	6	0	30	70/30	2	15.90
15	10	2	2	30	70/30	2	35.19
16	20	6	2	30	70/30	6	25.11
17	15	4	1	24	35/65	4	23.92
18	9	4	1	24	35/65	4	24.39
19	6	4	1	24	35/65	4	22.27
20	17	4	1	24	35/65	4	25.31
21	30	2	1	24	35/65	4	27.28
22	25	6	1	24	35/65	4	17.57
23	28	4	0	24	35/65	4	24.75
24	26	4	2	24	35/65	4	31.24
25	23	4	1	18	35/65	4	24.75
26	22	4	1	30	35/65	4	34.98
27	24	4	1	24	0/100	4	24.99
28	21	4	1	24	70/30	4	26.49
29	29	4	1	24	35/65	2	23.30
30	27	4	1	24	35/65	6	30.08

Table-3. Resulting ANOVA table for surface quadratic model for flux.

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Block	333.42	1	333.42			
Model	3090.28	20	154.51	114.57	< 0.0001	sig.
A	454.01	1	454.01	336.63	< 0.0001	
B	191.30	1	191.30	141.84	< 0.0001	
C	373.92	1	373.92	277.25	< 0.0001	
D	10.02	1	10.02	7.43	0.0260	
E	236.82	1	236.82	175.60	< 0.0001	
A^2	66.65	1	66.65	49.42	0.0001	
B^2	0.26	1	0.26	0.19	0.6730	
C^2	11.70	1	11.70	8.67	0.0186	
D^2	9.01	1	9.01	6.68	0.0323	
E^2	2.32	1	2.32	1.72	0.2260	
AB	109.99	1	109.99	81.55	< 0.0001	
AC	3.60	1	3.60	2.67	0.1409	
AD	242.50	1	242.50	179.81	< 0.0001	
AE	46.21	1	46.21	34.26	0.0004	
BC	0.15	1	0.15	0.11	0.7472	
BD	907.97	1	907.97	673.23	< 0.0001	
BE	0.80	1	0.80	0.59	0.4643	
CD	14.23	1	14.23	10.55	0.0117	
CE	23.06	1	23.06	17.10	0.0033	
DE	312.85	1	312.85	231.97	< 0.0001	
Residual	10.79	8	1.35			
Lack of Fit	5.92	5	1.18	0.73	0.6467	not sig.
Pure Error	4.86	3	1.62			
Cor Total	3434.49	29				

Std. Dev.	1.16	R-Squared	0.9965
Mean	21.83	Adj R-Squared	0.9878
C.V.	5.32	Pred R-Squared	0.8125
PRESS	581.35	Adeq Precision	40.083

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Table-4. Resulting ANOVA table for reduced quadratic model for flux.

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Block	333.42	1	333.42			
Model	3083.37	15	205.56	151.03	< 0.0001	sig.
A	454.01	1	454.01	333.58	< 0.0001	
B	191.30	1	191.30	140.55	< 0.0001	
C	373.92	1	373.92	274.74	< 0.0001	
D	10.02	1	10.02	7.36	0.0177	
E	236.82	1	236.82	174.00	< 0.0001	
A^2	80.36	1	80.36	59.05	< 0.0001	
C^2	11.18	1	11.18	8.21	0.0132	
D^2	12.11	1	12.11	8.90	0.0106	
AB	109.99	1	109.99	80.81	< 0.0001	
AD	242.50	1	242.50	178.18	< 0.0001	
AE	46.21	1	46.21	33.95	< 0.0001	
BD	907.97	1	907.97	667.13	< 0.0001	
CD	14.23	1	14.23	10.46	0.0065	
CE	23.06	1	23.06	16.95	0.0012	
DE	312.85	1	312.85	229.86	< 0.0001	
Residual	17.69	13	1.36			
Lack of Fit	12.83	10	1.28	0.79	0.6613	not sig.
Pure Error	4.86	3	1.62			
Cor Total	3434.49	29				

Std. Dev.	1.17	R-Squared	0.9943
Mean	21.83	Adj R-Squared	0.9877
C.V.	5.34	Pred R-Squared	0.9557
PRESS	137.29	Adeq Precision	46.471

Table-3 shows the ANOVA table responses for the surface quadratic model for flux. The value of “Prob > F” in Table-3 for the model is less than 0.05, which indicates that the model is significant. This is desirable because it indicates that the terms in the model have a significant effect on the flux. In the same manner, all the main effects of DER (*A*), AGL (*B*), CBT (*C*), BFR (*D*), PT (*E*), the second order effects of A^2, C^2, D^2, and the two-level interactions of *AB*, *AD*, *AE*, *BD*, *CD*, *CE*, *DE* are significant model terms. Other model terms are considered to be not significant. These insignificant model terms (not counting those required to support hierarchy) can be removed and this may result in an improved model. By selecting the backward elimination procedure to automatically reduce the terms that are not significant, the resulting ANOVA table for the reduced quadratic model for flux is shown in Table-4. Results from Table 4 indicate that the model is still significant. All the main effects of DER (*A*), AGL (*B*), CBT (*C*), BFR (*D*), PT (*E*), and the two-level interactions of *AB*, *AD*, *AE*, *BD*, *CD*, *CE*, *DE*, as well as the second order effects of A^2, C^2, D^2 are significant model terms. The lack-of-fit is still shown to be insignificant. The R^2 value is high, close to 1, which is desirable. The predicted R^2 is in agreement with the adjusted R^2. The adjusted R^2 value is particularly useful when comparing models with a different number of terms. However, this comparison is done in the background, when model reduction is taking place. Adequate precision compares the range of the predicted values at the design points to the average prediction error. Ratios greater than 4 indicate adequate model discrimination. In this particular case, the value is well above 4.

Figure-2 displays the normal plot of residuals for the model. The normal probability plot shows that the residuals are normally distributed along the normal probability line. It means that the error distribution is approximately normal for all series of data, which implies that the model is adequate. Figure-3 exhibits the studentized residuals versus predicted values in which all data are shown to be in the range, and no abnormal trend exists.

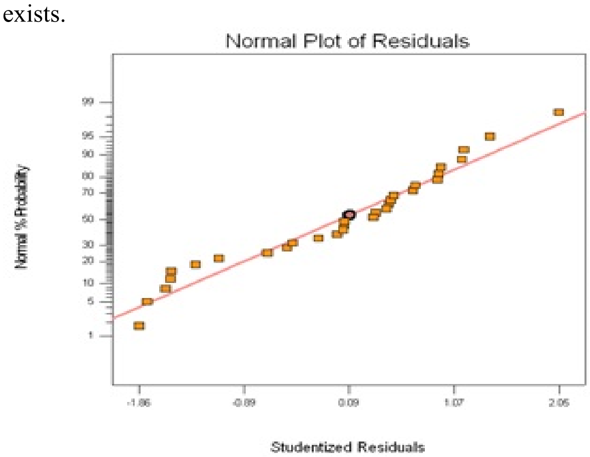

Figure-2. Normal probability plot of residuals for flux.

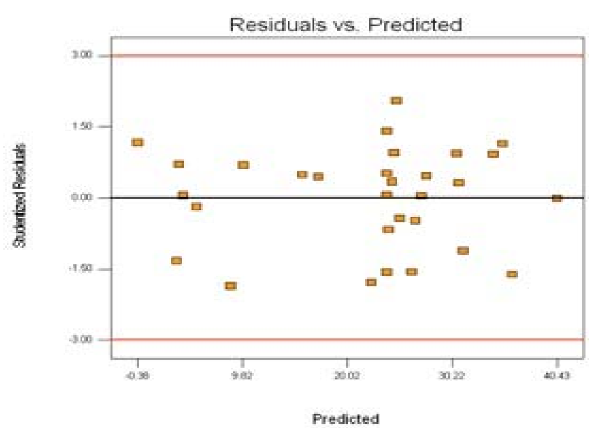

Figure-3. Plot of residuals vs. predicted responses for flux.

The final empirical flux equation model in terms of actual factors, which is obtained directly from the Design Expert software, can be written as follows:

$$
\begin{aligned}
J = & -2.58113 + 9.39033 \times \text{DER} + 0.97062 \times \text{AGL} \\
& -1.37785 \times \text{CBT} + 0.02167 \times \text{BFR} + 8.12517 \times \text{PT} \\
& -1.35471 \times \text{DER} \times \text{DER} + 0.056143 \times \text{CBT} \times \text{CBT} \\
& -0.00171743 \times \text{BFR} \times \text{BFR} - 1.31094 \times \text{DER} \times \text{AGL} \quad (2) \\
& +0.055616 \times \text{DER} \times \text{BFR} - 0.42484 \times \text{DER} \times \text{PT} \\
& +0.21523 \times \text{AGL} \times \text{BFR} - 0.00449107 \times \text{CBT} \times \text{BFR} \\
& -0.10005 \times \text{CBT} \times \text{PT} - 0.06317 \times \text{BFR} \times \text{PT}
\end{aligned}
$$

Equation (2) is valid under the following conditions:

$2 \leq \text{DER} \leq 6$
$0 \leq \text{AGL} \leq 2$
$18 \leq \text{CBT} \leq 30$
$0/100 \leq \text{BFR} \leq 70/30$
$2 \leq \text{PT} \leq 6$

MODEL VALIDATION AND CONFIRMATION RUN

In order to verify the adequacy of the model developed, 3 confirmation run experiments were performed as shown in Table-5. The test conditions for all confirmation run experiments were conditions that have not been used previously but were within the range of the levels defined previously. Using the point prediction capability of the software, the flux of the selected experiments was predicted together with the 95% prediction interval. The predicted value and the associated prediction interval were based on the model developed previously. The predicted value and the actual experimental value were compared, and the residual and percentage of error were calculated. All these values are presented in Table-5.

The percentage of error range between the actual and predicted values for flux is as follows: −5.88 to −1.83%. Thus, it can be said that the empirical model developed is reasonably accurate for the flux as all actual values for the confirmation runs are within the 95% prediction interval. The 95% prediction interval is the range in which one can expect any individual value to fall into, 95% of the time.

Table-5. Confirmation runs for flux.

No	DER	AGL	CBT	BFR	PT	Actual	Predicted	Residual	Error
	(cm^3/min)	(cm)	(°C)	(NMP/H_2O, wt. %)	(h)				(%)
1	3	0	18	0/100	5	30.26	32.04	-1.78	-5.88
2	5	1.5	30	0/100	6	27.71	29.32	-1.61	-5.81
3	4	1	21	0/100	3	17.51	17.83	-0.32	-1.83

CONCLUSIONS

In this research, a statistical DOE methodology has provided an efficient method to obtain a regression model for the spinning process. A half fractional factorial experiment was conducted and the experimental outputs demonstrated that dope extrusion rate, air gap length, coagulation bath temperature, bore fluid ratio, and post-treatment time have a significant effect on the flux performance of hollow fiber membranes. In addition, the best-fit regression model has been generated.

This study has proven that the resulting value for the estimation of flux could be acquired with small training and testing samples by using the available experimental data. With a sample size of 30 for experimentation, it was discovered that by applying the statistical regression technique, the resulting value for flux prediction was considerably close to the real flux value. This means that the statistical regression technique is capable of producing precise predictive values for the performance measure via a small number of training samples. It can be proven that the small number of testing samples is not a critical matter in generating an excellent prediction. Therefore, this study has established the practicality of the statistical regression method to develop a flux prediction model. This model was verified by conducting confirmatory tests. The empirical model can be subsequently utilized for predicting the optimum spinning conditions, thus, enabling a better approach to producing high performance membranes for sufficient supply of clean water to fulfill human, environmental and industrial demands.

In summary, this study has applied the statistical regression technique for model development to correctly predict flux values in the spinning process. In addition, it can be determined that the modeling stage of the spinning process is significant for constructing an accurate mathematical model. In the next stage, further investigation could be done to study the settings for optimizating the mathematical model, which is defined as the optimization stage. This stage will be of great significance in producing the optimal results of the predicted values acquired from the modeling stage. Several optimization methods such as genetic algorithm (GA), simulated annealing (SA), tabu search (TS), ant colony optimization (ACO), and particle swarm optimization (PSO) are suggested. All these methods can be categorized as artificial intelligence approaches, which possess the capability for optimizing the spinning parameters. For future work, it is proposed that these methods can be hybridized with the statistical regression technique in order to optimize all the spinning condition values which have influenced the predicted flux.

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