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Modeling and Optimization of Spinning Parameters on Selectivity of Polysulfone Hollow Fiber Membrane for CO₂/CH₄ Separation

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Abstract

Hollow fiber membrane (HFM) and related technologies have recently become highly in demand and widely used in most industries and other areas recently. HFM could be used in different functions, from water and gas treatment to blood and medical applications. The paper describes the modeling of the effects of the spinning condition of a nonporous HFM on the membrane's characteristics with the use of an automated hollow fiber fabrication system. An integrated, systematic experimental strategy based on the design of experiments was used to elaborate the effects of each parameter on selectivity. To obtain and sustain a satisfactory, significant selectivity of HFM, an automated system controlled the parameters during the fabrication process. The parameters involved in the fabrication of HFM and used in modeling are dope flow rate, bore flow rate, draw force, and air gap, which are considered inputs to fabricate polysulfone membrane. The fabrication process is improved by automating and instrumenting fabrication systems. An empirical model of the effects of fabrication parameters on the selectivity of the membrane is extracted experimentally. The mathematical model effectively explains the selectivity of CO₂/CH₄ with 95% fit.

Abstrak

Pemodelan dan Optimasi Parameter-parameter Pemintalan pada Pemilihan Serat Berongga Polysulfone. Membran serat berongga (MSB) dan teknologi-teknologi terkait akhir-akhir ini telah mengalami permintaan yang tinggi dan secara luas dipakai pada sebagian besar industri dan area lainnya. MBS dapat digunakan untuk berbagai fungsi yang berbeda, dari perlakuan air dan gas hingga aplikasi pada darah dan kesehatan. Makalah ini menjelaskan pemodelan pengaruh-pengaruh kondisi pemintalan dari MBS tidak berpori pada karakteristik-karakteristik membran dengan menggunakan sistem fabrikasi serat berongga otomatis. Sebuah strategi eksperimen yang sistematis dan terintegrasi berdasarkan perancangan eksperimen digunakan untuk meneliti pengaruh-pengaruh dari setiap parameter pada selektifitas. Untuk mendapatkan dan mempertahankan selektifitas yang penting dan memuaskan, sebuah sistem otomatis mengendalikan parameter-parameter selama proses fabrikasi. Parameter-parameter yang terlibat dalam fabrikasi MBS dan digunakan pada pemodelan adalah laju aliran *dope*, *bore*, gaya tarik dan celah udara yang dianggap sebagai masukan-masukan untuk membuat membran *polysulfone*. Proses fabrikasi ditingkatkan dengan otomatisasi dan pelengkapan sistem fabrikasi. Sebuah model dari pengaruh-pengaruh parameter-parameter fabrikasi pada selektifitas dari membran diperoleh secara eksperimen. Model matematika secara efektif menjelaskan selektifitas dari CO₂/CH₄ dengan kecocokan 95%.

Keywords: design of experiments, full factorial and selectivity, hollow fiber membrane, polysulfone

1. Introduction

A membrane is a semi-permeable barrier that separates mixtures by controlling the transport rate of particular components of the mixture [1]. In simple terms, selectivity is the ability of the membrane to allow specific gas to pass. Therefore, modeling the effects of spinning parameters of a membrane on selectivity is one of the most important

developments in the membrane separation process [2]. For example, CO₂ passes and CH₄ is blocked in this study. The selectivity value indicates the performance of the produced hollow fiber membrane (HFM) in selecting those gases.

Membranes play an important role in various fields of science and industry, such as gas separation (acid gas

removal from natural gas, oxygen and nitrogen production from air, ethylene separation from emissions in a polyethylene production unit), environment optimization, desalination of seawater, blood purification (hemodialysis), filtration of ground and underground water, filtration of municipal and industrial wastewater, the medicine industry, and biotechnology [3, 4].

In general, producing a high-performance membrane involves trial-and-error methods for evaluating models, which cover optimal material formulation, selection of critical operating variables, and proper conditions during the fiber spinning process [5].

A comprehensive model that describes and predicts the selectivity in different conditions during the production of HFM is necessary. Such a model can effectively assist in achieving higher selectivity in membranes. Many relevant works have been conducted over the past few years. However, evaluating membrane selectivity for specific applications remains a difficult task. Many factors need to be considered during membrane fabrication, and numerous assumptions can be made for this evaluation [6].

The membrane performance evaluation in this study focused on the modeling and optimization of fabrication conditions of the membrane during the process of manufacturing to gain higher selectivity, an optimized industry-grade product, and reproducibility of the same properties in membrane fabrication. Several factors involved in this property, which influence the performance and properties of the membrane such as take-up speed, dope flow rate, bore flow rate (BFR), coagulation bath temperature, room temperature/humidity, and air gap (AG), are spinning conditions that influence the properties of the membrane [7, 8]. In this study, an experiment was conducted to find effective factors to obtain a highly reproducible membrane fiber with

consistent properties for CO₂ and CH₄ separation [9]. The mathematical model was obtained by using the full factorial method to predict the selectivity of manufactured membranes on the basis of fabrication parameters during the fabrication of HFMs.

2. Fabrication System Instrumentation

A highly precise fabrication system was utilized to fulfill requirements that are necessary to achieve the aims of this work. A two-orifice spinneret was used in this research to fabricate HFMs (OD/ID = 0.6/0.3 mm and made of stainless steel imported from Gold Charm Engineering and Services, Singapore). The central unit that connects all the sensors, actuators, and computer unit, and provides an easy human interface with the system through a touchscreen human interface is the programmable logic controller (PLC). Three electric motors were used for the gear pump, AG distance, and spinning drum. A ISCO D-series precision syringe pump was used for either the dope solution or the bore fluid actuators, in which flow and pressure are controlled within a remarkable operation range [10].

In Figure 1, nos. 1 to 21 denote the following: 1: bore fluid; 2: dope solution; 3, 4: pump; 5: chiller; 6, 7, 8, 9, 10: controller; 11: spinneret; 12, 13, 14, 15: motor; 16: PLC system, 17: adjusting AG distance, 18: valve, 19: spooling, 20: tension sensor; and 21: coagulant bath and rollers. The conventional method for the manual hollow fiber fabrication process is shown in Figure 1 [10].

Figure 2 shows a schematic of the novel design of HFM fabrication. Design control using a mechatronics system changes the digital control manually on the current system with the PLC as a novel automation control system. Using limit switch, sensors, actuators, temperature transmitters, and tension control unit as input in the programming reduces the system operation time.

Figure 1. Schematic of the Conventional Spinning Process

Figure 2. Schematic of the Automated HFM Fabrication System

Table 1. Physical and Chemical Properties of Chemicals Used

Chemical	Supplier*	Appearance/color	Melting point (m.p)/boiling point (b.p)(°C)	Density, ρ (g/cm ³)	Molecular weight of repeat unit Mw (g/mol)
PSF Udel p-3500	Local, Malaysia	Transparent	188.3	1.24	~80,000
DMAc	Fluka, UK	Colorless liquid	-20.0	0.94	87.12
THF	Solvay, Belgium	Colorless liquid	-108.4	0.89	72.11
NMP	Fluka, UK	Colorless liquid	-24	1.03	99.13
EtOH	Sigma, US	Colorless liquid	-144.0	0.79	46.07

*local distributor

3. PSF Hollow Fiber Preparation

The dope solution for the formation of a defect-free asymmetric membrane in this study was formulated to comprise polysulfone (PSF Udel-P3500), N-dimethylacetamide (DMAc), tetrahydrofuran (THF), and ethanol with compositions of 30%, 35%, 30%, and 5%, respectively, by weight. In this study, the HFM is produced through a dry-jet wet-spinning process. A mixture of distilled water and N-methyl-2-pyrrolidone (NMP) was utilized as bore fluid. Table 1 summarizes the physical and chemical properties of the chemicals that were used.

Table 2 summarizes the spinning conditions of HFMs and their dope composition. [11, 12].

The spun hollow fiber is fabricated under steady-state conditions based on parameters shown in Table 1, and the sample for making the test module was chosen randomly 15 minutes after every change applied to the fabrication conditions. In other words, a 15-minute delay was set to pick up and cut the random sample between any new treatment combinations in inputs; this issue will be explained in Section 5. The fabrication parameters changed a total of 60 times to produce enough samples and datasets, thus ensuring that the experimentally produced membrane is uniform and reaches the stable condition of fabrication.

Table 2. Spinning Conditions

Parameter	Value for spun fiber
Spinneret dimensions (OD/ID), (mm)	0.60/0.30
Dope flow rate (cm ³ /min)	1 to 2
Bore fluid composition (v/v%)	NMP:H ₂ O (1:10)
Bore fluid rate (ml/min)	0.33 to 0.67
Bore fluid temperature (°C)	27 ± 2
AG distance (cm)	1 to 4
External coagulant	Tap water
Coagulant temperature (°C)	27 ± 2
Force convection gas	N ₂
Take-up speed (m/min)	9.1 to 11.7
Humidity (%) and room temperature (°C)	70 ± 5/27 ± 2

The spun hollow fiber is fabricated under steady-state conditions based on parameters shown in Table 1, and the sample for making the test module was chosen randomly 15 minutes after every change applied to the fabrication conditions. In other words, a 15-minute delay was set to pick up and cut the random sample between any new treatment combinations in inputs; this issue will be explained in Section 5. The fabrication parameters changed a total of 60 times to produce enough samples and datasets, thus ensuring that the experimentally produced membrane is uniform and reaches the stable condition of fabrication.

4. Membrane Module Preparation and Gas Permeation Test and Selectivity Calculation

Before a membrane separation can be performed successfully, the membrane module is required for packaging the hollow fibers. The membrane samples to be tested for the gas permeation experiment should undergo two crucial steps: sample or module preparation, and membrane coating treatment. To prepare the module, dried hollow fibers with a length of approximately 20 cm were potted into a bundle into a stainless-steel holder by using epoxy resin that consists of 5 to 6 fibers in each module [11].

Highly permeable silicone rubber polymer polydimethylsiloxane (PDMS) was used to coat the membrane. The targeted chemical composition for the coating solution is 3 wt.% of PDMS diluted in n-hexane.

The gas permeation experiments were conducted using pure methane and carbon dioxide with purity of more than 99.9%. Each pure gas was tested in the sequence of CH₄, and CO₂. To conduct the experiments under steady-state conditions, the HFMs have to be compacted at a pressure of 5 bar, using CO₂, until a steady-state permeate flux was reached. The flux was measured to monitor the compaction behavior so that the steady-state flux value was attained. The ideal selectivity was simply determined as the pressure-normalized flux ratio of the two-gas component (CO₂ over CH₄). [11, 12]

5. Design of Experiments Phase

A suitable set of experimental tests and parameter values for each test were selected as responses to a systematic experimental strategy based on the classical design of experiments (DOE). This method emphasizes modeling and simultaneous optimization of membrane performance parameters under various controllable and uncontrollable conditions during the fabrication of the HFM.

The classical DOE method in this study is involved in a full factorial design and environmental noise-blocking technique. Three facets of the procedure were examined: factors or inputs to the process, which can be categorized as either controllable or uncontrollable variables; levels or settings of each factor; and response or output of the experiment.

In this phase, empirical models based on the classical DOE methodology using Design-Expert® software were obtained. The DOE provides statistical analysis to determine which fabrication process inputs have a significant effect on the membrane selectivity to find the mathematical model that implies the system.

In this experiment, four parameters are chosen as the input factors: dope extrusion rate (DER), BFR, collection speed (CS), and AG. To perform this experiment, a two-level full factorial design was considered. Hence, four center points were defined to examine the presence of curvature in the experiment's response surface. On the basis of the number of factors and center points, 2⁴+4 treatment combinations were predicted. To obtain a better model from the 20 mentioned treatment combinations, three replicates identified in this article as a block for each treatment were considered. Consequently, a total of 60 treatment combinations were designed. In other words, the 20 treatments were repeated 3 times to reach a sufficient amount of data for modeling.

Design-Expert® (version 10.0.4, Stat-Ease Inc., USA) was used to evaluate and analyze the experimental design. Table 3 shows a summary of the design, factor, and response information.

Table 3. Design Summary

Name	Units	Type	Low (-1)	High (+1)	Mean	Std. Dev.	Analysis	Model
A: DER	ml/min	Factor (Numeric)	1	2	1.5	0.4510	-	-
B: BFR	ml/min	Factor (Numeric)	0.33	0.67	0.5	0.1533	-	-
C: CS	m/min	Factor (Numeric)	9.1	11.7	10.4	1.1726	-	-
D: AG	cm	Factor (Numeric)	1	4	2.5	1.3530	-	-
R: Selectivity	--	Response (Numeric)	8.04 4	33.44 3	18.89 6	6.2181	Polynomial	Quartic

Table 4. ANOVA Table for Selectivity

Source	Sum of Squares	Df	Mean Square	F-value	p-value (Prob > F)	Effect
Model	6.750948	16	0.421934	44.62687	8.2E-21	significant
A-DER	0.737677	1	0.737677	78.02213	4.91E-11	significant
B-BFR	0.028509	1	0.028509	3.015284	0.089993	not significant
C-CS	0.282404	1	0.282404	29.86909	2.48E-06	significant
D-Air Gap	3.148198	1	3.148198	332.9765	2.73E-21	significant
AB	0.100448	1	0.100448	10.6241	0.002248	significant
AC	0.228588	1	0.228588	24.17719	1.46E-05	significant
AD	0.090509	1	0.090509	9.572926	0.003549	significant
BC	0.252373	1	0.252373	26.69283	6.55E-06	significant
BD	0.010631	1	0.010631	1.124431	0.295173	not significant
CD	0.225276	1	0.225276	23.82688	1.64E-05	significant
A ²	0.500812	1	0.500812	52.96952	6.69E-09	significant
ABC	0.372893	1	0.372893	39.43987	1.73E-07	significant
ABD	0.180521	1	0.180521	19.09319	8.29E-05	significant
ACD	0.006518	1	0.006518	0.689386	0.411182	not significant
BCD	0.486484	1	0.486484	51.45409	9.4E-09	significant
ABCD	0.099108	1	0.099108	10.48243	0.002389	significant
Residual	0.387643	41	0.009455			
Lack of Fit	0.293267	32	0.009165	0.873968	0.637778	not significant
Pure Error	0.094376	9	0.010486			
Cor Total	7.147551	59				

6. Results and Discussion

In this case, on the basis of the ANOVA table (Table 4) that was obtained from the design and analysis by using the Design-Expert® software for selectivity, an F-value of 44.626 implies that the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Moreover, a natural log transform in this model needed to be performed.

Values of “Prob > F” less than 0.0500 indicate that the model terms are significant. In this case, A, C, D, AB,

AC, AD, BC, CD, A², ABC, ABD, BCD, and ABCD are significant model terms.

The table 4 shows the model center points defined in the first stages. Consequently, the model curvature is significant due to the existence of A², and the lack of fit F-value of 0.87 implies that the lack of fit is not significant relative to the pure error. Non-significant lack of fit is good, as it indicates the good fit of the model. Therefore, model reduction is possible. For more accurate and simplification, reduction of insignificant factors and terms is performed as the next step.

The ANOVA table (Table 4) is obtained after model reduction. Factor interactions such as BD and ACD were biased due to their low effect and low significance factor. Values greater than 0.1000 indicate that the model terms are not significant. If many insignificant model terms (not counting those required to support hierarchy) exist, then model reduction may improve the model.

In consideration of all the above adjustments, on the basis of the ANOVA table (Table 5) obtained from the design and analysis by using Design-Expert® for this response, the model F-value of 51.0938 implies that the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Similarly, the model for this reduced ANOVA used a natural log transform.

Table 5. Reduced or Modified ANOVA Table for Selectivity

Source	Sum of Squares	Df	Mean Square	F-value	p-value (Prob > F)	Effect
Model	6.733799	14	0.480986	51.0938	3.82E-22	significant
A-DER	0.737677	1	0.737677	78.36143	3.05E-11	significant
B-BFR	0.028509	1	0.028509	3.028396	0.088972	not significant
C-CS	0.282404	1	0.282404	29.99898	2.09E-06	significant
D-Air Gap	3.148198	1	3.148198	334.4245	6.69E-22	significant
AB	0.100448	1	0.100448	10.6703	0.002143	significant
AC	0.228588	1	0.228588	24.28232	1.28E-05	significant
AD	0.090509	1	0.090509	9.614555	0.003402	significant
BC	0.252373	1	0.252373	26.80891	5.64E-06	significant
CD	0.225276	1	0.225276	23.9305	1.44E-05	significant
A^2	0.500812	1	0.500812	53.19987	4.84E-09	significant
ABC	0.372893	1	0.372893	39.61138	1.37E-07	significant
ABD	0.180521	1	0.180521	19.17622	7.5E-05	significant
BCD	0.486484	1	0.486484	51.67785	6.87E-09	significant
ABCD	0.099108	1	0.099108	10.52802	0.002279	significant
Residual	0.404792	43	0.009414			
Lack of Fit	0.310417	34	0.00913	0.870658	0.642296	not significant
Pure Error	0.094376	9	0.010486			
Cor Total	7.147551	59				
Std. Dev						0.0970
Mean						2.8821
R-Squared						0.943295
Adj R-Squared						0.924833
Pred R-squared						0.887956

Table 6. Confirmation Experiments

Run	Factor				Response		Error (%)
					Experimental	Predicted	
	A: DER ml/min	B: BFR ml/min	C: CS m/min	D: AG cm	R: Selectivity	R: Selectivity	(R-R)/R
1	1	0.33	9.1	4	33.45	31.30	6.85
2	1	0.33	11.7	1	15.25	15.00	1.69
3	1.5	0.5	10.4	2.5	22.69	21.53	5.38
4	2	0.67	9.1	1	12.84	14.14	9.20
5	1.16	0.33	9.1	3.9	30.71	33.47	8.24
6	1.40	0.46	9.1	4	29.41	29.68	0.91
7	1.75	0.67	11.7	4	27.40	30.21	9.28
8	1.21	0.43	11.7	4	26.64	28.04	5.01
9	1.25	0.43	10.35	4	28.18	29.11	3.22
10	1.08	0.33	10.48	4	26.74	29.34	8.88
						AVG	5.87

Figure 3. Normal Probability Plots of Residuals for Selectivity

Values of Prob > F less than 0.0500 indicate that the model terms are significant. In this case, A, C, D, AB, AC, AD, BC, CD, A², ABC, ABD, BCD, and ABCD are significant model terms.

The lack of fit F-value of 0.31 implies the lack of fit is not significant relative to the pure error. The Pred R-squared of 0.8879 is in reasonable agreement with the Adj R-squared of 0.9248, i.e., the difference is less than

0.2. The ordinary R² with a value of ~0.95 indicates that about 95% of the total variability was explained by the model.

The normal probability plot of residuals (Figure 3) was selected to check the normality of the residuals. The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line. The plot clearly shows that the residuals pursue a straight line. Some moderate scatter is expected to exist even with normal data. The x-axis indicates the externally studentized residuals, which are the deleted residual divided by its estimated standard deviation. Studentized residuals are more effective than standardized residuals for detecting outlying Y observations.

Figure 4 shows a plot of the residuals versus the ascending predicted response values, providing a visual check of the assumption of constant variance. The plot shows a random scatter that has a consistent top-to-bottom range of residuals across the predictions on the X1 axis. This plot aims to determine the existence of any outliers. Outliers are runs with residuals outside the red lines on the plot. The existence of an outlier indicates that an observation is not fitted well by the model. In this plot, all residuals are between the red lines. This situation can mean that the observation has no problem and that the correct model was used.

Figure 4. Residual Versus Predicted Probability Plots of Residuals for Selectivity

In terms of actual factors, Equation 1 was obtained to make predictions about the response for given levels of each factor based on the real value mentioned in Table 3. This equation shows the impact of each parameter and their interactions on selectivity by considering their coefficients.

$$\begin{aligned} \text{Ln(R1:Selectivity)} = & -5.69797 - 7.994628 * \text{DER} - 8.367067 * \text{BFR} - 0.593342 * \text{CS} + 0.741203 * \text{AG} \\ & - 5.6757 * \text{DER} * \text{BFR} - 0.50499 * \text{DER} * \text{CS} - 0.20757 * \text{DER} * \text{Air Gap} \\ & - 0.75367 * \text{BFR} * \text{CS} - 0.02356 * \text{CS} * \text{Air Gap} - 0.91361 * \text{DER}^2 + 0.52553 * \text{DER} * \text{BFR} \\ & - 0.83265 * \text{DER} * \text{BFR} * \text{Air Gap} - 0.04588 * \text{BFR} * \text{CS} * \text{Air Gap} + 0.108845 * \text{DER} * \text{BFR} * \text{CS} * \text{Air Gap} \end{aligned} \quad (1)$$

At this stage, the correct model and equation for predicting the response as the selectivity of fabricating the PSF HFM (with its specific composition) for the separation of CO_2 and CH_4 has been achieved.

To confirm the adequacy of the obtained models, 10 confirmation runs were performed (Table 6). The first three runs are treatment combinations that were performed previously, while the other runs are verification runs that were not performed previously but are within the range of the examined factors. The responses of these combinations were predicted using the 95% prediction interval, and the trials were run eight times. The experimental and predicted response values are listed in Table 6. These values were compared to calculate and analyze percentage errors. The range of the percentage errors for selectivity is ~0.91% to 9.28%. One can conclude from the confirmation tests that the developed experimental models are reasonably accurate. All the actual values for the confirmation runs are within the 95% prediction interval range. Also, the average error of 5.87% shows that the model closely describes the system to the experimental data.

Further research presents the possibility of using this mathematical model instead of an experimental setup to find the selectivity of other conditions of spinning polysulfone membranes.

7. Conclusions

In this study, a fitted and confirmed model for polysulfone hollow fiber fabrication for separation of CO_2 and CH_4 was successfully developed and optimized by using the DOE methodology. The mathematical model adequately explains the selectivity of CO_2/CH_4 . This model could be a new paradigm to model and optimize the hollow fiber fabrication system. Moreover, this model facilitates early recognition of utilized variables on the performances. By using the same method, we can obtain the mathematical model for other types and membranes in future works. The proposed mathematical model could be used instead of an experimental setup.

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