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Polyester thin film composite nanofiltration membranes via interfacial polymerization: influence of five synthesis parameters on water permeability

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ABSTRACT

Polyester thin film composite nanofiltration membranes were synthesized on the polyethersulfone (PES) support via the interfacial polymerization between triethanolamine (TEOA) and trimesoyl chloride (TMC). Water permeability measurement were conducted on 16 polyester thin film composite membranes to evaluate the influences and interactions of five synthesis parameters: TEOA concentration (X_1), TMC concentration (X_2), reaction time (X_3), pH of aqueous phase solution (X_4), and curing (X_5). These parameters were varied simultaneously between two limit levels using fractional factorial design, allowing investigation of parameters with lesser samples as well as statistical analysis of results. The regression model between the response and the parameters were developed and the fitted model were tested with analysis of variance (ANOVA). The R^2 for the model was 0.94 implying the predicted values were in reasonable agreement with the experimental data, confirming the high predictability of the applied model. The relative size of effects is visually demonstrated in a Pareto chart. It could be concluded that the significant effects were in the order of $X_2 > X_5 > X_2X_5 > X_3 > X_1$. This study leads up to a regression model that will allow the synthesis of polyester thin film composite membranes via interfacial polymerization with desired water permeability within the range studied.

Keywords: Thin film composite membrane; interfacial polymerization; synthesis parameters; fractional factorial design; water permeability.

INTRODUCTION

Transmembrane transport of solutes and efficiency of membrane process are greatly affected by the membrane's surface chemistry and morphology [1]. Therefore, modification of previously formed membranes' surface is a promising approach to grant new properties to the existing membranes by providing surfaces with tailor-made separation properties, energies and chemical functionalities [2]. The emergence of this asymmetric composite membrane also known thin-film composite (TFC) membrane has significantly changed the membrane industry. TFC membranes is synthesized by first fabricating the asymmetric membranes and then coat another ultrathin barrier layer on top of the fabricated membranes. The coating techniques that had been introduced were interfacial polymerization, grafting polymerization, and layer-by-layer deposition [3,4].

Interfacial polymerization techniques gained popularity over the other coating techniques when a variety of TFC membrane was successfully developed by many companies, allowing wide application in the separation processes industry. This technique allows properties of both support and top thin layer to be individually personalized to achieved desired separation and efficiency [4]. Generally, the transport property (flux and rejection) of TFC membrane is mostly determined by the membrane intrinsic properties like surface charge, morphology, hydrophilicity/hydrophobicity, pore size and their geometry and thickness of the thin-film. All these properties are influenced by the membrane preparation conditions like polymerization reaction time, curing temperature, curing time, monomer type and concentration.[5–9]. Works on thin-film polyamide membranes can be easily found but only few works have been carried out on thin-film polyester membranes. Polyester membranes are more tolerant to chorine attack [2] and some had higher permeate flux than polyamide membrane [10]. Polyester membrane developed using monomers with tertiary amino group such as TEOA produces TFC membrane with its surface flexibly changes its hydrophilicity at different feed pH [7,8].

In our previous study, we attempted to produce self-made TFC membrane via interfacial polymerization for separating xylose from glucose, using TEOA and TMC as monomer on polyethersulfone (PES) ultrafiltration membrane. Five synthesis parameters were studied using fractional factorial design to determine their influences toward xylose separation factor. The self-made TFC membrane proved to be able to separate xylose from glucose with the highest xylose separation factor achieved at 1.64 comparable with commercial membranes [11].

This paper aims to add information on the influences of those five synthesis parameters toward water permeability. The five synthesis parameters are TEOA concentration, TMC concentration, reaction time, pH of the aqueous solution, and curing. Selection of low and high level values were based on the values presented by previous studies [7,12,13] and further literature screening on possible parameters that had an effect xylose separation such as curing [14]. These studies [7,12,13] employed $MgSO^4$ and humic acid as solutes to be retained in the upstream side (retentate) of membrane, with both having molecular weight of ~120 g mol^{-1} and ~230 g mol^{-1}, respectively. The highest rejection achieved by these studies were more than 70 % using $MgSO^4$ [7], and more than 80 % using humic acid [12,13].

Xylose is having smaller molecular weight at ~150 g mol^{-1}. Tailoring TFC membrane to have more than 90 % rejection for xylose would not be able to achieve if TFC membranes were synthesized using values presented by previous studies [7,12,13]. Considering rejection trends in these studies [7,12,13], rejection can be further increase by increasing the high value for factors such TEOA concentration and reaction time. Other factors such as TMC concentration and pH of aqueous solution does not exhibit better rejection when their values were manipulated. Therefore, no changes were made to pH of aqueous solution's low value and high value. A tenfold reduction to TMC concentration at low value were made to observe any possible significant effect to separation performances. Reduction of TMC concentration will greatly benefit the overall cost and safety aspect in synthesizing TFC membranes via interfacial polymerization. A categorical approach was first used for curing factor due to lack of evidence and previous studies on how it can improve rejection of organic solutes.

MATERIALS AND METHODS

Materials

The asymmetric commercial PES membrane was purchased from AMFOR Inc. (China) with the commercial name of UF PES50. The membrane has a nominal molecular cut-off of 50 kDa and water flux (at 25 °C) of 260 LMH. The chemicals used in this study were triethanolamine (R & M Marketing, Essex, UK), trimesoyl chloride (Alfa Aesar, UK), sodium hydroxide (Merck, Germany), n-hexane (Merck, Germany), xylose (Sigma Aldrich, USA), glucose (Sigma Aldrich, USA), and acetonenitrile (J.T. Baker, USA). All chemicals were analytical grade with high purity (> 99%) and acetonitrile with High Performance Liquid Chromatography (HPLC) grade.

Design of Experiment

The experiments were designed out using Design Expert version 7.0.0 (Stat-Ease Inc., USA). A 2^{5-1} fractional factorial design (Resolution V) were used to analyse the statistical significance of each synthesis parameter influencing water permeability, and consequently, this design included 16 experimental runs. Water permeability was taken as the response or output variable of the factorial design experiments. Five independent variables considered for the factorial design were the TEOA concentration (X_1), TMC concentration (X_2), reaction time (X_3), pH of the aqueous solution (X_4), and curing (X_5). All the synthesis parameters studied in this were numerical factors except curing which is a categorical factor. Each variable was examined at a high (coded +1) and low (coded −1) level. Table 1 showed the independent variables for screening process using fractional factorial design. The low level for curing was determined to be "No" where the membrane is left dried at room temperature. Meanwhile, curing at the high level "Yes" is where membrane is dried inside an oven (UF 55, Memmert, USA) at 60 °C for 30 minutes.

The statistical analysis of the models was performed in the form of analysis of variance (ANOVA) with 95% confidence level. Coefficient of determination (R^2), F-test and p-value were used to test the statistical significance of the models. Regression analysis was performed and fitted into the empirical factorial model (first-order polynomial model) based on the fractional factorial design for the experimental data as shown in the following equation:

$$y = b_0 + \sum_{i=1}^{5} b_i X_i + \sum_{i=1}^{4} \sum_{j=i+1}^{5} b_{ij} X_i X_j \quad (1)$$

where b_0, b_i, and b_{ij} are the intercept, regression coefficients of the linear, and interaction terms of the model respectively whilst X_i and X_j are the independent variables and y is the dependent variable.

Preparation of TFC membrane

The aqueous solution was prepared by dissolving sodium hydroxide in ultrapure water according to pH of aqueous solution as base medium for TEOA solution. TEOA is then dissolved in the sodium hydroxide solution. The organic solution was composed of TMC in the n-hexane. Firstly, commercial PES was soaked in an aqueous solution a period of 30 minutes. After that, the membrane was then drained and immersed in an

organic solution for a certain period of time. Finally, the TFC membrane was dried in an oven (UF 55, Memmert, USA).

Table 1. Fractional factorial experimental design for preparation of flat-sheet TFC membranes by IP method.

Synthesis parameters			Code	Levels		
				Low (-1)		High (+1)
TEOA concentration (% (w/v))			X_1	4 %		8 %
TMC concentration (% (w/v))			X_2	0.05 %		0.25 %
Reaction time (minute)			X_3	25		45
pH of aqueous solution			X_4	8		12
Curing			X_5	No		Yes
Std Order	X_1	X_2	X_3	X_4	X_5	*Responses*
						P_m ($L.m^{-2}.h^{-1}.bar^{-1}$)
1	- 1	- 1	- 1	- 1	+ 1	6.7
2	+ 1	- 1	- 1	- 1	- 1	33.0
3	- 1	+ 1	- 1	- 1	- 1	2.3
4	+ 1	+ 1	- 1	- 1	+ 1	2.4
5	- 1	- 1	+ 1	- 1	- 1	12.4
6	+ 1	- 1	+ 1	- 1	+ 1	6.3
7	- 1	+ 1	+ 1	- 1	+ 1	0.7
8	+ 1	+ 1	+ 1	- 1	- 1	2.3
9	- 1	- 1	- 1	+ 1	- 1	18.9
10	+ 1	- 1	- 1	+ 1	+ 1	15.7
11	- 1	+ 1	- 1	+ 1	+ 1	0.9
12	+ 1	+ 1	- 1	+ 1	- 1	1.7
13	- 1	- 1	+ 1	+ 1	+ 1	6.5
14	+ 1	- 1	+ 1	+ 1	- 1	18.5
15	- 1	+ 1	+ 1	+ 1	- 1	1.6
16	+ 1	+ 1	+ 1	+ 1	+ 1	1.4

Experimental set-up and permeation tests

Permeation tests have been carried out using the stirred cell system schematized in Figure 1. A Millipore stirred cell (Model 8200, Millipore-Amicon Corporation, USA) having a maximum volume uptake of 200 mL and an effective membrane area of 2.87 x 10^{-3} m^2 was used in all experiments. Prepared TFC membrane and virgin PES membrane was fitted into the membrane holder at the bottom of the stirred cell. Other parts are then assembled together and place on top of a magnetic stirrer (Model MS-20D, Daihan Scientific Co. Ltd., South Korea). The 180 mL of ultrapure water was poured into the stirred cell. Pure water flux experiment was performed at different pressure (2, 3, and 4 bar) by measuring the time taken for 20 mL of ultrapure water collected with constant stirring speed of 300 rpm using nanofiltration set-up mentioned. Water fluxes in this work was calculated using the following equation:

$$J_w = \frac{\Delta V}{A.\,\Delta t} \tag{2}$$

where J_w denoted pure water fluxes, ΔV is the total volume of the permeate collected (0.02 L), Δt is the duration taken to collect 20 mL of permeate in hour, and A is the effective area of the membrane (2.87 x 10^{-3} m^2). A pure water fluxes against applied pressure graph was plotted to find the pure water permeability, P_m. The gradient for fitted linear lines with 0 as intercept was the P_m for the respective membranes.

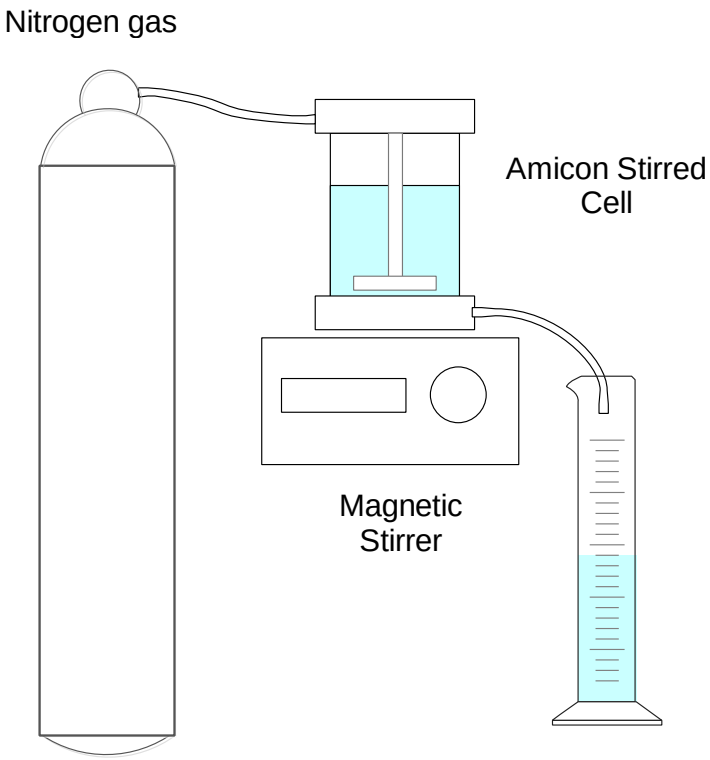

Figure 1. Schematic diagram of nanofiltration system.

RESULTS AND DISCUSSION

Statistical Modeling

The fractional factorial design converted all the data into a first order polynomial equation in terms of the coded synthesis parameters as described by Eq. (3) for water permeability, P_m. Eq. (3) were obtained after performing model reduction by dropping interaction terms one-by-one that have p-value higher 0.100 in ANOVA analysis. The positive signs in the equations show synergic effects whereas the negative signs indicate antagonistic effects. From Eq. (3), each coefficients for linear (b_1, b_2, b_3, b_4, b_5) and interaction terms (b_1b_2, b_2b_3, b_2b_5) are lower than the intercept term (b_0), which indicated the existent of the design plateau. Thus, these plateau showed that the design had an optimum point, where further optimization experiment can be performed [15].

$$P_m = +8.19 + 1.96X_1 - 6.55X_2 - 1.99X_3 - 0.057X_4 - 3.14X_5 - 1.67X_1X_2 + 1.83X_2X_3 + 2.82X_2X_5 \quad (3)$$

Table 2 summarizes the model's sum of squares from the ANOVA results for the response (P_m). The statistical significance of the factorial model has been estimated in terms of *F*-value and *p*-value. Generally, the more *F*-value deviates from unity, the

more certain is that the synthesis parameters adequately explain the variation of the data. For the *p*-value, the lower is this value, the more trustable is the model. The model had *F*-value 15 implying the model is significant. The *p*-value for model was 0.001, which is less than 0.05 implying that the model fit the experimental data well.

In addition, the coefficients of multiple determinations R^2, adjusted R^2, and predicted R^2 have been calculated. The goodness-of-fit is validated when the coefficient R^2 has the tendency to be more close to unity and when predicted R^2 is in agreement with the adjusted R^2. The ANOVA results given in Table 2 indicate that the factorial model have R^2 higher than 0.9, and the predicted R^2 of 0.71 was in reasonable agreement with the adjusted R^2 of 0.87. A graphical examination on the predicted versus actual plot shown in Figure 2 reveal that the actual values are distributed relatively near to the predicted straight line. This signifies that for the ranges of parameters studied, the models gave potent estimates of the response. Thus, the model can be considered reliable and reproducible.

Table 2. ANOVA results for water permeability.

Source	df	Sum of Squares	Mean Squares	*F*-Value	*p*-value	
Model	8	1195	149	15	0.001	significant
X_1-Conc. TEOA	1	62	62	6	0.043	
X_2-Conc. TMC	1	687	687	68	< 0.0001	
X_3-Reaction time (TMC)	1	63	63	6	0.041	
X_4-pH aqueous solution	1	5.12×10^{-2}	5.18×10^{-2}	5.10×10^{-3}	0.945	
X_5-Curing	1	158	158	16	0.006	
X_1X_2	1	45	45	4	0.074	
X_2X_3	1	53	53	5	0.056	
X_2X_5	1	128	128	13	0.009	
Residual	7	71	10			
Cor Total	15	1266				
R^2=0.94; Adjusted R^2=0.87; Predicted R^2=0.71						

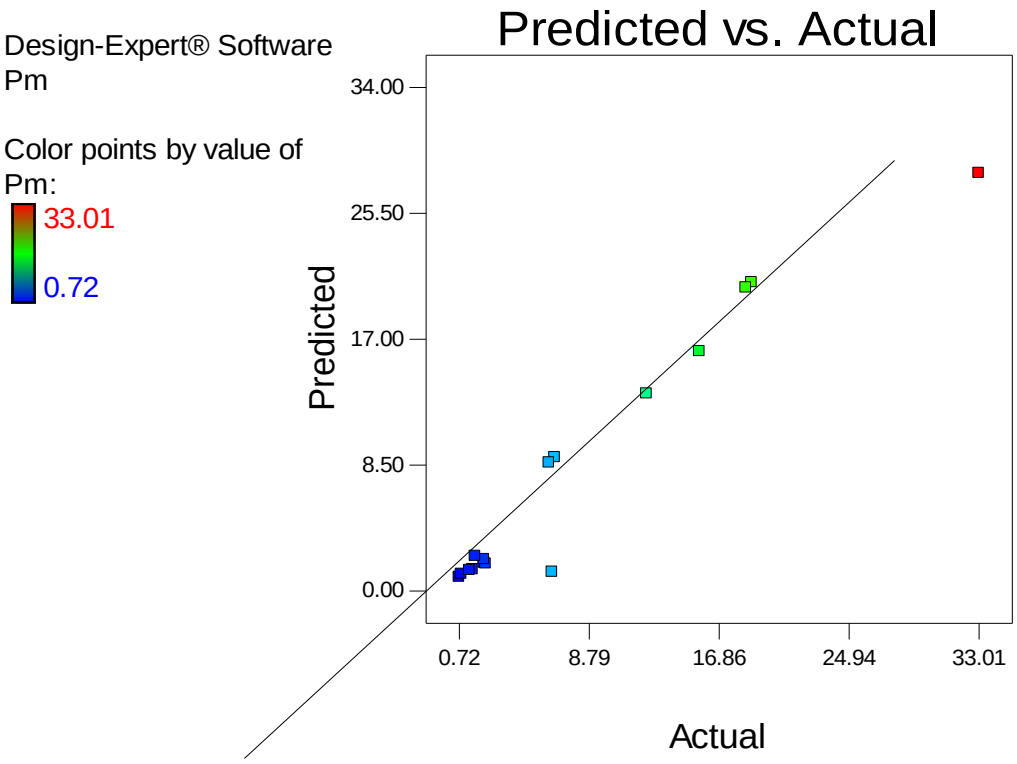

Figure 2. Predicted vs actual data for water permeability.

The relative size of effects are visually demonstrated as Pareto chart in Figure 3. Bar lengths in Figure 3 are proportional to the absolute value of the estimated effects, which helps to compare relative importance of the effects.The value of the Student's *t*-test parameter for p = 0.05 (95 % confidence level) and seven degrees of freedom (df) was 2.3645. Thus, a *t*-value for the model coefficient which surpasses the critical value of 2.3645 is considered to be statistically significant over the range of analytical response at the 95% confidence level. The following viewpoints can be seen in Figure 3.

(1) Four of the independent variables, namely TEOA concentration (X_1), TMC concentration (X_2), reaction time (X_3), and curing (X_5) were statisitically significant. Hence, these factors had major influence on the response within the limits of studied levels except pH of the aqueous solution (X_4).

(2) The interaction effect between TMC concentration and curing (X_2X_5) was the only two-way interaction effect that is statistically significant. The insignificance of effects does not mean that these factors are unimportant, but just implies a little influence on response.

(3) The significant effects could be ranked based on *t*-value. It could be concluded that the significant effects were in the order of $X_2 > X_5 > X_2X_5 > X_3 > X_1$. From Eq. (3), The effect of X_1 and X_2X_5 have positive sign in their regression coeffieicents signify theses factors have positive effect on the response, while the rest has negative effect on the response.

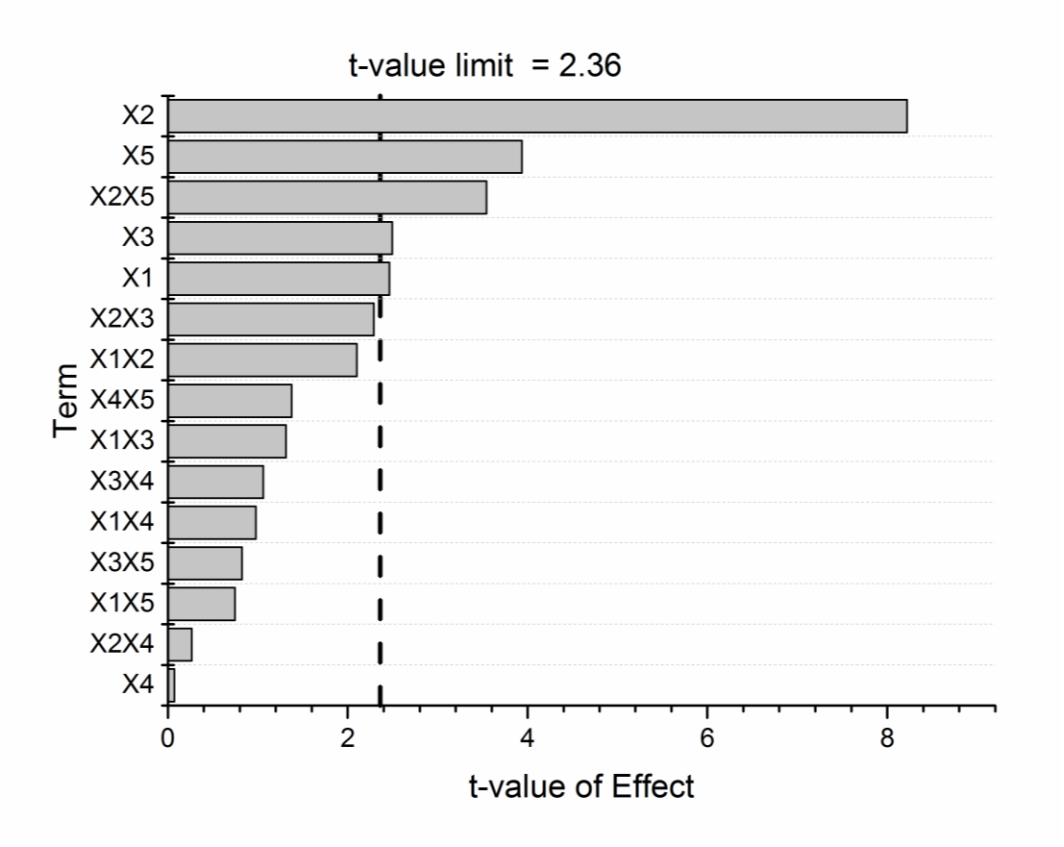

Figure 3. Pareto chart obtained for the fractional factorial design.

Effects of Synthesis Parameters on Water Permeability

Water permeability was commonly used to characterized membranes [16], where water permeability of commercial nanofiltration membrane ranges from 1.3 to 50.5 $L.m^{-2}.h^{-1}.bar^{-1}$ [17]. Also, water permeability of self-made nanofiltration membranes ranges from 1 to 20 $L.m^{-2}.h^{-1}.bar^{-1}$ in a review by Lau et al. (2015) on more than 80 research articles published in the peer-reviewed journals. From Table 1, TFC membranes developed in this study could be classifying as nanofiltration membranes since their water permeabilities have close resemblance to both commercial and self-made membranes. Water permeability could be used as a relative measure of membrane's pore size and thickness. Low water permeability could be associated with membranes smaller pore size and thicker in overall membrane thickness, while high water permeability associate to membranes with larger pore size and thinner in overall membrane thickness. This can be explained by the transport mechanisms of solute passing through the membrane, in this case water. In general, the transport of ionic solute (water) through nanofiltration is a complex process since nanofiltration membrane exhibit properties between reverse osmosis membranes and ultrafiltration membrane. Commonly, the size exclusion, solution diffusion mechanism, and charge effects were considered in modelling the tranport phenomena in nanofiltration [18].

The most common transport in membrane proceses is size exclusion, where solute smaller than membrane pore pass through the membrane and solute larger than membrane pore were retained. Transport of solute through size exclusion were based on the size of solute and pore size of membrane. Membranes with smaller pores only allow a small amount of solute to pass through at one time reducing the fluxes which bring to lower water permeabilities. Clearly, membranes with larger pore will have higher fluxes

and high water permeabilities. Solution diffusion mechanism is another common tranpsort in non-porous membrane processes where solute and solvent dissolve onto the active layer of membrane and diffuse through the layers. Membranes which are thicker in general would have lower fluxes and water permeabilities due to the need of solute to travel longer distance through the membrane. Another transport in nanofiltration is charge effects which relies on ionic charge of solute and membrane, attracting each other if they have same charges, repelling each other if they have different charges through electrostatic repulsion phenomenon. The transport of solute through charge effects relies heavily on the interaction between solute and active layer of membrane, where it boils down to the charateristics of membrane surface, especially membrane surface charge and hydrophilicity. Since there is no variation in type of monomers used for interfacial polymerization in this study, thus the charateristics of membrane surface among the prepared membranes may not have significant differences. Thus, it is safe to assume that the transport through charge effects do not have significant effect on water permeability in this study.

Fractional factorial design with resolution V do not have confounding effects between main effects, between two-way interactions, or between main effects and two-way interactions. The influences of five synthesis parameters studied in this work on water permeability were illustrated in the main effect plots shown in Figure 4. A main effect plot represent the average of all the responses produced by changing the level of a factor. This plot could be used to determine factors that significantly influence the response and to compare the relative strength of the effects. The more steeper a slope is, that factor has more influences toward the response studied. The steepest slope was shown by factor TMC concentration in Figure 4b, where increasing TMC concentration decreases the water permeability of a membrane. Relatively speaking, the decreases of water permeability could be interpreted as either decrease in membrane pore size, or increase in overall membrane thickness, or as a result of both. In any three situations, any changes to membrane's pore size and thickness mentioned is the result from more cross-linking occurrence between both aqueous and organic monomers during formation of thin-film via interfacial polymerization. Apart from TMC concentration, curing process is another noteworthy factor that have huge influence on water permeability. Curing is a process where heat is introduced after interfacial polymerization reaction to remove residual solvent. It is a widely held view that curing process promotes additional crosslinking between aqueous and organic monomers [14], densify the thin-layer composite by making the polymer chain packed more closely to each other [19], and improve membrane flux and separation performance [20]. Water permeability decreases with the introduction of curing ("Yes" in Figure 4e) signifying changes to membrane's pore size and thickness has resemblances to mentioned factor TMC concentration as predicted by the above hypothesis. Figure 4a and 4c present the effect of TEOA concentration and reaction time on water permeabilites of the TFC membranes. It can be observed that changing the TEOA concetration from 4 % (w/v) to 8 % (w/v), and reaction time from 25 minutes to 45 minutes did no result in a appreaciable change in water permeability. This signifies both factors do play a role in formation of thin-film composite however the outcomes were not distint as factor TMC concentration and curing. In Figure 4d, the slope was nearly parallel to the x-axis implies pH of aqueous solution has very little or no influence on water permeability.

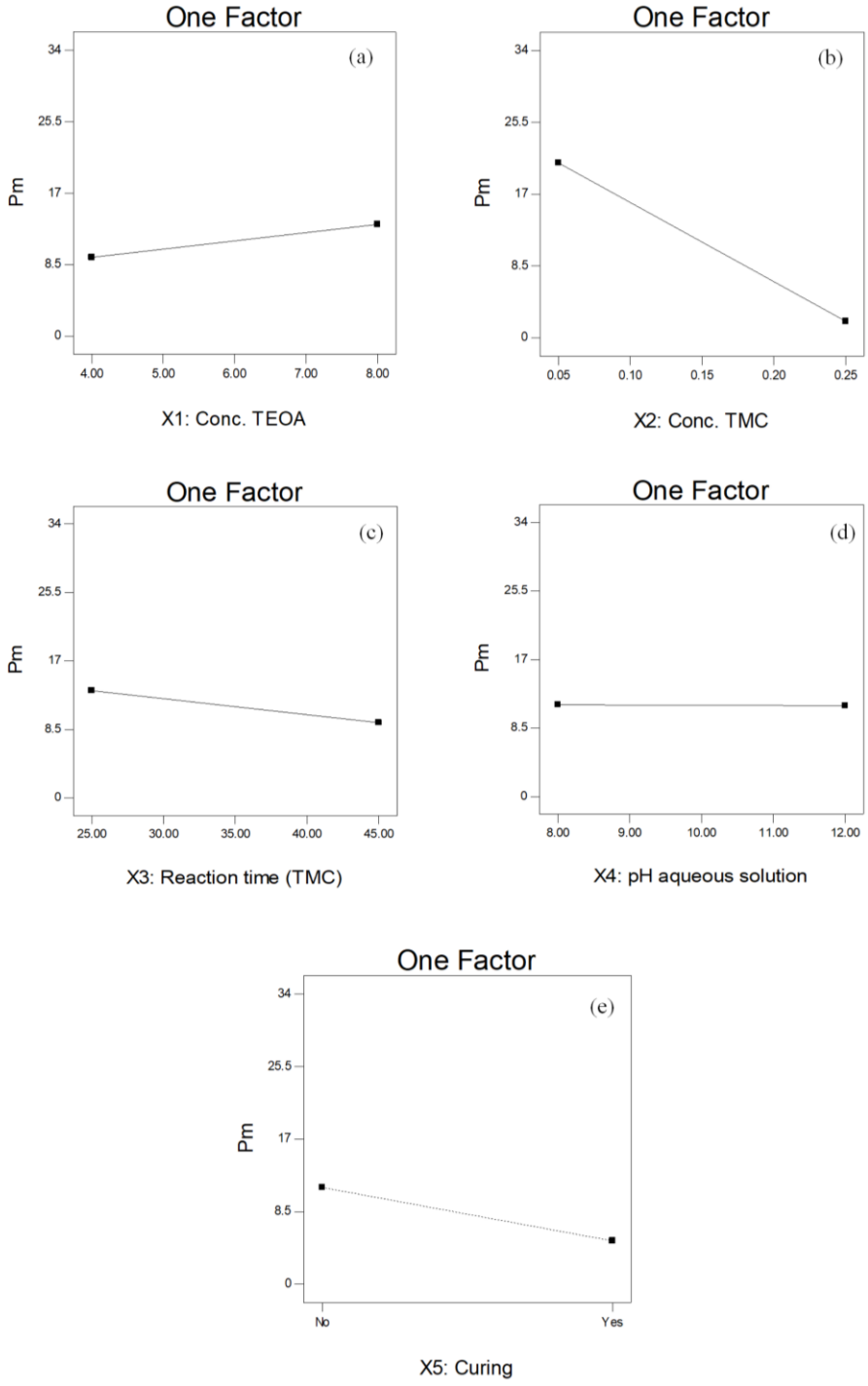

Figure 4. Main effect plots obtained for the fractional factorial design. [(a) X_1, (b) X_2, (c) X_3, (d) X_4, and (e) X_5]

CONCLUSION

Our previous study evaluated the five synthesis parameters of interfacial polymerization and identified the key factors affecting xylose separation. The five synthesis parameters are TEOA concentration, TMC concentration, reaction time, pH of the aqueous solution, and curing. This paper attempt to add information on the influences of those

five synthesis parameters toward water permeability using Fractional factorial design with resolution V. Mathematical model was developed utilizing experimental data, and fitness of the model was verified by employing ANOVA. The findings were presented suggest that four of the synthesis parameters, namely TEOA concentration (X_1), TMC concentration (X_2), reaction time (X_3), and curing (X_5) were statistically significant affecting water permeability. The magnitude of each effects on water permeability were in the order of $X_2 > X_5 > X_2X_5 > X_3 > X_1$, from high to low. Only the effect of X_1 and X_2X_5 have positive effect on water permeability, while the rest has negative effect on water permeability. Decrease in water permeability can be associated to the decrease in pore size and increase in overall membrane thickness, where more occurrence of crosslinking between monomers in formation of thin-film.

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