Characterization Assessment on Nanofiltration Membrane using Steric-Hindrance Pore (SHP) and Teorell-Meyer-Sievers (TMS) Models

(Penilaian Pencirian pada Membran Penapisan Nano menggunakan Model *Steric-Hindrance Pore* (SHP) dan *Teorell-Meyer-Sievers* (TMS))

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ABSTRACT

Interfacial polymerization (IP) is a simple process for modifying thin-film composite (TFC) polymers that can be used as separation membranes in water treatment. This work describes the IP process for the preparation of polyester TFC membranes using organic monomers, in particular triethanolamine (TEOA) and trimesoyl chloride (TMC). This work includes an evaluation of monomer concentration and polymerization reaction time as variables to determine the membrane properties and its performance as acid humic removal. The characterization of TFC membranes was investigated using field emission scanning electron microscopy (FESEM), steric hindrance pore (SHP) and Teorell-Meyer-Sievers model (TMS). This IP technique resulted in the membrane (NF-PES8-35) having the lowest contact angle (θ=34.0±0.35) and lower hydrophobicity (θ=62.6 ± 0.33) compared to the unmodified membrane. The rejection of NaCl by NF-PES8-35 membrane showed the highest 0.001 M NaCl (62.42%), while NF-PES4-15 membrane showed the lowest (2.4%). The highest removal of humic acid (97.8%) was achieved when separation was performed with the NF-PES6-35 membrane and the high performance polyester TFC membranes were exhibited in the water purification filtration system.

Keywords: Characterization; nanofiltration membrane; steric-hindrance pore; Teorell–Meyer–Sievers model

ABSTRAK

Pempolimeran antara muka (IP) ialah proses mudah untuk mengubah suai polimer komposit saput nipis (TFC) yang boleh digunakan sebagai membran pemisahan dalam rawatan air. Kertas ini menerangkan proses IP bagi penyediaan membran poliester TFC dengan menggunakan monomer organik, khususnya triethanolamine (TEOA) dan trimesoyl chloride (TMC). Kertas ini meliputi penilaian terhadap kepekatan monomer dan masa tindak balas pempolimeran sebagai pemboleh ubah bagi menentukan sifat membran dan prestasinya sebagai penyingkir asid humik. Pencirian membran TFC dikaji menggunakan mikroskopi pancaran medan elektron penskanan (FESEM), medan pelepasan mikroskop elektron pengimbas (FESEM), Model *steric-hindrance pore* (SHP) dan Model *Teorell-Meyer–Sievers* (TMS). Teknik IP ini menghasilkan membran (NF-PES8-35) yang mempunyai sudut sentuh terendah (θ=34.0±0.35) dan kehidrofobian yang lebih rendah (θ=62.6 ± 0.33) berbanding membran yang tidak diubah suai. Penolakan NaCl oleh membran NF-PES8-35 menunjukkan yang tertinggi 0.001 M NaCl (62.42%), manakala membran NF-PES4-15 menunjukkan yang terendah (2.4%). Penyingkiran tertinggi asid humik (97.8%) diperoleh apabila pemisahan dilakukan menggunakan membran NF-PES6-35 dan menunjukkan membran TFC poliester berprestasi tinggi dalam sistem penapisan pembersihan air.

Kata kunci: Membran nanofiltrasi; model *steric-hindrance pore*; model *Teorell–Meyer–Sievers*; pencirian

Introduction

Nanofiltration (NF) membrane separation includes reverse osmosis (RO) and ultrafiltration (UF) are widely used in industry to treat wastewater before discharge. NF has advantages such as low operating pressure, higher permeation flux, high retention of polyvalent salt compounds and low capital, operating and maintenance costs (Viet et al. 2021). Most commercial NF and RO membranes prepared by interfacial polymerisation (IP) have a thin polyamide layer (PA) over the membrane support, using m-phenylenediamine (mPD) and reacting with acyl chloride to form PA (n-hexane). Sometimes, the bisphenol-A (BPA) monomer has been used to prepare the thin film polyester (PE) and polyesteramide (PEA) membranes via the IP method (Abu Seman, Khayet & Hilal 2010; Jayarani et al. 2000; Mohammad, Hilal & Abu Seman 2003). Despite the excellent performance of BPA in producing membranes with high retention capacity and minimal organic fouling, its toxicity has proven to be a serious problem due to various adverse health effects at high temperatures (Geens, Goeyens & Covaci 2011; Kubwabo et al. 2009; Michałowicz et al. 2014; Sun et al. 2021).

In this study, triethanolamine (TEOA) is used instead of carcinogenic BPA to produce the polyester thin film nanofiltration membrane (PE-TFNF) for humic acid separation. TEOA is an organic molecule containing a tertiary amine and a triol. These two components together form a strong base and form hydrogen bonds. By changing the pH of the feed solution, the tertiary amino group in the molecule can be converted to the quaternary ammonium group, improving the performance of the membrane. TEOA was used as the primary monomer to create an active layer of polyester membranes TFC NF. PE active layer membranes using (TEOA) are utilised in desalination at varying pH values (Arribas et al. 2020). PE membrane has a negative charge at high pH than its isoelectric point of the membrane surface (between pH 4 and pH 8). Therefore, it can distinguish between polyvalent and monovalent anions (Wu et al. 2020). As the pH of the feed solution decreased, the flow rate of NaCl solution increased, showing that the PE-TFNF membrane could function well. Trimesoyl chloride (TMC) is the most commonly used monomer for the preparation of TFNF membranes because it contains acyl chloride groups, which affect the cross-linked network structure of PE, and -COOH groups, which affect the hydrophilicity and salt rejection properties of the membranes through hydrolysis of the unreacted acyl chloride groups in TMC (Farahbakhsh et al. 2021).

As a result, the developed membranes exhibited high permeate flux and salt rejection and were suitable for seawater desalination (Sharabati et al. 2022).

The main objective in developing an antifouling membrane is to adjust its surface properties, including roughness, hydrophobicity, surface charge and toxicity to foulants, by adding nanoparticles (NP) or organic molecules (Saffarimiandoab at al. 2021). Different monomers produce the different characteristic properties of the membrane. In addition, the membrane characteristics are influenced by the reaction time, the pH of the aqueous solution, the concentration of the reactants and the curing temperature (Ji et al. 2021). Higher monomer concentrations, faster reaction rates and longer polymerisation times lead to thicker layers with higher rejection and poorer membrane flow performance.

This study investigates the effects of TEOA monomer ratio and IP duration on TFC membrane properties, including structural, electrical and performance. The IP process can be used to produce a high molecular weight polymer at ambient temperature, with little need for stoichiometric equilibrium of reactants and low purity requirements (Feng, Khulbe & Matsuura 2010). In this way, an ultrathin active polyester layer can be prepared without balancing the stoichiometric ratio with the unstable polymers at high temperatures. The theoretical model was used to determine the pore radius (rp) and the ratio of membrane thickness to porosity ($\Delta x/Ak$) using the Steric-Hindrance Pore (SHP) and Teorell-Meyer-Sievers (TMS) models. The TMS model was applied to determine the electrostatic effects, such as Xd and the ratio of the effective charge density to the concentration of the main solution. These modelling results also showed the relationship between the modified variables and the membrane properties. This study aims to fabricate nanofiltration (NF), thin film composite (TFC) membranes using different TEOA concentrations and reaction times of the IP process. Physical and chemical characterisation studies evaluated the NF TFC membrane and the SHP and TMS modelling study.

Materials and Methods

MATERIALS AND CHEMICALS

The commercial flat Ultrafiltration Polyethersulfone membrane, UF-PES50, with a molecular weight cut-off (MWCO, 50 kDa) and water flux (260 L/m2.h) was purchased from AMFOR INC (China) as a support for this study. Triethanolamine (TEOA) from R&M Marketing

(Essex, UK) and trimesoyl chloride (TMC) from Alfa Aesar (UK) were used as monomers in the aqueous and organic phases, respectively. Sodium hydroxide (NaOH), vitamin B12, sodium chloride (NaCl), and n-hexane were supplied by Merck (Germany). R&M Marketing (Essex, UK) supplied glycerol and sodium disulphide to preserve the membranes.

INSTRUMENTATION FOR MEMBRANE CHARACTERIZATION

Thermo Scientific™ Nicolet™ iS™50 FT-IR spectrometer characterised the connections of the functional groups in the membrane and the modified membrane in absorbance regions between 4000 and 400 cm^{-1}. FESEM (MODEL JOEL) was used to visualise the surface and cross-section of the membrane at high magnification. According to the researchers, the FESEM technique has been widely used to classify new membranes with pore sizes ranging from UF to MF. In addition, image analysis can provide information about the average pore distribution on the surface, which is often not often provided by membrane manufacturers. A goniometer (Rame-Hart, model 290) was used to measure the contact angle measurements via a static sessile drop system. For sodium chloride (NaCl), a conductivity meter (Eutech Instruments, CON 2700) was used to determine the salt concentration.

MEMBRANE PREPARATION VIA INTERFACIAL POLYMERIZATION

Interfacial polymerization (IP) technique was implemented to cast the polyester NF membrane. First, the small pieces of UF-PES50 membrane were immersed in an aqueous 4% TEOA solution for 30 min and allowed to drain. Then the membrane was placed in a TMC solution for the IP process for 15 min and left. Then, the membrane was post treated to form a thin layer by placing it in an oven at 60 °C for 30 min. Finally, the prepared membrane was immersed in deionized water overnight to remove excess chemical solutions. These procedures were repeated for 6% and 8% monomer concentration and the IP process varied to 25 min and 35 min, respectively (Jalanni, Abu Seman & Mohammad Faizal 2015, 2013). As a result, the PS membranes were prepared as shown in Figure 1. The membranes prepared were named NF-PE4-15, NF-PE4-25, NF-PE4-35, NF-PE6-15, NF-PE6-25, NF-PE6-35, NF-PE8-15, NF-PE8-25 and NF-PE8-35 depending on the TEOA concentration (4%, 6% and 8%) and IP reaction time (15 min, 25 min and 35 min).

DETERMINATION OF NATURAL SOLUTE REJECTION STUDY

Before each use, all membranes were soaked in distilled water for at least 10 min, and then placed in the filtration cell (Amicon, model 8200) with 400 kPa of pressure. For the retention of uncharged solutes, 100 mg/L Vitamin B12 was used in this experiment and tested at different operating pressures (100, 200, 300, 400 kPa). The time (Δt) to reach a constant permeate volume of 20 mL was recorded and fluxed for each operating pressure.

$$J = \frac{V}{A\Delta t} \tag{1}$$

where A is the effective surface area of the membrane (28.7 cm^2), and V is the volume of permeation collected (20 mL). The absorption value of the feed solution in the Amicon stirred cell body before and after the test, including the permeate, was analyzed with a UV spectrophotometer. Equation (2) was used to measure the average bulk concentration of the feed solution.

$$C_b = \frac{C_i + C_f}{2} \tag{2}$$

C_b is the average bulk concentration; and C_i and C_f are concentrations before and after the test. To reduce the concentration polarization effect, the feed solution was stirred at 300 rpm. Equation (3) represents the calculation for the effect of concentration polarization, the real rejection, R_{real} (Lau & Ismail 2009).

$$R_{real} = \left(1 - \frac{C_p}{C_w}\right) \times 100\% \tag{3}$$

where C_p and C_w are the concentration of the permeate and the concentration of boundary layer (wall). As previously discussed, the volume of flux for this Vitamin B12 solution was measured following Equation (1).

DETERMINATION OF IONIC COMPONENT RETENTION

All experimental procedures followed the procedure for neutral solute rejection. For the ionic solutions, salt solutions (NaCl) of 0.001M - 0.1M were prepared. As mentioned earlier, the flux volume for all salt solutions was measured according to Equation (1). Equation (3) was also applied for salt retention, as concentration polarization was considered in this experiment.

THE STERIC-HINDRANCE PORE (SHP) AND THE TEORELL– MEYER–SIEVERS (TMS) MODELS ANALYSIS

Physical and chemical characterization studies evaluated the NF-PE membrane, as well as the SHP and TMS modelling study. Structural factors include pore radius (r_p), ratio of porosity to the thickness of the membrane (A_k/Δ_x), and electrical properties, including the ratio of the effective volume charge density of the membrane to the electrolyte concentration of the feed solution (ξ) and effective volume charge density (X). These are crucial elements in developing models for membrane rejection and flux predictions. The alternative method for determining the pore size at the nanoscale, the Spiegler-Kedem equation (SK), is shown in Equation (4):

$$R=\frac{\sigma\left(1-exp\left(-\frac{1-\sigma}{P}Jv\right)\right)}{1-\sigma \exp\left(-\frac{1-\sigma}{P}Jv\right)} \qquad (4)$$

According to Equation (4), an increase in retention R leads to an increase in solvent flux. Once the volume flux, J_v, becomes countless as the filtration flow reaches the solution diffusion, the retention, R, is comparable to the reflection coefficient, σ at the maximum solute rejection, and the experimental data of R as a function $1/J_v$ can be calculated directly. This model is only applicable to a solution containing a single neutral solute such as sodium chloride (NaCl). The membrane parameters can be measured using Equations (5) and (6).

$$\sigma_s=1\text{-}H_F\,S_F \qquad (5)$$

$$P_s=H_DS_DD_S\left(\frac{A_k}{\Delta x}\right) \qquad (6)$$

S_F and S_D refer to the distribution coefficient of solutes under convection and diffusion conditions. HF and HD are devoted to steric parameters correlated with the wall correction factors in the convection and diffusion coefficients. The membrane is assumed to have porous support with straight, uniformly distributed cylindrical pores through which the uncharged solute spheres are transported (Imbrogno & Schäfer 2019).

The study of the rejection of uncharged solutes cannot provide meaningful information on quantitative data on the negative surface charge of NF membranes. Therefore, TMS is used to investigate the electrical properties of the membrane (Sun & Song 2021). The σ_{salt} and P_{salt} parameters of a membrane can be determined using a mono-mono electrolyte (NaCl) as an aqueous solution by combining the extended Nernst-Planck model and the Donnan equilibrium theory (Wang & Lin 2021). This model assumes that the volumetric flux penetrating the membrane is insignificant and that the ion concentration profiles within the pore are equal (Kristensen et al. 2017).

$$\sigma_{salt}=1-\frac{2}{(2\alpha-1)\xi+ \sqrt{(\xi^2+4)}} \qquad (7)$$

$$P_{salt}=D_s\ (1\text{-}\sigma_s)(\frac{Ak}{\Delta x}) \qquad (8)$$

where C is electrolyte concentration; and X is effective volume charge density.

$$\alpha=\frac{D\ cation}{D\ cation+D\ anion} \qquad (9)$$

$$\xi=\frac{X}{C} \qquad (10)$$

RESULTS AND DISCUSSION

CHARACTERIZATION OF THE NF MEMBRANE STRUCTURE AND SURFACE CHEMISTRY

The interfacial polymerization process formed the active polyester layer from the reaction between TEOA monomers and TMC, involving the carboxyl and the hydroxyl of the functional group (an organic acid and an alcohol monomer) (Figure 1). This reversible esterification process produces an ester compound. The acyl chloride and the alcohol react readily at room temperature, especially if both are aliphatic.

Figure 2(a) and 2(b) shows the differences between the tops of the unmodified membrane (UF-PES50) and the modified membrane (NF-PE6-35). The modified membrane has a rougher top surface, indicating a new layer successfully formed on the surface of the NF-PE6-35 membrane. The micrographs in Figure 1(c)-1(f) shows that the porosity of the NF-PE6-35 membrane also increased from top to bottom, which can be seen from the formation of the active layer and support layer of the membrane.

A rougher membrane surface represents a higher value of effective surface area, membrane hydrophilicity,
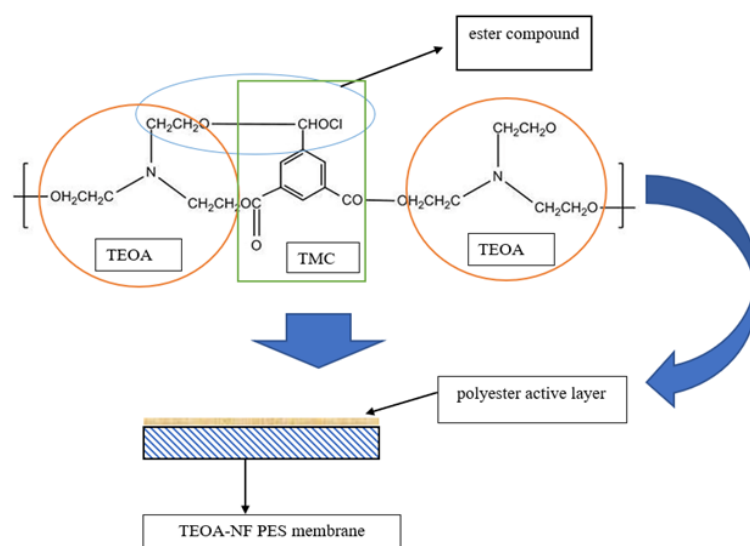
FIGURE 1. The polyester active layer formation from the IP process
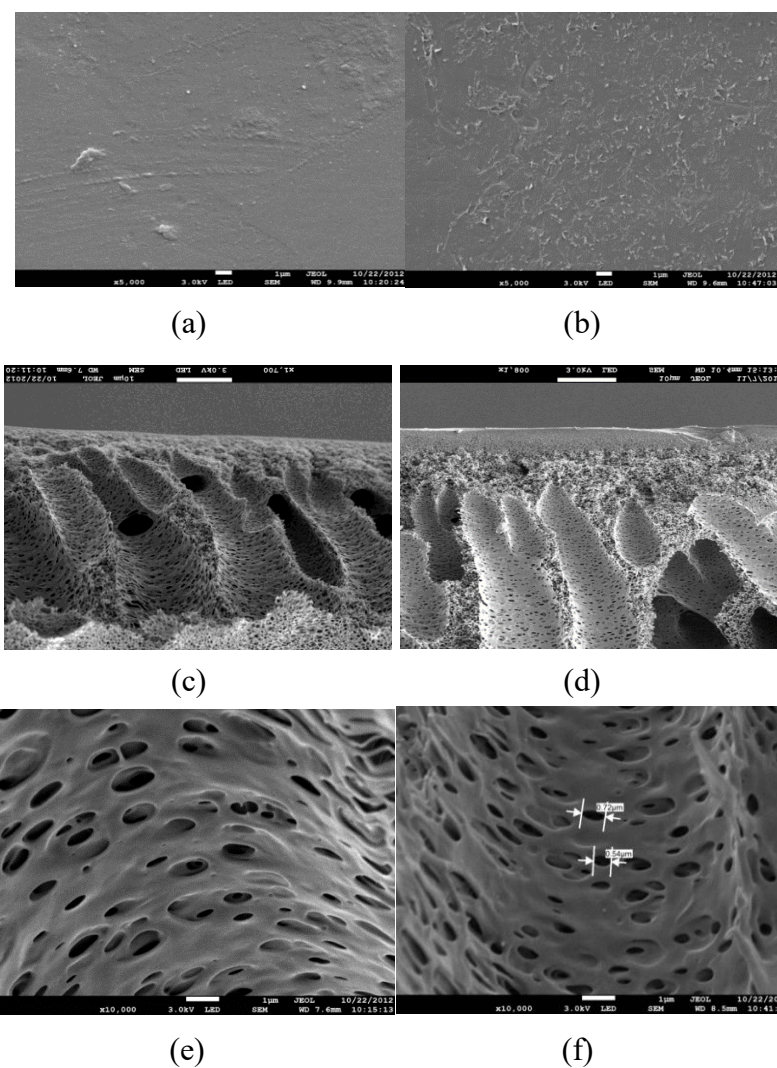

(a)
(b)
(c)
(d)
(e)
(f)

FIGURE 2. FESEM images at a different magnification of (a) UF-PES50 membrane (b) NF-PE6-35 membrane and cross-section images of (c) UF-PES50 membrane (1700x) and (d)NF-PE6-35 membrane 1800x; morphology of (e) UF-PES50 membrane 10,000x and (f) NF-PE6-35 membrane 10,000x

and permeate flow (Wang et al. 2007). The polyester TFC membranes formed a smaller radius and were more hydrophilic, as shown in Figures 2(f) and 3, respectively. This high hydrophilicity was attributed to the amino groups attached to the modified PE membrane surfaces, which were converted to R_3HN^+. The contact angle values decreased (the hydrophilicity increased), except

for the NF-PE6-35 membrane. At a constant time, the reaction time of the IP process is generally directly proportional to the thickness of the membrane top surface and the hydrophobicity. Membranes modified with the highest monomer concentration (8%) and the longest reaction time (35 min) exhibited the most hydrophilic properties and decreased contact angle values by over 50%.
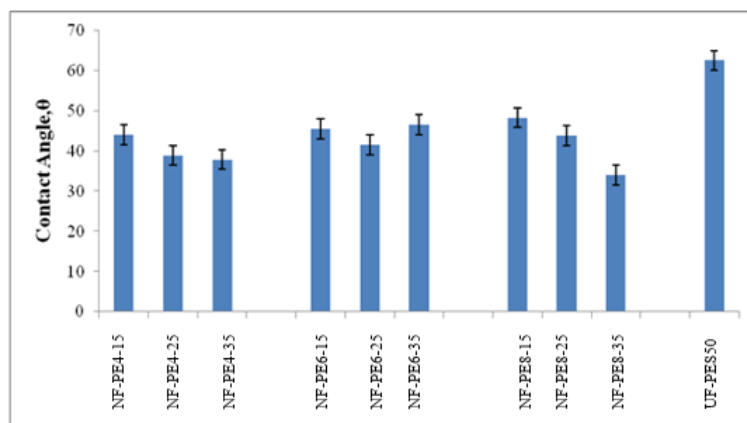

FIGURE 3. The contact angle values for modified and unmodified membranes

CHARACTERIZATION OF PORE SIZE AND Δx/Ak OF POLYESTER TFC MEMBRANES USING UNCHARGED SOLUTES

The graph of SHP modelling for the fabricated membranes is shown in Figure 4. The details of the structural properties of the membranes derived from the SHP modelling analysis are summarized and tabulated in Table 1. Most of the NF-PES membranes produced have a smaller pore radius than unmodified membranes. In addition, due to increasing transmembrane pressure, solute rejection increased with increasing monomer concentration and reaction time (Wang et al. 2007).

Previous studies have found that increasing the water flow with pressure (permeate solution) leads to a decrease in solute concentration, increasing the solute rejection (Wang & Chung 2006; Wang et al. 2007). The concentration of the monomer TEOA was directly proportional to Δ_x/A_k at constant reaction time, as shown in Table 1. The PES8-35 membrane has the highest value of Δ_x/A_k (3.87×10^{-5}) compared to Δ_x/A_k (2.45×10^{-5}) PES4-35 membrane. However, the pore radius (r_p) is

inversely proportional to the monomer concentration at a constant IP time, which is r_p (1.01) PES8-35 compared to r_p (1.07) PES4-35. Membranes prepared at different IP times at a constant monomer concentration showed different trends in Δx/Ak values, especially at 6% and 4%. However, the data of the r_p values show a significant influence of the IP time. The r_p values show an inversely proportional trend with increasing IP time at constant monomer concentration for higher monomer concentrations (8% and 6%). On the other hand, the r_p values of the lowest monomer concentration (4%) show a directly proportional trend to IP time.

These measurements of pore radius are consistent with values obtained for commercial nanofiltration (NF) membranes, as the calculated r_p values are 1.01-1.34 nm (Bowen, Mohammad & Hilal 1997). Minor changes in pore size dispersion and effective number of pores on the NF membrane during filteration were attributed to variations in pore size with monomer concentration and reaction time (Hilal et al. 2004). As monomer concentration and reaction time increased, a denser active
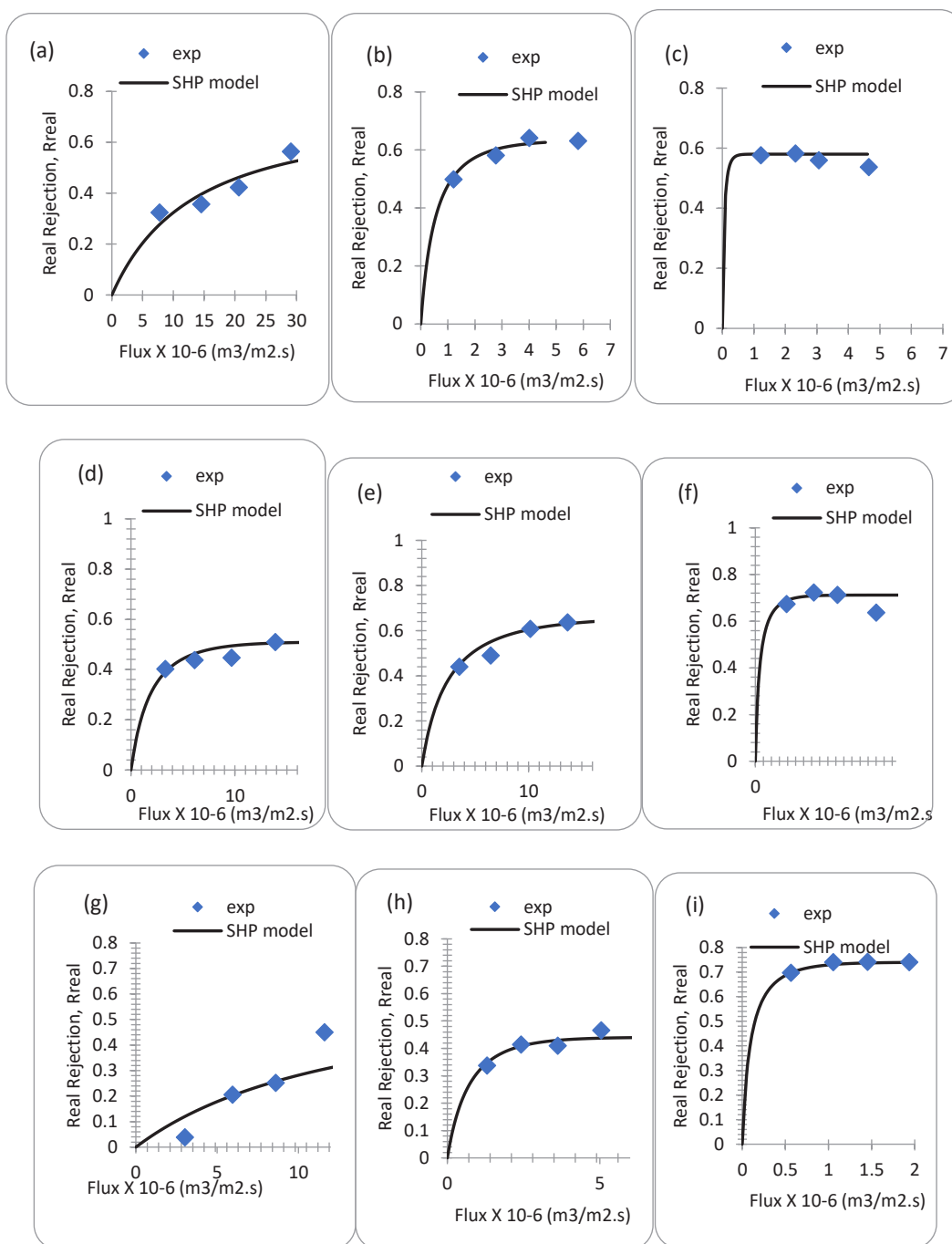
FIGURE 4. The Vitamin B12 rejection of PES membrane plotted by SHP model. (a)
NF-PES4-15, (b) NF-PES4-25, (c) NF-PES4-35, (d) NF-PES6-15, (e) NF-PES6-25, (f)
NF-PES6-35, (g) NF-PES8-15, (h) NF-PES8-25 and (i) NF-PES8-35

TABLE 1. The membrane parameters were obtained from the Spiegler-Kedem Equation and SHP model

Membrane	σ (-)	P ($\times 10^{-7} ms^{-1}$)	λ (-)	$\Delta x/A_k$ (m)	r_p (nm)
NF-PES8-35	0.74	0.89	0.73	3.87×10^{-5}	1.01
NF-PES8-25	0.44	6.48	0.55	2.01×10^{-5}	1.34
NF-PES8-15	0.53	110	0.61	7.4×10^{-6}	1.22
NF-PES6-35	0.71	1.00	0.72	3.10×10^{-5}	1.03
NF-PES6-25	0.66	22	0.69	2.26×10^{-5}	1.07
NF-PES6-15	0.51	17	0.60	1.67×10^{-5}	1.23
NF-PES4-35	0.58	0.5	0.64	2.45×10^{-5}	1.16
NF-PES4-25	0.63	4.62	0.67	1.86×10^{-5}	1.10
NF-PES4-15	0.66	120	0.69	4.21×10^{-6}	1.07

layer developed on the UF PES membrane. These results showed a substantial Δ_x/A_k and r_p value, suggesting that both monomer concentration and polymerization time have an effect on membrane performance (Ding, Zhu & Liu 2021).

DETERMINE THE EFFECTIVE VOLUME CHARGE DENSITY X_d AND σ

The TMS model is one of the most effective methods quantitively measure electrical properties. The TMS and irreversible models can accurately predict the rejection of a single salt solution throughout the NF separation process (Ahmed et al. 2019). By fitting the actual NaCl rejection data to the TMS model (Figure 5), the electrical properties (X_d and ξ) of the membranes can also be calculated using Equation (7). Equation (4) was used to determine the salt permeability (P_{salt}) and reflection coefficient (σ); and the results are shown in Table 2. The NF-PES8-25 membrane exhibited the highest Xd value at 0.001 M NaCl, reflecting steric effects affecting rejection only (Bouchoux, Roux-de & Lutin 2005).

The results showed that the rejection of NaCl was proportional to the concentration of NaCl. The rejection of salt depended on steric factors and electrostatic interactions between the membrane and solutes. Diffusion affected the transport mechanism at low NaCl concentrations (Donnan 1995). The NF-PES6-35 membrane performed well at high salt concentrations. A possible explanation for this observation was that the NF-PES6-35 membrane had a higher effective charge density and repelled charged solutes more effectively. The PES membrane became negatively charged due to the formation of ester compounds during the IP process. The performance of the prepared membranes was studied for the separation of humic acid at pH7 (Jalanni, Abu Seman & Mohammad Faizal 2015, 2013; Seman et al. 2013). At pH7, the degree of dissociation of the carboxyl group of humic acid increases. The membrane surface absorbs OH- and converts to more negative electric charges. These situations increased the electrostatic repulsion between the membrane surface and the organic molecules and decreased the HA absorption and precipitation (Jalanni, Abu Seman & Mohammad Faizal 2013). Therefore, the fouling of the membrane in this condition is low state. The results indicate that the membranes formed during the longest polymerization method (35 min) perform very well, with a removal rate of over 95% (Jalanni, Abu Seman & Mohammad Faizal 2015, 2013; Seman et al. 2013). The experimental parameters influenced the character of the membrane produced and have great commercial potential.
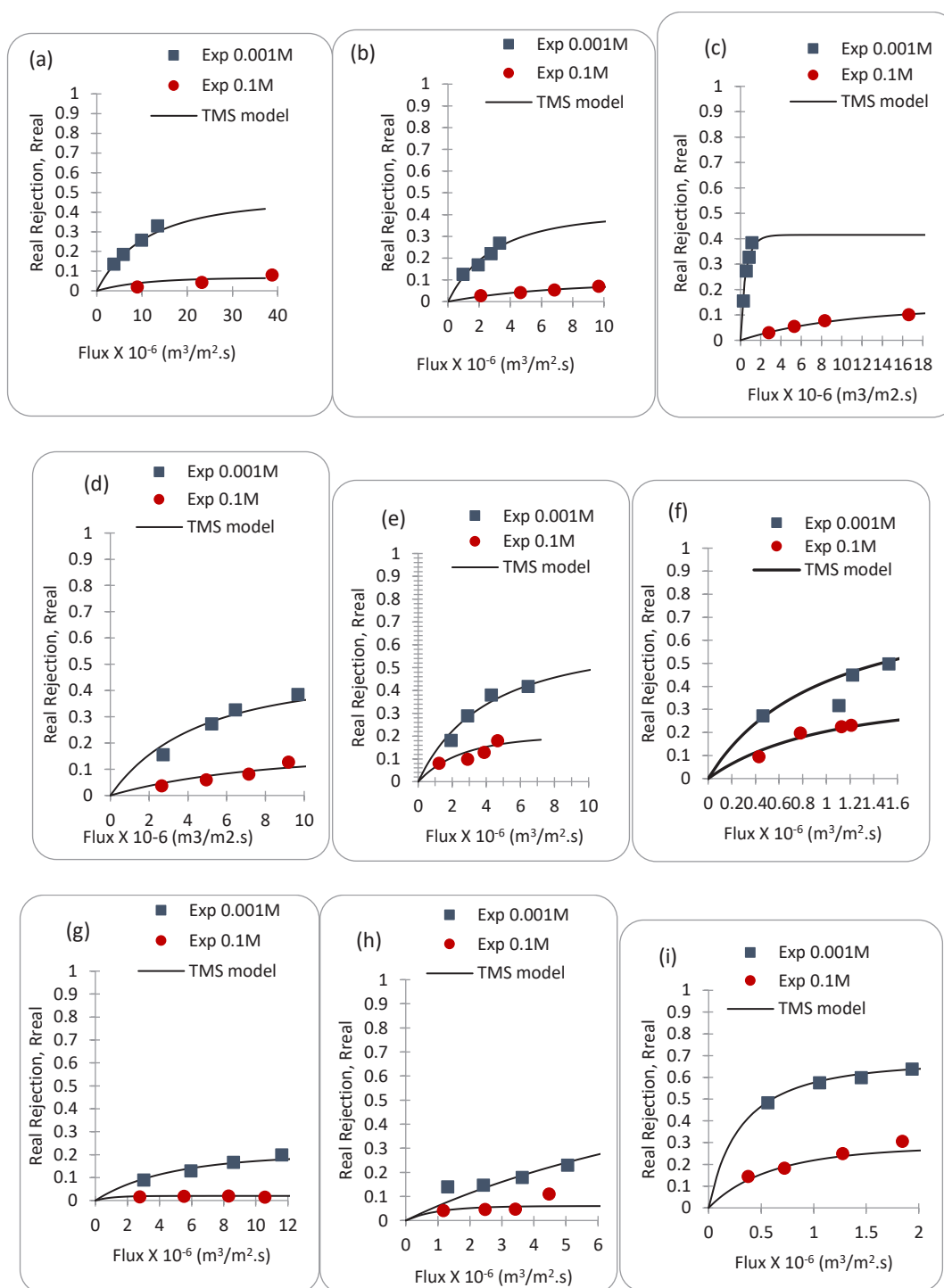
FIGURE 5. The salt rejection caused by the salt concentration of membrane polyester NF with different conditions of experiment (%= percentage weight/volume; min= reaction of time) by the TMS model. (a) NF-PES4-15, (b) NF-PES4-25, (c) NF-PES4-35, (d) NF-PES6-15, (e) NF-PES6-25, (f) NF-PES6-35, (g) NF-PES8-15, (h) NF-PES8-25 and (i) NF-PES8-35

TABLE 2. The ratio of effective charge density to bulk concentration for NF-PES membranes produced

Membranes	The ratio of effective charge density to bulk concentration, X_d	
	0. 001M	0. 1M
NF-PES4-15	3. 996	124
NF-PES4-25	3. 500	150
NF-PES4-35	3. 675	176
NF-PES6-15	3. 000	188
NF-PES6-25	5. 680	217
NF-PES6-35	9. 820	297
NF-PES8-15	3. 680	105
NF-PES8-25	25. 160	133
NF-PES8-35	7. 104	266

CONCLUSIONS

The structural parameters of Steric-Hindrance Pore (SHP) and Teorell–Meyer–Sievers (TMS) models were addressed. Analysis using this model is highly recommended for the evaluation of nano-sized membranes. Monomer concentration and polymerization time affect the membrane properties (pore size, thickness, effective charge density) and are essential in producing membrane. The developed membrane meets the requirement of RO and can be used for the treatment of various impurities in wastewater. The RO membrane is an environmentally friendly system for the treatment of industrial wastewater, as it uses a minimum of chemicals.

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