

Synthesis and characterization of polyamide membrane for the separation of acetic acid from water using RO process

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Abstract. The main challenge in many applications of acetic acid is acid dehydration and its recovery from wastewater streams. Therefore, the performance of polyamide thin film composite is evaluated to separate acetic acid from water. To reach this goal, the formation of polyamide layer on polysulfone support membrane was investigated via interfacial polymerization (IP) of meta-phenylenediamine (MPD) in water with trimesoyl chloride (TMC) in hexane. Also, the effect of synthesis conditions, such as concentration of monomers and curing temperature on separation of acetic acid from water were investigated by reverse osmosis process. Moreover, the separation mechanism was discussed. The solute permeation was carried out under applied pressure of 5 bar at 25°C. Surface properties of TFC membrane were characterized by ATR-FTIR, SEM and AFM. The performance test indicated that 3.5 wt% of MPD, 0.35 wt% of TMC and curing temperature of 75°C are the optimum conditions. Moreover, the permeate flux was $4.3 \frac{\text{L}}{\text{m}^2 \text{ h}}$ and acetic acid rejection was about 43% at these conditions.

Keywords: acetic acid rejection; interfacial polymerization; thin film composite; reverse osmosis

1. Introduction

Acetic acid is an important chemical with wide applications in chemical industry. It's most common uses are as a solvent for production of terephthalic acid, synthesis of vinyl acetate, acetic anhydride and as a food additive (Hilioglu *et al.* 2001). Most of the chemical processes which use acetic acid as a solvent or intermediate are accompanied by waste and/or recycling streams containing acetic acid/water mixtures (Huang *et al.* 2001). Furthermore, water is produced as a by-product in the synthesis of acetic acid (Yoneda *et al.* 2001). In order to obtain pure acetic acid, its dehydration is required and plays an important role in the chemical industry.

Several processes have been proposed for the separation of acid acetic from aqueous solutions,

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such as liquid-liquid extraction (Kersten *et al.* 2014), distillation, azeotropic distillation (Chien *et al.* 2005), reactive extraction (Ki and Li 2005) and extractive distillation (Lei *et al.* 2004). Moreover, membrane processes including pervaporation, electrodialysis, reverse osmosis and nanofiltration are used for the separation of acetic acid from water (Kersten *et al.* 2014). These processes are more energy-saving and cheaper than distillation processes. Also, membrane processes have advantages over the liquid-liquid extraction, because back mixing is prevented and microbes are not directly exposed to the extraction liquid (Joglekar *et al.* 2006). Therefore, scientists are trying to use membrane processes for the separation of acetic acid/water mixture. Generally, pervaporation process is used at high concentrations of acetic acid while at low concentrations, reverse osmosis is used for the separation of acetic acid from water.

Koyama *et al.* (1982) investigated the performance of reverse osmosis (RO) separation for monocarboxylic acids by an anionic charged composite membrane. The composite membrane was composed of polypropylene as the support layer and copolymer of poly(vinyl alcohol)-poly(styrene sulfonic acid) with sulfonic acid groups as the top selective layer. They have concluded that the rejection of acetic acid strongly depends upon the dissociation constant and its degree of dissociation. Also, Huang *et al.* (1998) used cross-linked polyacrylic acid (PAA) composite membranes for separation of aliphatic acids by RO process. In their work, PAA acts as a selective layer and polysulfone as a porous support layer. They reported that the rejection of the membrane for acetic acid was relatively low (27.8%). Therefore, Huang *et al.* (1998) concluded that the cross-linked PAA composite membrane is not applicable for the separation of aliphatic acid aqueous solutions by RO.

Ozaki and Li (2002) evaluated the performance of ultra-low pressure reverse osmosis (ULPRO) membranes for the rejection of organic compounds. They tested ES20 membrane which consists of aromatic polyamides with charged chemical groups (carboxyl and amine groups) on a polysulfone support layer. Finally, they concluded that the degree of dissociation have a significant effect on the rejection of acetic acid.

RO separation of formic (C1), acetic (C2), propionic (C3), and n-valeric (C5) acids from aqueous solutions has been studied by Ragaini *et al.* (2004). They used polyamide thin film composite membranes. Their results showed that rejection increases as operating pressure increases while it decreases with an increase in temperature of the feed.

Laufenberg *et al.* (1996) investigated the effect of intermolecular interactions on the separation of acetic acid from water by RO process. They reported rejection of acetic acid from aqueous solution when acetic acid combined with one of 27 further organic acids. They concluded that formic and propionic acids strongly influence the acetic acid rejection.

Choi *et al.* (2008) evaluated the performance of nanofiltration membranes in the rejection of organic acids (acetic acid, formic acid, citric acid and ...) from wastewater. They used ES10 and NF270 membranes and investigated the effect of operating parameters such as pH, feed concentration and operating pressure.

As noted previously, few researchers investigated the separation of acetic acid from water by RO process and the results of their work indicate that more study is required in this field. In this study, polyamide thin film composite membranes were prepared to improve the performance of RO process in separation of acetic acid from water. The effect of curing temperature as well as two different monomers were also studied on the quality of acetic acid/water separation.

2. Experimental

Table 1 Chemicals used in this study

Material Specifications	Source company	CAS number
M-phenylenediamine (MPD, 99+%)	Sigma-Aldrich	108-45-2
Trimesoyl chloride (TMC, 98+%)	4422-95-1	Sigma-Aldrich
N-hexane (99%)	180-54-3	Dr mojallali
Acetic acid (99.8%)	64-19-7	Dr mojallali
N-dimethylformamide (DMF)	68-12-2	Industrial grade
Polysulfone	25135-51-7	Industrial grade
Sodium hydroxide (99+%)	1310-73-2	Merck
Phenolphthalein	77-09-8	Merck
Hydrochloric acid (37%)	7647-01-0	Merck

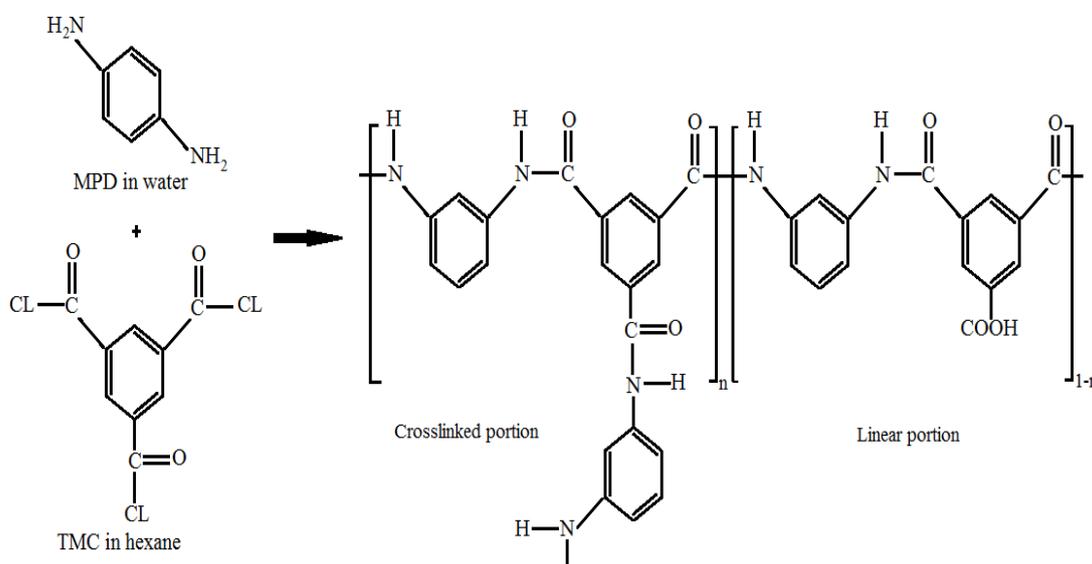


Fig. 1 Polyamide membrane synthesized from m-phenylenediamine and trimesoylchloride by interfacial polymerization

2.1 Material

All of the chemicals are listed in Table 1. Also, deionized water (conductivity $\leq 1 \mu\text{s cm}^{-1}$) was used as a solvent. All of the materials were used without any further purification.

2.2 Membrane synthesis

2.2.1 Preparation of ultrafiltration support membrane

Polysulfone (PSf) ultrafiltration membranes were synthesized via phase inversion method. This process was done according to the method described by Loeb and Sourirajan in 1960 (Lee *et al.* 2011). Homogeneous solutions of 17 wt% PSf (dried for 2 h at 60°C) in DMF were casted on a non-woven polyester with a thickness of 200 μm . Finally, PSf membranes were moved to the de-

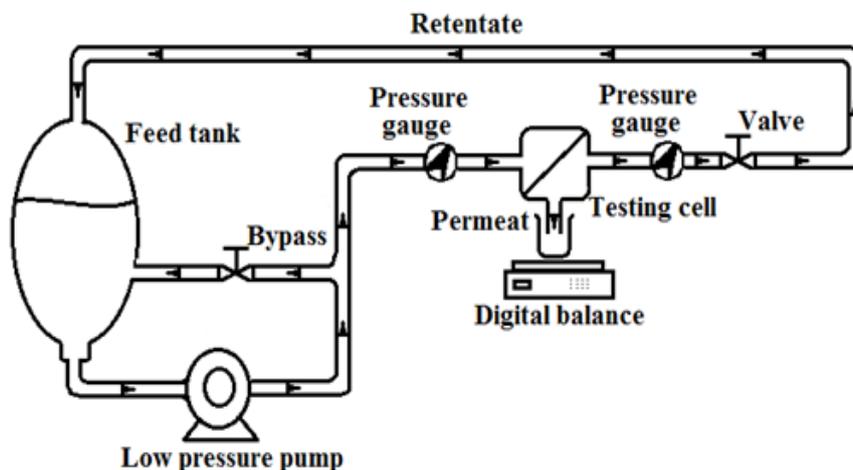


Fig. 2 The experimental RO membrane set up

Table 2 Performance test conditions

Parameter	quantity
Concentration of acetic acid in feed	$0.05 \frac{\text{mol}}{\text{L}}$
Operating pressure	5 bar
Temperature	25 °C
Retentate flow	$1.2 \frac{\text{L}}{\text{min}}$
Effective area of membrane	0.003629 m^2

ionized water bath for 24 h until most of the solvent and water-soluble polymers were removed.

2.2.2 Fabrication of thin film composite membrane

A thin polyamide (PA) layer was synthesized on top of the PSf ultrafiltration support via interfacial polymerization (Fig. 1). PA layer is formed according to the process described by Cadotte (1981). In this work, meta-phenylenediamine and trimesoylchloride were used. This process is as follows:

- 1) Immersing of polysulfone membrane in MPD aqueous solution (90 s)
- 2) Removing the excess MPD solution from the support membrane surface using rubbery wiper
- 3) Immersing of polysulfone membrane in a solution of TMC in hexane (30 seconds)
- 4) Soaking of polysulfone membrane in a curing bath at the specified temperature (65 °C) for a specified time (5 min)
- 5) Washing the membrane with distilled water

2.3 Membrane performance evaluation

The performance of TFC membranes is investigated to separate acetic acid from water by a cross-flow cell (Fig. 2).

In order to enhance mixing, the feed flow is divided into two streams after the pump. Permeate and retentate flow returned to the feed tank after sampling until the feed concentration does not

change. Circular membrane placed in stainless flat module and operating pressure is set by needle valves. Also, plastic and stainless materials were used in this set up to prevent the corrosion effects. Test conditions are given in Table 2.

Flux of permeate and rejection of acetic acid are two important parameters for evaluation of the membrane performance. The rejection is calculated by Eq. (1)

$$\text{Rejection} = \left(1 - \frac{C_p}{C_f}\right) \times 100 \quad (1)$$

where C_p and C_f are the concentrations of permeate and feed solution, respectively. The concentration was determined by titration. The permeate flux is considered to be equal with water flow rate and is calculated by Eq. (2)

$$\text{Permeate} = \frac{\Delta V}{A \cdot \Delta t} \quad (2)$$

where ΔV is volume of permeate, A is the membrane surface and Δt is time.

2.3.1 Titration process

Titration process is one of the methods which widely used to determine the unknown concentration of different solutions. In this method, a standard solution is added till the moles of standard and unknown solutions are equivalent (equivalence point). In this work, sodium hydroxide is used as standard solution. Since NaOH is a humidity absorber, its solution should be standard. The known concentration of HCl solution is used to standardize the base solution. Certain amount of sodium hydroxide is weighted and then a volumetric flask is used to have a specified volume of NaOH solution. Acid solution is poured to a burette. Certain volume of base solution is poured to Erlenmeyer flask and two drops of Phenolphthalein are added. Acid is added to base solution very slowly, until the colour of base solution was changed. Then, the final volume of acid solution is read. To make sure about the test results, this process was repeated three times. Finally, the concentration of base solution is calculated according to the Eq. (3)

$$C_1 V_1 = C_2 V_2 \quad (3)$$

Where C_1 is the concentration of base solution, V_1 is the volume of base solution, C_2 is the concentration of acid solution and V_2 is the volume of base solution. After determining the exact value of base concentration, it was used as the standard solution. Certain volume of permeate solution of RO process was poured to Erlenmeyer flask and two drops of indicator were added. For determining the concentration of acetic acid in permeate, the abovementioned titration process is repeated. Finally, the concentration of acetic acid is calculated according to the Eq. (3).

2.4 Membrane characterization

2.4.1 Attenuated total reflection-fourier transform infrared spectroscopy (ATR-FTIR)

Surface chemical characterization of the TFC membranes was analysed by ATR-FTIR (Perkin Elmer Spectrum Rx1, USA). ATR-FTIR analyse is carried out in transmittance mode in the wave number range of 4000-400 cm^{-1} .

2.4.2 Scanning electron microscopy (SEM) analysis

Cross-section and surface morphology of TFC membranes are visualized by SEM (Tescan Vega, Czech Republic) with a magnification of 20000. All of the samples, were dried at room

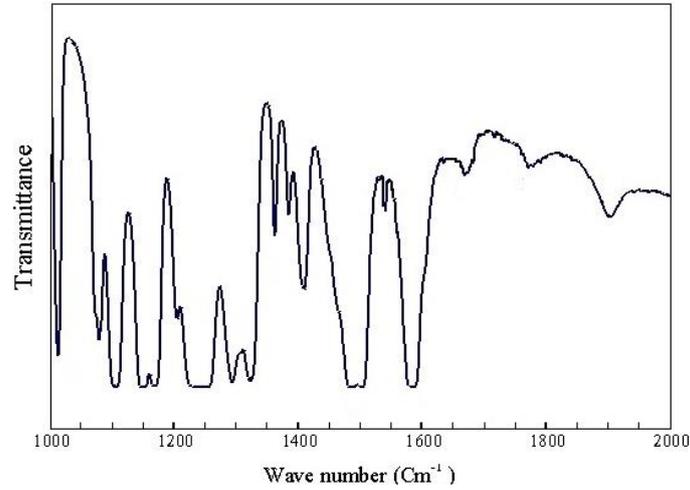


Fig. 3 ATR-IR spectrum of TFC

temperature and then coated with a thin layer of gold with a thickness of 40°A.

2.4.3 Atomic force microscopy (AFM) analysis

Quantitative surface roughness of TFC membranes was characterized by AFM (FemtoScan SPM 2012, Russia). The surface roughness can be reported in term of various roughness parameters. In this study it is expressed in terms of the mean (R_a) and root mean square roughness (RMS). R_a is expressed as

$$R_a = \frac{1}{L_x L_y} \int_0^{L_x} \int_0^{L_y} |f(x, y)| dx dy \quad (4)$$

where $f(x, y)$ is the surface relative to the centre plane and L_x and L_y are dimensions of the surface. Also, RMS is expressed as

$$\text{RMS} = \sqrt{\frac{\sum (z_i - z_{avg})^2}{N_p}} \quad (5)$$

where Z_i is the current Z value, Z_{avg} and N_p are the average of the Z values and the number of points within a given area (Singh *et al.* 1998).

3. Result and discussion

3.1 ATR-FTIR spectra of TFC

In order to investigate the surface chemical structure, ATR-FTIR of TFC membranes was performed and is given in Fig. 2. This figure indicates that the polyamide layer is formed since the acid band at 1770 cm^{-1} ($-\text{COOH}$, pendant carboxylic acid group), a band at 1660 cm^{-1} (amide I, C=O band) and a band at 1540 cm^{-1} (N-H band, amide II) are observed. In addition, other bands of PA layer are also seen at 1595 and 1500 cm^{-1} which are characteristics of aromatic nature (benzene ring, C=C stretching). Also, 1010 cm^{-1} band is present and represents

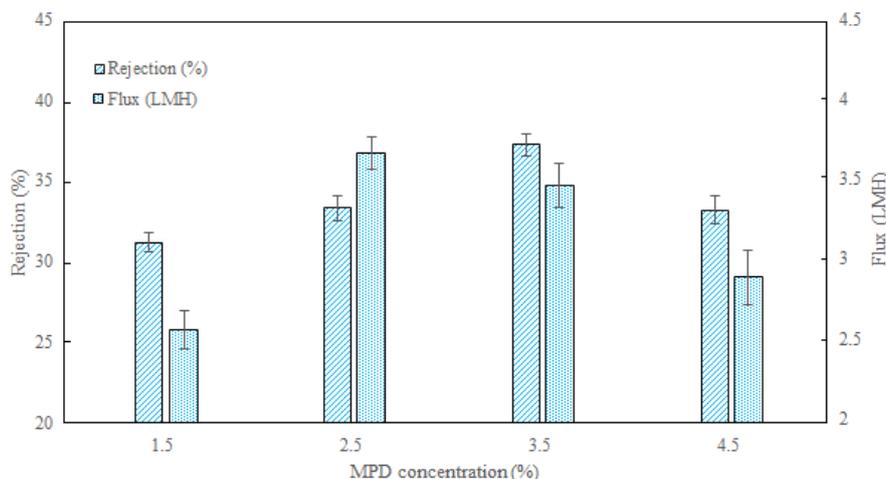


Fig. 4 Rejection and permeate as a function of MPD concentration

C–N band (stretching band).

In ATR-FTIR test, the calculated minimum penetration depth of radiation was about 0.45–0.55 μm . Therefore, ATR-FTIR spectrum of the 0.45–0.55 μm top of the membrane surface could be analysed. As a result, radiation penetrates into the polysulfone region and characteristic band of polysulfone layer could be observed (Saha and Joshi 2009). IR spectra of PSf layer are peaks around 1150, 1244, 1297 and 1320 cm^{-1} correspond to asymmetric stretching of O=S=O, stretching of C–O–C, asymmetric stretching of O=S=O and C–H of C(CH₃).

3.2 Effect of concentration of reactant on acetic acid rejection and flux

Monomer concentration greatly affects the acetic acid/water separation. Therefore, the effect of monomer concentration was investigated to find the optimum value. Fig. 4 shows the experimental results of water permeate and acid acetic rejection under different concentrations of MPD. At monomer concentration of 2.5 wt% MPD, water flux is maximum, while it decreases at higher concentrations (3.5 or 4.5 wt % of MPD) or at lower concentration (1.5 wt % of MPD). Driving force of MPD penetration in the organic phase increases with increasing of MPD concentration which, makes the polyamide layer thicker. Therefore, flux decreases with increasing the thickness of the polyamide layer. As previously noted, due to low solubility of TMC in water and higher solubility of MPD in hexane, MPD monomers penetrate into the organic phase and polymerization reaction occurs in this phase. As MPD concentration decreases, penetration of MPD monomer into the organic phase decreases. On the other hand, the reaction zone is enriched with TMC monomer. Therefore, decreasing MPD and increasing TMC monomers at reaction zone caused a denser polyamide layer and as a result, flux decreases (Xie *et al.* 2012). Therefore, based on the interaction between the aforementioned factors, there is a maximum in the flux.

It should be noted that a similar trend is observed for rejection and the maximum is at 3.5 wt% of MPD. Polyamide should be completely connected to the polysulfone layer. If MPD concentration exceeds from optimum, a gap may be formed between polyamide and PSf layers. This distance characterized by a tiny bubble on the membrane. This phenomenon reduces the performance of TFC membrane. Also, lower concentrations of MPD result in ineffective

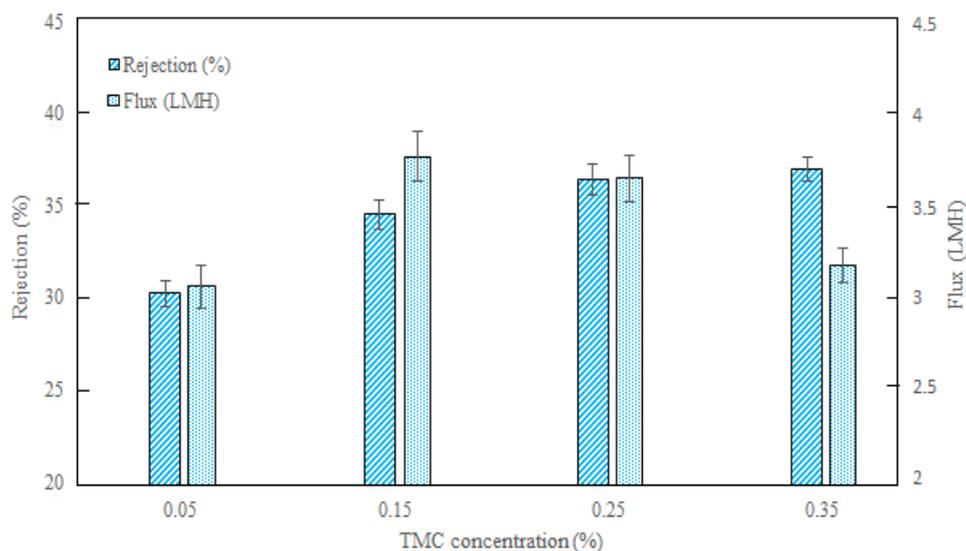


Fig. 5 Rejection and permeate as a function of TMC concentration

polyamide layer on PSf.

TMC concentration determines the polymerization reaction rate while the polymerization reaction time determines the amount of crosslinking (Buch *et al.* 2008). In low concentrations of TMC, polymerization reaction is controlled by its diffusion while at higher concentrations, this reaction is limited by MPD diffusion (Chai and Krantz 1994). Changes in TMC concentration affects amine/acyl chloride molar ratio and therefore the density of polyamide layer changes. Amine/acyl chloride molar ratio decreases with increasing TMC concentration. This decline increases the density of polyamide layer and hence leading to lower permeation. On the other hand, at low concentrations of TMC (where interfacial polymerization reaction was controlled by TMC concentration), low concentration of acyl chloride in the reaction zone increases the thickness of polyamide layer. Fig. 5 presents the effect of TMC concentration on acetic acid rejection and flux.

3.3 Effect of curing temperature on acetic acid rejection and flux

Curing is one of the most important parameters in the manufacture of TFC membranes. The optimum curing conditions such as temperature depend on the organic solvent (Ghosh *et al.* 2008). Curing process is often used to facilitate the removal of residual organic solvent from polyamide thin films. Moreover, it promotes additional crosslinking by dehydration of unreacted amine and carboxyl groups (Soroush *et al.* 2011). With increasing the curing temperature, organic solvent will completely evaporate. This evaporation avoids any defect in the structure of polyamide layer. The effect of curing temperature on water flux and acetic acid rejection are depicted in Fig. 6.

Since the membrane was quickly placed in curing bath, hydrolysis of unreacted acyl groups ($-COCl$) of TMC occurs. Also, this phenomenon is faster at higher temperatures. Therefore, more crosslinking occurs with increase in temperature. As a result, acetic acid rejection increases while permeate flux decreases. If the curing temperature is high enough, water will evaporate from the membrane surface. Therefore, high temperatures may result in the shrinkage of polysulfone layer

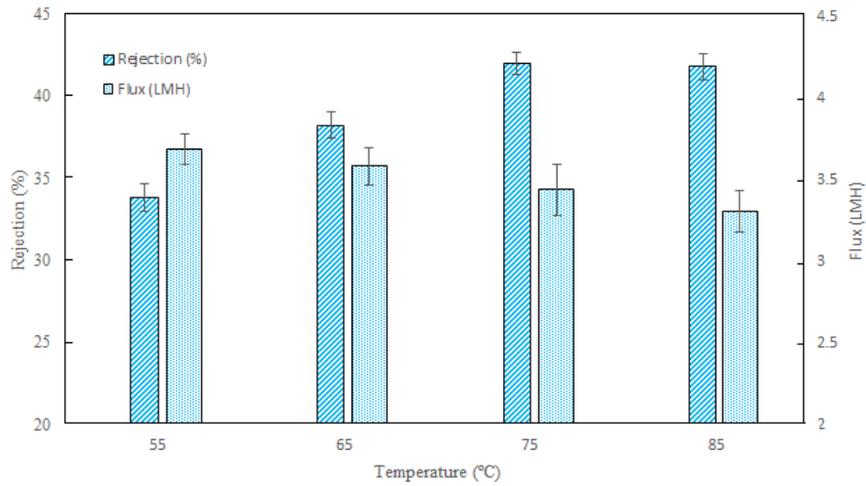


Fig. 6 Rejection and permeate as functions of curing temperature

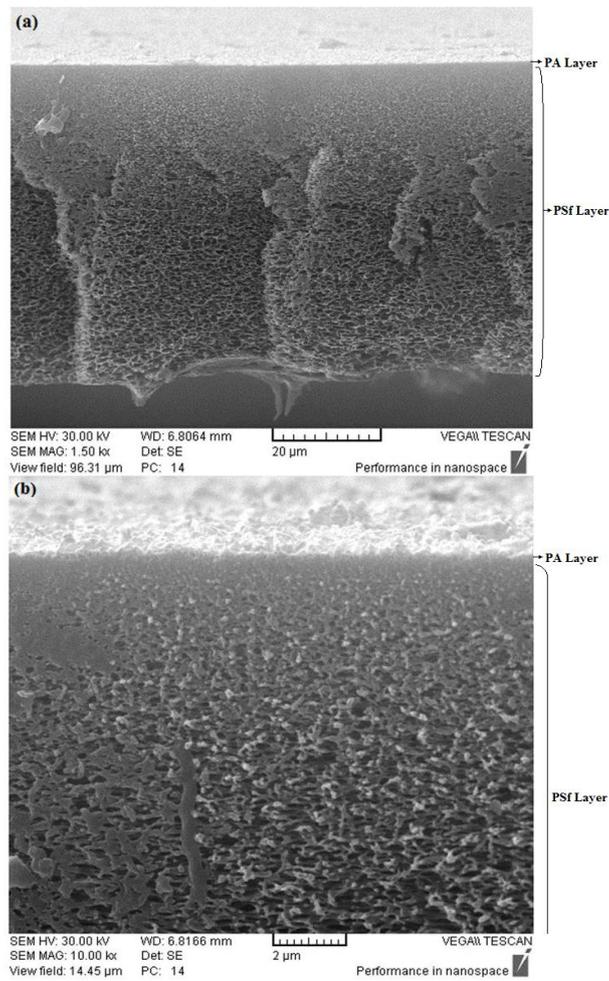


Fig. 7 SEM image of cross-section of polyamide membrane under (a) 1500 (b) 10000 magnification

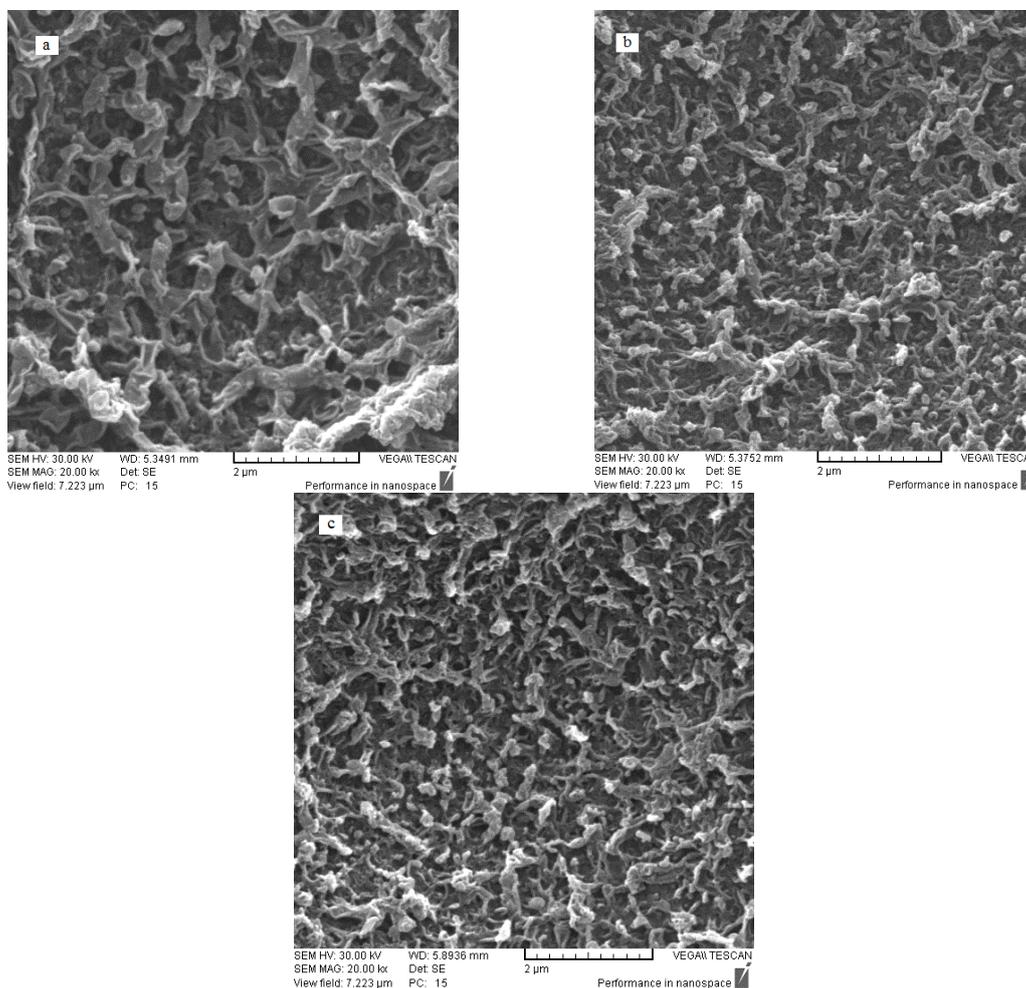


Fig. 8 SEM images of surface membrane when the curing temperatures are (a) 55°C (b) 65°C and (c) 75°C

and the flux reduces significantly. However, evaporation is not complete at low curing temperatures. Therefore, residuals of organic solvent result in the reduction of crosslinking (Meihong *et al.* 2008).

3.4 Morphological structure of the TFC

Membrane structure was analysed via SEM and AFM. SEM images of the cross section of TFC membrane are provided in Fig. 7. Asymmetric and sponge-like structures of ultrafiltration polysulfone membranes are depicted in this figure. The porosity of polysulfone membrane gradually reduced from the bulk to the surface of membrane. Also, a polyamide layer with very low thickness and high density can be observed in this region.

SEM images of the membrane surface are shown in Fig. 8. According to this figure, as temperature goes up, the entanglement of polymer chains increases and as a result density of polyamide layer increases. At the molecular level, transitional motion of macromolecules and the

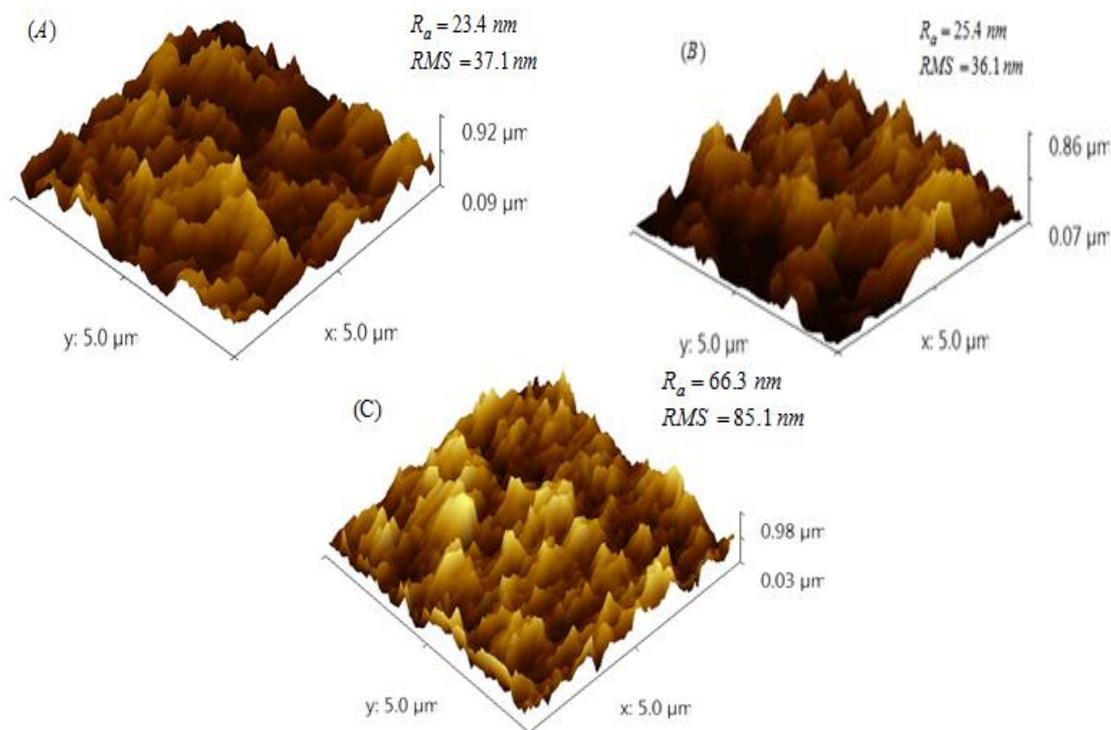


Fig. 9 AFM images of surface membrane when the curing temperatures are (A) 55°C (B) 65°C (C) 75°C

probability of the interaction between functional groups increases. Therefore, the reaction rate and crosslinking of polymer layer increase. In other words, the polyamide layer has a gel structure at the beginning and becomes compact after curing process. As curing temperature increases, the polyamide gel structure becomes more and more compact. Therefore, pore size of the polyamide layer decreases (Buch *et al.* 2008).

One of the important features of the membrane is surface roughness. The possibility of entrapment and aggregation of molecules/ions in the hill and valleys of membrane surface increases by increasing the surface roughness. This causes the fouling to increase (Abu *et al.* 2008). As previously noted, curing temperature has a significant effect on the performance of TFC. However, the relationship of hydrophilicity and surface roughness with curing temperature is not so important. According to Fig. 9, surface roughness increases with increasing of curing temperature. Therefore, the entanglement of polymer chains increases and as a result, surface roughness increases.

3.5 Discussion

The molar mass of acetic acid is $60 \frac{\text{gr}}{\text{mol}}$ and the rejection of this acid from water is very low (about 43%). Therefore, it indicates that separation mechanism of acetic acid is not only molecular screen. According to Koyama *et al.* (1982) and Ozaki and Li (2002) rejection of acetic acid is changed significantly at high pH (more than 5). At low pH, acetic acid is not dissociated while dissociation of acetic acid occurs at high pH. Under acidic conditions, membrane surface has

Table 3 Rejection of acetic acid using RO/NF process

Feed	Membrane	concentration of acetic acid ($\frac{mol}{L}$)	Pressure (bar)	Rejection (%)	Flux ($\frac{L}{m^2hr}$)	Reference
Acetic acid/water	Polyamide composite (RO)	3.33	78.8	60 @ pH=4	-	Koyama et al. (1982)
Acetic acid/water	Polyamide composite (RO)	3.33	78.8	90 @ pH=7	-	Koyama et al. (1982)
Acetic acid/water	polyacrylic acid composite (RO)	0.0167	50	27.8	3.36	Huang et al. (1998)
Acetic acid/water	Polyamide composite (RO)	0.000167	2.942	33 @ pH =3	-	Ozaki and Li (2002)
Acetic acid/water	Polyamide composite (RO)	0.000167	2.942	100 @ pH =9	-	Ozaki and Li (2002)
Acetic acid/water	Polyamide composite (RO)	0.57	17.2	47 @ T=27°C	9.61	Ragaini et al. (2004)
Acetic acid/water	Polyamide composite (RO)	0.51	17.2	61.9 @ T=21°C	10.8	Ragaini et al. (2004)
Acetic acid/water	Polyamide composite (RO)	0.01	10	46.6	-	Laufenberg et al. (1996)
Acetic acid/Malonic acid/water	Polyamide composite (RO)	0.01	10	54.5	-	Laufenberg et al. (1996)
Acetic acid/Formic acid/water	Polyamide composite (RO)	0.01	10	35.5	-	Laufenberg et al. (1996)
Wastewater	Polyamide composite (NF)	0.0083	1.5	55 @ pH=7.3	-	Choi et al. (2008)
Wastewater	Polyamide composite (NF)	0.0083	1.5	8 @ pH=4.7	-	Choi et al. (2008)
Acetic acid/water	Polyamide composite (RO)	0.05	5	43	4.3	This work

cationic property and because of the carboxyl functional group of acetic acid, this acid approaches the membrane surface easily. Therefore, the rejection of acetic acid is low. In addition, the membrane surface under alkaline condition has anionic properties which caused to reject acetic acid from the membrane surface (Ozaki and Li 2002). Therefore, we can conclude that in addition to molecular weight, the dissociation degree of acetic acid (pH) is important in separation of acetic acid from water. Finally, a comprehensive review of previous articles with the present work is presented in Table 3.

5. Conclusions

Thin-film composite polyamide membranes are synthesised by the polymerization reaction of TMC with MPD on the polysulfone supporting layer. The effects of curing temperature as well as different concentrations of MPD, and TMC on the reverse osmosis performance and surface properties are studied. Surface properties of the TFC membranes are characterized by ATR-FTIR,

SEM and AFM. The conclusions are as follows: The investigation of amino monomer concentration effect on membrane performance indicates that permeate flux and acetic acid rejection are maximum at 2.5 and 3.5 wt% of MPD, respectively. Also, the results show that the rejection of acetic acid increases with increase in TMC concentration while permeate flux is maximum at 0.15 wt% of TMC. The influence of curing temperature on the membrane performance indicates that the density of polyamide layer increases with increasing of temperature. Therefore, permeate flux decreases while acetic acid rejection increases with increase in curing temperature. Also, SEM images of polyamide surface confirms the increase in density of polyamide layer with increase in curing temperature. Finally, at optimum conditions, the permeate flux and acetic acid rejection are $4.3 \frac{L}{h m^2}$ and 43%, respectively.

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