Performance of aromatic polyamide RO membranes synthesized by interfacial polycondensation process in a water–tetrahydrofuran system

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ARTICLE INFO

ABSTRACT

In the present study, an attempt is made to synthesize thin film reverse osmosis membranes based aromatic polyamide polymer. Poly(m-phenylene isophthalamide), an aromatic polyamide, is synthesized in the laboratory by high-speed stirring interfacial polycondensation of m-phenylene diamine and isophthaloyl chloride in water and tetrahydrofuran solvents, respectively. FTIR results confirm the polymer synthesis. The yield of the polymer obtained is ∼100% of the theoretical value with an inherent viscosity of 1.99 dL/g. Different RO membranes were synthesized from this aromatic polyamide (PA) under different casting conditions by varying the casting solution composition and casting thickness using N,N-dimethylacetamide (DMAC) as solvent. As a result, five different membranes were obtained containing PA concentration ranged from 10 to 20% in three membrane casting thicknesses 0.1, 0.2 and 0.3 mm. The membranes were characterized by their physico-chemical properties. Mechanical characteristics showed that the increase in the polymer concentration from 10 to 20% in the membrane matrix as well as the increase in the membrane casting thickness from 0.1 to 0.3 mm resulted in increasing membrane strength and Young's modulus. On the other hand, this increase in the polymer concentration and membrane casting thickness resulted in membranes with poor elongation and toughness. SEM approved the anisotropic structure of the synthesized membranes. The reverse osmosis tests for the synthesized membranes were performed with 4000 ppm aqueous sodium chloride at operating pressures varied from 30 to 50 kg/cm^2. All the membranes were characterized for their salt rejection (%), water flux (L/h m^2) and permeability (L/h m^2 bar). The data obtained for the RO performance showed that the increase in polymer concentration in the membrane content as well as the increase in the membrane casting thickness result in increasing salt rejection and decrease in both water flux and permeability. In 0.2 mm casting thickness membranes, the increase in polymer concentration from 10 to 20% results in an increase in salt rejection from 25.5 to 79.7%. Meanwhile, decrease in water flux and permeability ranged 34–12 L/m^2 h and 0.9–0.3 L/m^2 h bar, respectively also occurred in the membranes having polymer concentrations from 10 to 20% with the same 0.2 mm casting thickness.

1. Introduction

RO is more and more widely accepted as one of the important processes for industrial separations and purifications as well as a source of water supply [1–4]. Besides attempting to enhance solute rejection, solvent permeability, chemical and mechanical stability of the membranes as well as economical availability, intensive attention was also paid to basic research on the relationship between morphological structure and RO performance of membranes as well as to the correlation of structure with the polymeric materials and conditions of membrane preparation [5–7]. As one of the alternate membrane materials, aromatic polyamides are entering the fields of reverse osmosis/ultrafiltration applications

[8–11]. Polyamide membranes are available commercially in two types of configurations, hollow fine fibers and ultrathin composite membrane, for the desalination of different salinities ranging from brackish to sea waters [12,13]. One possible reason for selection of such polymeric materials for the said purpose is their close cross-linked structures, which lead to a critical void size and preferential sorption of water on the membrane surface in contact with salt solution, that from the important criterion for the effective desalination in the reverse osmosis process [3,14]. If the vacancy or pore size is too large, the solute from the feed solution runs a risk of leaking through, and as a result effective desalination cannot be achieved.

The principle of interfacial polycondensation, also known as two-phase process, is to react two multifunctional reactive components at the interface of two mutually immiscible solvents [15]. It is a well-known method for the preparation of a variety of polyamides under very mild conditions starting from a solution of

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a diamine in water and a diacyl chloride dissolved in an organic solvent. Immediately after the two components are brought together a polymer is formed at the interface. If the formed polymer is continuously removed from the interface, e.g. by stirring, this is a valuable method for the preparation of larger amounts of polymer materials [2]. Therefore, interfacial polycondensation seemed to be an interesting method for direct synthesis of polymer membranes [16–18]. The more usual methods in membrane formation consist of special processing of suitable polymers in order to give the material a more or less well defined physical microstructure [19]. Contrary to that, interfacial polycondensation allows the simultaneous polymerization and formation of the membrane [20]. High purity and the equivalence of the reactants, as well as high-speed rate of stirring, are the most important factors affecting the product in the interfacial polycondensation [2]. Since the general process employs the diacid chloride of an organic diacid in an inert water-immiscible solvent and a diamine in a water solution with inorganic alkali, one can see at once that the choice of the proper solvent will also affect on the polymer obtained [21].

This article is concerned with the synthesis of aromatic polyamide (PA) polymer from m-phenylene diamine and isophthaloyl chloride by means of the interfacial polycondensation in water–tetrahydrofuran system. FTIR measurement was used to confirm the polymer synthesis. Additionally, stable polyamide membranes are synthesized using different PA concentrations in N,N-dimethylacetamide (DMAC) as solvent and LiCl as additive. SEM and micro-strain analyzer were used to characterize the surface morphology and mechanical strength of the resultant membranes. The reverse osmosis performance for the synthesized membrane is also evaluated by the measurement of salt rejection, water flux and permeability.

2. Experimental

2.1. Materials

N,N-dimethylacetamide (DMAC), HPLC grade (VWR) was used without further purification. LiCl, m-phenylenediamine (99+%) and isophthaloyl chloride (99+%), analytical reagent grade (Fisher), were used without further purification.

2.2. Synthesis of poly(m-phenylene isophthalamide)

Polymerization was carried out in 500 mL flask with a very high-speed magnetic stirrer. A solution of 4.326 g (0.04 mole) of m-phenylenediamine and 8.48 g (0.08 mole) of sodium carbonate in 120 mL deionized water was placed in the flask. Vigorous stirring was begun and a solution of 8.12 g (0.04 mole) of isophthaloyl chloride in 150 mL of tetrahydrofuran was rapidly poured into the flask from a beaker. Stirring was continued for 5 min. The product thus obtained is a white fibrous precipitate. The polymer was separated by filtration, washed with excess deionized water and thus dried under vacuum at 80–90 °C. The yield of polymer is about 100% of the theoretical value (9.53 g).

2.3. Poly(m-phenylene isophthalamide) flat membrane

Asymmetric flat membrane was prepared from the casting solution containing polymer, LiCl and solvent. The solution was first filtered and evacuated to remove the impurities and dissolved gas and then casted onto a dried clean glass Petri dish and shaped into a film with expected thickness by changing the amount of solution. Petri dish together with the liquid film was immediately placed in an oven. When the solvent was partially evaporated, Petri dish with the membrane was cooled and immersed in deionized water bath

for at least 15 h at room temperature. All the membranes were in deionized water until testing at ambient temperature.

2.4. Viscosity

Polymer viscosity was measured by a Brookfield DV-II+ Viscometer at a constant temperature of 25 °C and expressed as inherent viscosity (η_{inh}). Where $\eta_{inh} = 2.3 \log \eta_{rel}/c$ and η_{rel} is ratio of the viscosities for polymer solution to the solvent.

The test was carried out on solution containing 0.5 g of polymer per 100 mL of concentrated (95–98%) H_2SO_4.

2.5. Membrane characterization

2.5.1. ATR-FTIR spectroscopy characterization
In order to observe the presence of functional groups of the synthesized membrane, Fourier transform infrared spectroscopy (Nicolet 8700, ThermoScientific, USA) with an Attenuated Total Reflection (ATR) unit (ZnSe crystal, 45°) was employed. The membrane samples were completely rinsed with deionized water and then dried in vacuum oven before analysis.

IR spectra of the membranes were recorded in transmittance mode over a wave number range of 4000–650 cm^{-1} at 25 °C.

2.5.2. Mechanical properties
The equipment used for carrying out the mechanical properties was micro-strain analyzer, TA instruments RSA III, USA. Maximum force applied was 500 g with force and strain resolutions, 0.0001 N and 1 nm, respectively. Cross-sectional area of the samples of known width and thickness was calculated. The films were then placed between the grips of the testing machine. The grip length was 5 cm and the speed of testing was set at the rate of 2 mm/min.

2.5.3. Specific water content (S)
2 cm × 2 cm specimens of the membrane were cut. Water adhering to the surface of the membrane was removed by pressing it in between filter papers. The specimens were weighed and then dried to constant weights at 110 °C. The specific water content determined was expressed in grams of water present per cubic centimeter (g/cm^3) of membrane.

2.5.4. Membrane morphology
The morphology of the membrane was investigated using scanning electron microscope (SEM) of a model FEI XL30 SEM-FEG (FEI company). Specimen for SEM measurement was prepared by freezing the membrane in liquid nitrogen. Fresh cut piece was coated under vacuum with a thin layer of Au.

2.5.5. Reverse osmosis test
The water permeability tests were performed for the flat membrane circles for deionized water at room temperature. The tests were conducted with an interval of 5 min at 30, 40 and 50 kg/cm^2 in Sterlitech™ HP4750 high-pressure Stirred Cell, Sterlitech, USA. The feed concentration was kept constant in all the test experiments. Meanwhile, salt rejection and water flux characteristics of the synthesized membrane were conducted for 4000 ppm aqueous sodium chloride solution at room temperature also. The tests were performed with interval of 15 min and at constant operating pressure (40 kg/cm^2). The permeate volumes (fluxes) were measured in milliliters. The concentration of electrolyte (NaCl) in the feed and in permeate was measured using a standardized digital conductivity meter, pH/conductivity meter model XL 20, Fisher Scientific, USA. The pure water permeability constant (A) was expressed in L/h m^2 bar. Permeate fluxes for 4000-ppm aqueous sodium chloride feed solutions were recorded as cubic meter per square meter

of membrane per day (L/h m^2) and salt rejections as percent salt rejection.

3. Results and discussion

3.1. Polymer synthesis

Synthesis of poly(m-phenylene isophthalamide) has been carried out successfully on a laboratory scale by the interfacial polycondensation of m-phenylenediamine dissolved in water with isophthaloyl chloride in tetrahydrofuran in the presence of 2 mole of sodium carbonate per 1 mole of diamines. Fig. 1 illustrates the reaction thought to dominate the polymerization. The yield of the polymer obtained is ~100% of the theoretical value.

It is well known that the length and concentration of oligomer chains in the solution during synthesis are determined by the temperature of the process and the presence of basic and acid additives [21]. The hydrogen chloride formed in acylation of aromatic amines by acid chloride blocks the amino groups of the monomer and oligomers, which prevents obtaining a high molecular weight polymer. To eliminate this, sodium carbonate as a hydrogen chloride acceptor is added to the reaction mixture.

What is needed for a good reverse osmosis membrane is a polymer with good mechanical strength, high permeability to water and low permeability to salt. It was well known that membrane strength is basically dependent on the viscosity [1,2,5]. The inherent viscosity of the optimized product is 1.99 dL/g. This high value indicates a high molecular weight polymer, which in turn, results in casting membranes that can be easily handled and has good mechanical strength.

3.2. Membrane preparation

Aromatic polyamide polymer is not soluble in common solvents. The addition of inorganic salts like CaCl$_2$, LiCl, LiNO$_3$, Li$_2$CO$_3$, etc. is necessary to enhance the solubility of the polymer in the amide type of solvents, like dimethyl acetamide and N-methyl-2-pyrollidone [23]. Different concentrations of the polymer and lithium chloride were added into a polar solvent N,N-dimethylacetamide (DMAC) and heated with agitation to complete dissolution. The solution was casted, shaped into a film and then heated in an oven. The
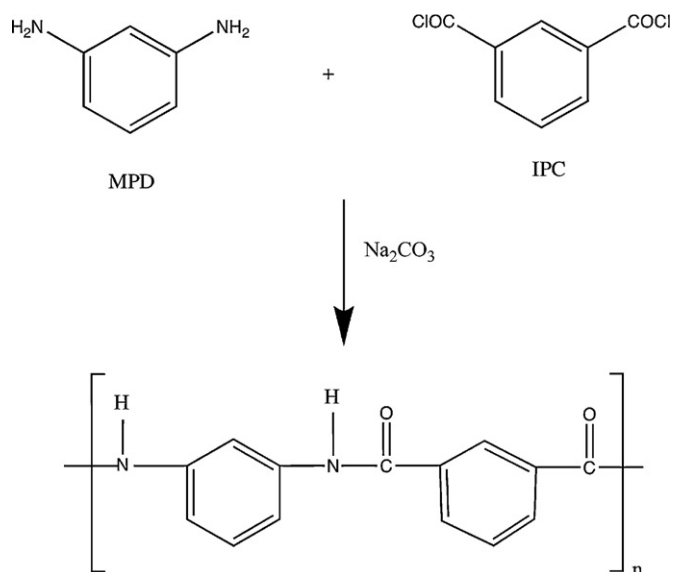
Table 1
Compositional variables of the membrane preparation.

Composition (wt%)			P/S
P	S	A	
10	89	1	0.11
12.5	86.5	1	0.14
15	83	2	0.18
17.5	79.5	3	0.22
20	76	4	0.26

P: polymer, S: solvent and A: LiCl.

transport characteristic of the poly(m-phenylene isophthalamide) membrane can be controlled by adjusting the solvent evaporation temperature and oven exposure time which influence the rate of solvent evaporation in the cast membrane leading to the formation of the pore structure [23]. When the solvent was partially evaporated, the membrane was cooled and immersed in deionized water bath at room temperature. This ensured the preservation of the anisotropic membrane structure as well as completely removes the residual salt and solvent from the membrane.

Different kinds of membranes were prepared applying this procedure using polymer concentration varied from 10 to 20% in three thickness of casting solution: 0.1, 0.2 and 0.3 mm. Lithium chloride was varied between 1 and 4%. The processing and casting conditions employed for the membrane preparation are presented in Table 1. Other variables for the formation of membrane process were fixed as 90 °C and 60 min as the evaporation temperature and time, respectively. Higher oven temperature and longer exposure time results in a membrane of a denser structure and reduced product flux and salt rejection to a less extent [8].

3.3. PA membrane characterization

3.3.1. ATR-FTIR spectroscopy

ATR-FTIR provides a convenient and effective way to determine the functional group of membrane. Fig. 2 shows that the FTIR spectrum is quite similar to the previously published IR spectra of similar PA-membranes [22–24] and shows the absence of the acid chloride band at 1770 cm^{-1}, indicating that successful polymerization has occurred. The band at 1652.7 cm^{-1} (amide I) is characteristic of the C=O stretching vibrations of the amide group (Table 2). In addition, other bands characteristic of PA occur at 1541.7 cm^{-1} (amide II, in-plane N–H bending and C–N stretching vibrations), 1609.6 and 1488.9 cm^{-1} (aromatic ring breathing), and 1249.6 cm^{-1} (amide III). Also, the stretching peak at 3384.2 cm^{-1}
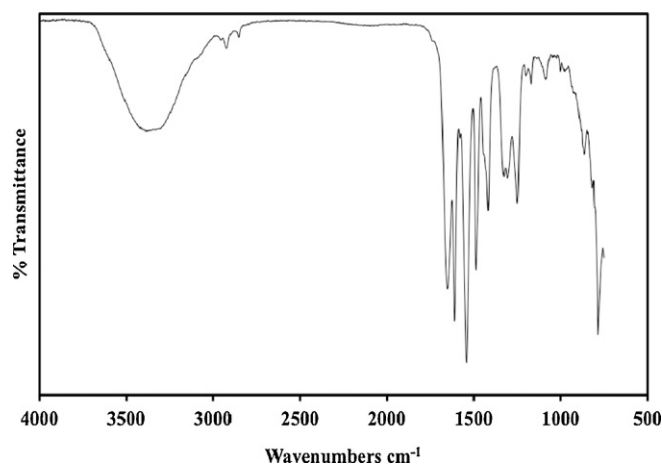

Fig. 1. Preparative reaction to produce cross-linked polymeric polyamide membrane.

Fig. 2. FTIR spectra of laboratory synthesized poly(m-phenylene isophthalamide).

Table 2
The significant peak assignment for FTIR spectra.

FTIR peaks (cm^{-1})	Peak assignment
1652.7	C=O of amide
1541.7	C–N of amide
1609.6	Aromatic ring
1488.9	
3384.2	O–H and N–H

can be assigned to N–H (and O–H) and suggests several loose associative features like NH···N hydrogen bonds as also NH···O=C hydrogen bonds [24,25].

Thus, it can be seen from the experimental data of FTIR spectroscopy that the polyamide membrane prepared is expected to have high degree of cross-linking through polyamide chains with more or less a homogeneous structure. The homogeneity could be disrupted if either of the two reagents MPD or IPC is present in excess. This is because the polymer growth mechanism is such that both the surfaces exhibit different end groups and there is a continuous change in the concentration of end groups across the membrane producing asymmetry [5].

3.3.2. Mechanical properties

Mechanical testing on the rectangular strip of the pure polyamide was carried out at 25 °C. The mechanical properties of the synthesized membranes are presented in Table 3 and typically shown in Figs. 3 and 4. The Young's modulus is calculated from the initial slopes of the linear portion of the stress–strain isotherm. Also, toughness of the membranes is determined by integrating the area under the stress–strain isotherm up to maximum extension, which corresponds to the energy or work required for rupture [6,7].

The results of the mechanical properties show that at a fixed casting thickness (0.2 mm), increasing the polymer concentration from 10 to 20% in the membrane content results in an increase in both maximum strength and Young's modulus ranged 29.8–44.5 and 83.8–416.0 MPa, respectively. On the other hand, this increase in polymer concentration leads to decrease in elongation from 40 to 9.6% and in toughness from 8.3 to 2.0 MPa. Same trends of mechanical properties are also found when increasing casting thickness. Briefly, at constant polymer concentration (15%), an increase in the casting thickness from 0.1 to 0.3 mm results in increasing maximum strength and Young's modulus from 26.7 to 35.9 MPa and 206.4 to 365.1 MPa, respectively. Also, decreases in both elongation from 26.9 to 14.1% and in toughness from 5.2 to 3.2 MPa occurred upon increasing the membrane thickness.
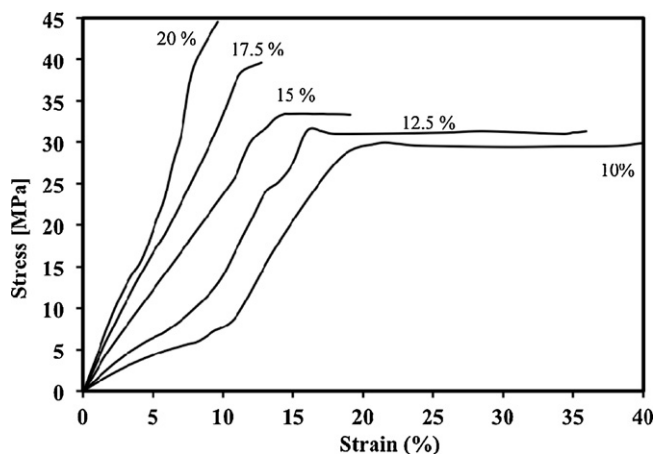

Fig. 3. Stress–strain curves for PA membranes of different concentrations, membranes casting thickness: 0.2 mm.
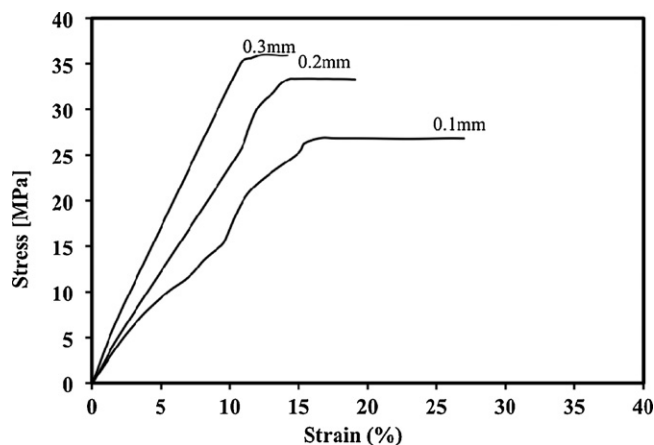

Fig. 4. Stress–strain curves for PA membranes of different casting thicknesses, membranes concentration: 15%.

These mechanical characteristics of the membranes may be attributed to the effect of the rigid aromatic structure. The increase in polymer concentration as well as casting thickness results in the introduction of more aromatic rigidity into the membrane matrix, which causes a higher intermolecular stiffness [24]. This resulted in an increase in maximum strength and Young's modulus. This increase in rigidity was accompanied by a decrease in the membrane elasticity leading to a decrease in the elongation% and toughness [6,7,24].

3.3.3. Specific water content

The specific water content (S) is a measure of the porosity of the membrane. It depends on the casting solution composition, casting conditions, evaporation temperature, and evaporation time. The specific water content decreases with increase in evaporation temperature and period and is ultimately a measure of membrane porous characteristics [26]. For this reason, all membranes are synthesized under constant evaporation temperature (90 °C) and exposure time 60 min. Therefore, any change in the specific water content will be related only to the casting solution composition and casting conditions. The results obtained for membranes prepared under various polymer concentrations under the three casting thicknesses are as shown in Fig. 5. The values reported are for intact membrane as it was difficult to estimate the water content for dense skin alone. As seen from Fig. 5, the specific water content for 0.3 mm membranes decrease from 1.83 to 0.41 g/cm^3, as the polymer content is increased from 10 to 20%. For the same membranes on casting to 0.2 and 0.1 mm observe specific water content between 1.57–0.28 and 1.1–0.11 g/cm^3, respectively. The higher values could be due to condensed water in the voids of the porous layer. Thus, the results confirm that the specific water content depends on the casting solution composition and casting thickness.

3.3.4. Membrane anisotropy

Electron microscopic studies (Fig. 6) reveal that the cast membranes have anisotropic structure i.e., a thin-skin layer (active layer) and supporting core type matrix structure [26]. During the solvent evaporation step, the free surface exposed to atmosphere assumes a dense skin type of structure, and the surface in contact with the glass plate (substrate) retains a more porous structure. This structure was preserved by coagulation in water.

3.4. RO performance under various preparation conditions

The RO performances of membranes synthesized from polymer prepared from the interfacially reacting *m*-phenylene diamine

Table 3
Mechanical properties of the synthesized membranes.

Sample	Tensile strength (MPa)	Elongation (%)	Young's modulus (MPa)	Toughness (MPa)
Membrane concentration (%)[a]				
10	29.8	40	83.8	8.3
12.5	31.4	35.9	125.3	8.2
15	33.2	19.1	236.7	4.1
17.5	39.5	12.7	329.2	2.7
20	44.5	9.6	416.0	2.0
Casting thickness (mm)[b]				
0.1	26.7	26.9	206.4	5.2
0.2	33.2	19.1	236.7	4.1
0.3	35.9	14.1	365.1	3.2

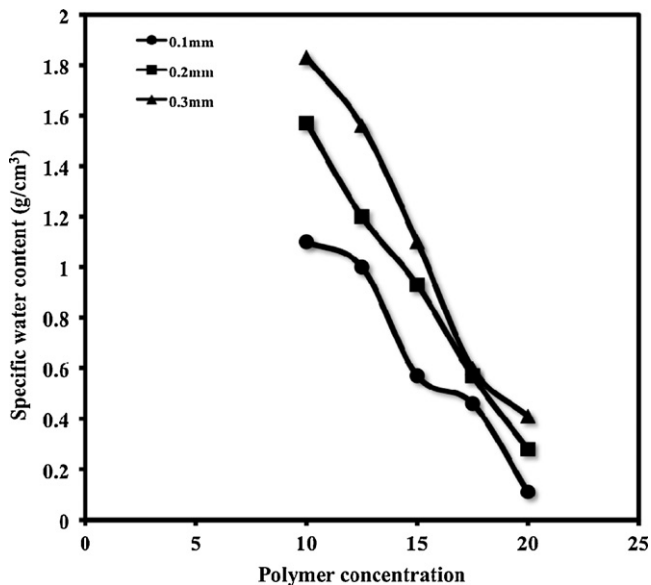[a] Membrane thickness (0.2 mm).
[b] Membrane concentration 15%.

Fig. 5. Effect of polymer concentration on the specific water content of membranes.

with isophthaloyl chloride in water–tetrahydrofuran were examined. Salt rejection, water flux and permeability were measured to examine the membrane performance. Different PA/solvent ratios in three membrane casting thickness were used in the experiments.
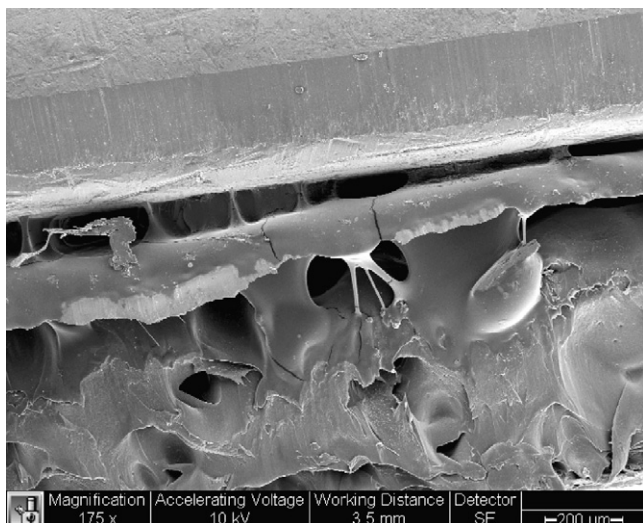

Fig. 6. Scanning electron micrograph of PA membrane cross-section.

Figs. 7–9 show the effect of the change in PA concentration and casting thickness on the RO performance of the membranes. From Fig. 7 it can be seen that the increase in the PA contents from 10 to 20% results in an increase in salt rejection ranged 24–64.5%, 25.5–79.7% and 19–55.6% for 0.1, 0.2 and 0.3 casting thicknesses, respectively. Meanwhile, this increase in the PA content in the membrane matrix results in decreasing water flux (Fig. 8) and permeability (Fig. 9). The decrease in water flux is from 32 to 11 L/m^2 h, 34 to 12 L/m^2 h and 49 to 18 L/m^2 h for the thicknesses 0.1–0.3 mm, respectively. Also, the decrease in permeability is ranged 0.7–0.27, 0.9–0.3 and 1.2–0.44 L/m^2 h bar in the three thicknesses under investigation.

The membrane RO performance is dependent on the molecular weight of the polymer. Polymers of higher inherent viscosity have better RO performance [26]. The polymers having higher inherent viscosity are more rigid and are associated with increased intermolecular hydrogen bonding. Hence they offer higher resistance to water and salt resulting in better RO performance [27]. The inherent viscosity of the synthesized polymer is high (1.99 dL/g) and the mechanical properties of the membranes prove that the increase in polymer content as well as casting thickness resulted in more rigid membranes. This may explain the better RO performance in membranes of higher PA contents and casting thickness.
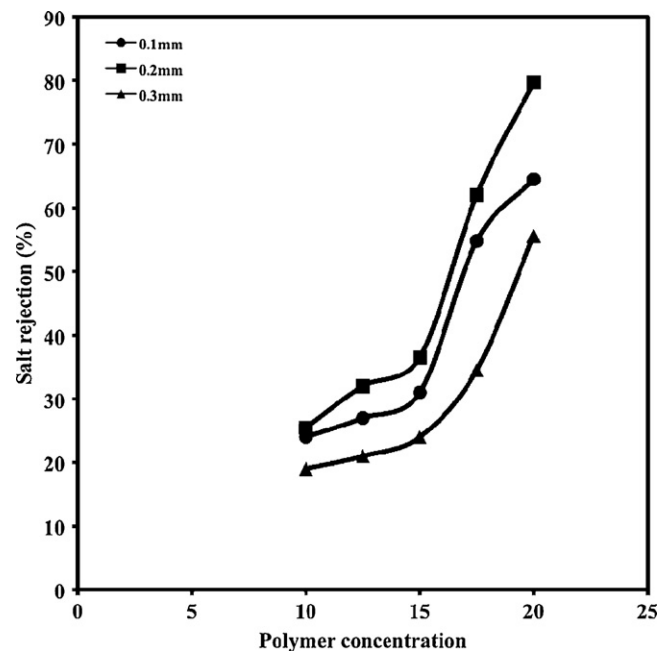

Fig. 7. Effect of polymer concentration on salt rejection (%) performance at different membrane casting thicknesses.
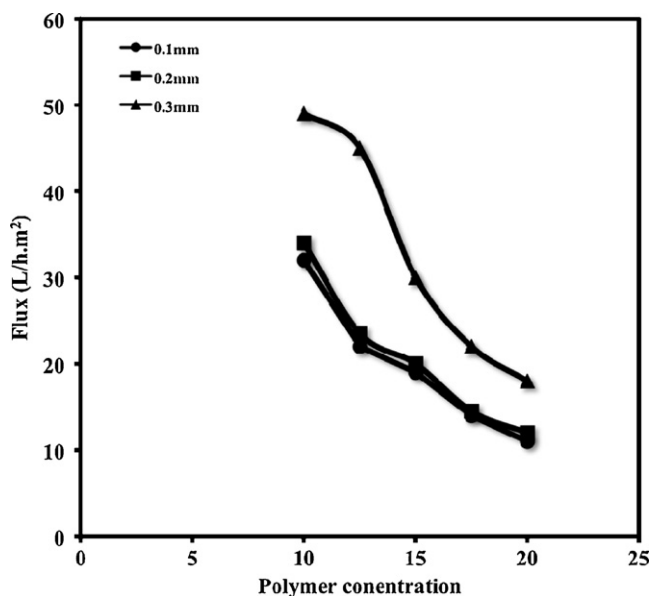
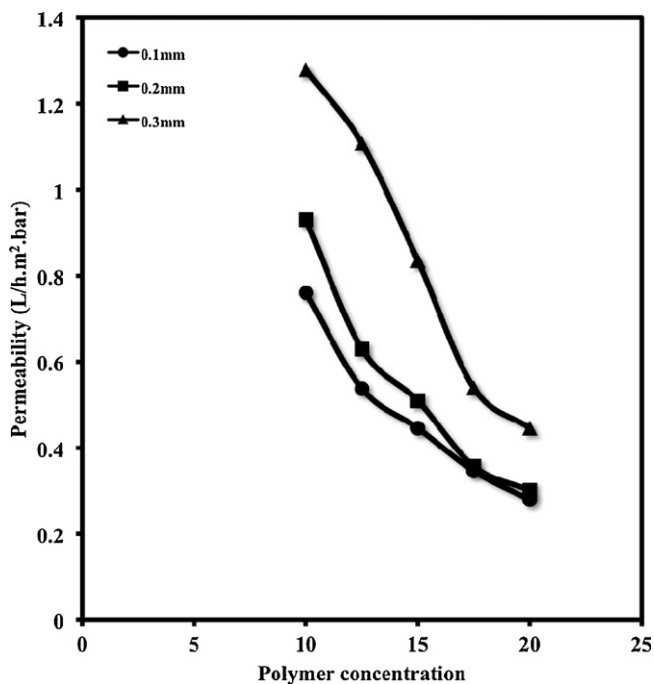**Fig. 8.** Effect of polymer concentration on water flux performance at different membrane casting thicknesses.

Fig. 9. Effect of polymer concentration on permeability performance at different membrane casting thicknesses.

4. Conclusion

In order to develop high performance membranes for the reverse osmosis process, thin film membranes were synthesized from aromatic polyamide. The polymer was synthesized by interfacial polycondensation of *m*-phenylene diamine and isophthaloyl chloride in water and tetrahydrofuran solvents, respectively. Casting of different concentrations from the prepared aromatic polyamide in

DMAC was used for membrane synthesis. Physico-chemical characteristics of the synthesized polymer were investigated. FTIR, stress–strain and SEM were utilized to characterize the synthesized membranes. The results also reveal that the casting solution composition as well as casting thickness has pronounced effect on membrane RO performance.

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