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Synthesis and characterization of stable anion exchange membranes for desalination applications

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ABSTRACT

This research reports the synthesis of stable anion exchange membranes (AEMs) from brominated poly (2,6-dimethyl-1,4-phenylene oxide) (BPPO) and triethylamine (TEA) for desalination applications. The successful synthesis of AEMs was confirmed by Fourier transform infrared (FTIR) spectroscopy. Prepared membranes exhibited dense morphology. The physico-chemical properties of developed membranes were discussed in detail. They exhibited ion exchange capacity of 0.39 mmol/g to 1.98 mmol/g, water uptake of 6.90% to 18.27%, linear expansion ratio of 1.20–10.76 %, and tensile strength of 8.76 MPa to 21.41 MPa. Prepared membranes (M3 & M4) were used in desalination of NaCl by electrodialysis (ED) and their performance was compared with commercial membrane *Neosepta AMX* under same experimental conditions. These selected membranes showed better desalination performance than commercial membrane *Neosepta AMX* under similar experimental conditions.

Keywords: BPPO; Triethylamine; Ion exchange capacity; Anion exchange membrane; Desalination

1. Introduction

The shortage of water resources is one of the world's most concerning problems. Presently, over a third of the world's population suffers from inadequate safe drinking water [1,2]. The desalination of saltwater including seawater (a salinity of about 3.5%) and brackish water (a salinity of 0.05–3%) is an important technology for solving the water crisis [3,4]. To solve this problem, a lot of desalination technologies both thermally-driven and membrane based have been increasingly employed to treat salt water and to increase the limited fresh water supply. Among all desalination applications, electrodialysis (ED) appeared to be the prime choice for modern researchers due to its exclusive properties [5]. The advantages of ED during brackish water treatment over other desalination processes includes lower energy used for desalination, minimal requirement for per-treatment, easier and lower cost of maintenance, higher membrane life due to no bacterial/pathogenic fouling and higher recovery of water [6]. Moreover, ED process has proven its feasibility and higher performance in effluent treatment and recycling of industrial effluents [7,8]. This process has been widely applied for the production of drinking as well as process water from brackish and seawater, the treatment of industrial effluents, the recovery of useful materials from effluents and salt production [9–12].

Ion exchange membranes (IEMs) play an important role in chemical and biochemical industry. As an important part of electrodialysis, AEMs are applied to facilitate the selective permeation of anions [13]. In order to achieve high energy efficiency and long cycle life, they should be prepared with increased ion conductivity, good chemical and mechanical properties, and thermal stability [14]. Thus, the fabrication of AEMs with higher stability is one of the challenging issues for desalination applications via ED process. Shahi et al. reported an alternative method for preparation

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of anion exchange membranes for desalination application [15]. Similarly, Kulshrestha et al. reported the fabrication of hybrid conducting membranes for desalination applications [16]. Moreover, Shen et al. reported the development of internal cross-linked anion exchange membranes with improved dimensional stability for desalination applications Via electrodialysis [17].

Considerable research efforts have been consumed during the last years about the preparation of AEMs, ranging from polysulfone [18,19], poly(ether ether ketone) [20,21], poly(2,6-dimethyl phenylene oxide) (PPO) [22] to organic–inorganic hybrid composite membranes [23,24]. Compared with cation exchange membranes (CEMs), the preparation of common AEMs mainly involves two steps: methylation (chloromethylation or bromomethyation) and quaternary amination. Some authors have reported the drawbacks of the methylation phenomenon in terms of being relatively difficult and environmentally unfriendly [25,26]. Especially for chloromethylation, the commonly employed chloromethyl methyl ether (CEM) and bis-chloromethyl ether (BCME) are highly toxic and carcinogenic, which have been restricted since the 1970s [27,28]. To avoid the use of chloromethylation in the preparation of AEMs, a lot of efforts have been made. Bromination of benzylmethyl and chloroacetylation of phenyl are useful methods to prevent the use of chloromethylation [29–32]. In the light of above discussion, we choose brominated poly (2,6-dimethyl-1,4-phenylene oxide) (BPPO) for the preparation of AEMs in the present research because it is free from the use of chloromethylation process. Moreover, it is very cheap polymer and it provides a thermo-mechanically stable backbone which is necessary for electrodialysis process.

In our previous work we have synthesized a series of AEMs from BPPO by empolying different ion exchange groups [5,33–37]. To extend this, in this research we have reported the synthesis of BPPO-based AEMs by using triethylamine (TEA) as a cationic head group in the membrane matrix. These membranes were characterized in terms of FTIR, SEM, ion exchange capacity (IEC), water uptake (W_R), linear expansion ratio (LER), thermal stability, mechanical stability, membrane area resistance and transport number. Further, their applications in the desalination of NaCl from aqueous solution via ED have also been studied and the obtained results are compared with the commercial membrane *Neosepta AMX* under the same experimental conditions.

2. Experimental

2.1. Materials

Brominated poly (2,6-dimethyl-1,4-phenyleneoxide) (BPPO) was supplied by Tianwei Membrane Co. Ltd., Shandong of China. Triethylamine (TEA) N-methyl-2-pyrrolidone (NMP), silver nitrate ($AgNO_3$), potassium chromate (K_2CrO_4), sodium chloride (NaCl) and sodium sulphate (Na_2SO_4) were kindly supplied by Sinopharm Chemical Reagent Co. Ltd, Shangahi, China. All the chemicals were of analytical grade and used as received. Commercially available ion exchange membranes (IEM) such as anion exchange membrane *Neosepta AMX* and cation exchange membrane *Neosepta CMX* were kindly supplied by ASTOM, Japan. Deionized (DI) water was used throughout this work.

2.2. Membrane preparation

Preparation of membranes was carried out by solution casting method as reported in our previous work [5,33,35–37]. In this method, firstly 8% solution of brominated poly (2,6-dimethyl-1,4-phenylene oxide) (BPPO) was obtained by dissolving it into N-methyl-2-pyrrolidone (NMP) solvent at room temperature. Four anion exchange membranes named as M1, M2, M3 and M4 with different properties were synthesized by adding different concentration of TEA (Table 1) in the polymer matrix. The obtained mixture was stirred at 40ºC overnight. After that it was casted onto glass plate and dried at 60ºC in air for 24 h. After that the membranes were peeled off from glass plate, washed with DI water, and then stored in water. Chemical structure of AEMs is also shown in Fig. 1.

2.3. Characterizations

2.3.1. Instrumentation

FTIR spectra of dried square shaped membrane sample was recorded by using the technique attenuated total reflectance (ATR) with FTIR spectrometer (Vector 22, Bruker) having resolution of 2 cm^{-1} and a total spectral range of 4000–400 cm^{-1}. Morphology of prepared membranes was investigated by SEM (FE-SEM, Sirion 200, FEI Company, USA). The thermal stability of prepared membranes was measured by thermo-gravimetry analysis (Shimadzu TGA-50 H) under nitrogen flow with a heating rate of 10ºC/min within the temperature range 25ºC to 700ºC. The tensile strength of prepared membrane was measured by Q 800 dynamic mechanical analyzer (DMA, TA Instruments) in wet state. The membrane sample with length of 5 cm and width of 1 cm was used for this test. The thickness of prepared membranes was measured in dry sate using screw gauge at room temperature.

2.3.2. Ion exchange capacity (IEC)

IEC of the prepared AEMs was determined by Mohr's method as reported in our previous work [5,33,34,38]. In a typical procedure, AEMs were firstly dried and then immersed in 1 M NaCl solution for 2 d to replace the Br^- ions with Cl^- ions. After that the membrane samples were thourghly washed with DI water and then put into 0.5 M Na_2SO_4 for 2 d. The concentration Cl^- released was measured by titration with $AgNO_3$ solution employing potassium chromate as an indicator. IEC was measured according to the following equation.

Table 1
Composition of anion exchange membranes M1–M4

Membranes	BPPO (g)	TEA (g)	Thickness (μm)
M1	0.8	0.02	59
M2	0.8	0.07	63
M3	0.8	0.12	66
M4	0.8	0.17	54

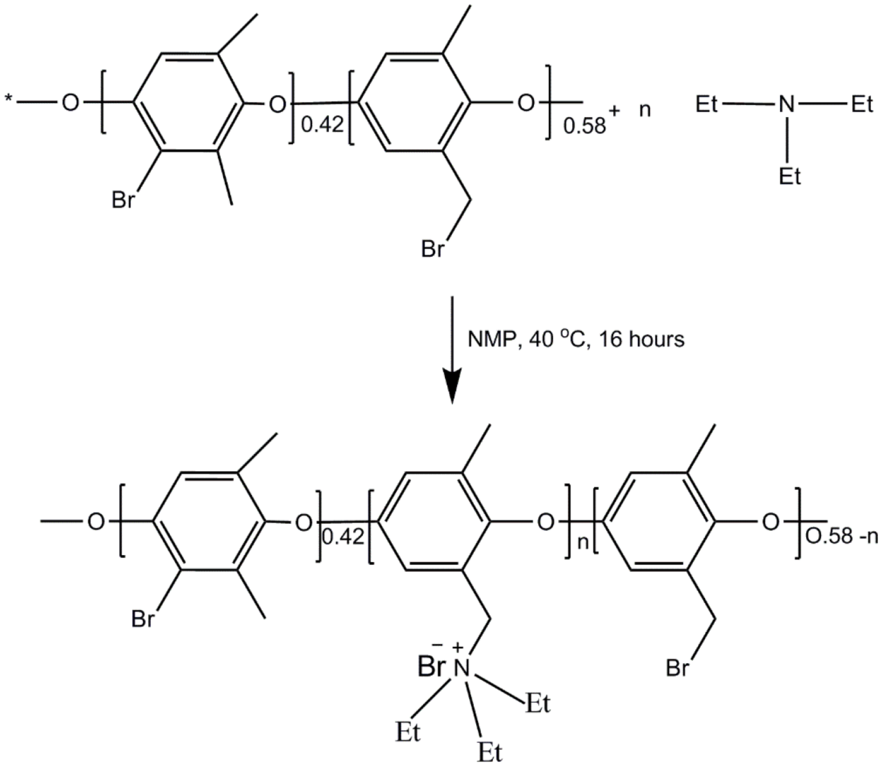

Fig. 1. The preparation of BPPO-based anion exchanged membranes.

$$IEC = \frac{VC}{W_{dry}} \quad (1)$$

where W_{dry} is the weight of membrane sample in dry state, V and C are the volume and concentration of $AgNO_3$ respectively.

2.3.3. Water uptake and linear expansion ratio

In a typical procedure, a piece of membrane was immersed in DI water for 24 h at room temperature. After taking from water, the excess surface water was removed with tissue paper and the mass was measured quickly. After drying at 60°C for 12 h, the weight of dry membrane was again recorded. The water uptake (W_R) was determined by employing the following equation [5,33].

$$W_R = \frac{W_{WET} - W_{DRY}}{W_{DRY}} \times 100\% \quad (2)$$

where W_{WET} and W_{DRY} are the weights of wet and dry membranes, respectively.

The linear expansion ratio (LER) of prepared membranes was investigated cutting them into (2*2) cm^2 pieces at room temperature. It was measured from the following equation [5,34]:

$$LER = \frac{(L_{WET} - L_{DRY})}{L_{DRY}} \times 100\% \quad (3)$$

where L_{WET} and L_{DRY} are the lengths of wet and dry membranes, respectively.

2.3.4. Area resistance

The area resistance of prepared AEMs was measured by commercial cell-assembly (MEIEMP-I, Hefei Chemjoy Polymer Material Co., Ltd.) made up of three compartments under a constant current mode. The effective area of AEM was 7.07 cm^2. Prepared AEMs were equilibrated in 0.5 M NaCl solution for 24 h before measuring the area resistance. After that they were removed from it and adhering solution was wiped off. It was tightly sandwiched between the electrodes. After this 0.3 M Na_2SO_4 solution was provided to the electrodes compartment whereas 0.5 M NaCl was given to the central compartment. Membrane area resistance was measured by using DC power supply (SHEKONIC, Yangzhou Shuanghong Co., Ltd.) provide constant current and digital multimeter (model: GDM 8145), Good will instrument Co. Ltd., Taiwan) measure the potential between the electrodes.

2.3.5. Transport number

It was measured by previously reported method [5,35,36]. In this method, the potential difference (E_m) across the membrane was measured by a multimeter connecting to Ag/AgCl reference electrodes immersed in 0.01 mol/dm^3 NaCl in one compartment and 0.05 mol/dm^3 NaCl in the other compartment. The t^+ was determined from the following equation [5].

$$E_m = \frac{RT\left(2T^+ - 1\right)}{nF} In\left(\frac{C_1}{C_2}\right) \quad (4)$$

where R is the gas constant (8.314 J/K·mol), n is the electro valence of counter-ion (n_i = 1 in this case), F the Faraday constant (96487 C/mol), T the absolute temperature (K), t^+ is the transport number, C_1 and C_2 are the concentrations of NaCl solution in the testing cell.

2.3.6. Desalination by electrodialysis

The desalination performance of prepared AEMs was evaluated using the setup as shown in Fig. 2. It is made up of an anode and a cathode of stainless steel sheets coated with platinum. It has six electrode compartments separated by two CEMs and three AEMs. Each electrode compartment has an effective area of 7.0 cm^2 to the membrane. Anion exchange membranes M3, M4 and commercial *Neosepta AMX* were employed in the experiments for comparison. This experiment was carried out under constant current of 28 mA/cm^2. The feed solution of NaCl with initial concentration of 0.1 M was pumped at 25 mL/min through each compartment. The electrode compartment were circulated with 0.3 M Na_2SO_4 solutions at 25 ml/min to prevent pH change. The salt concentration was measured by conductivity meter after specific time interval. The performance of developed membrane M3 and M4 was compared with the *Neosepta AMX* in term of flux, current efficiency and energy consumption calculated by the following equations [28].

$$Flux = \frac{\Delta N}{At} \tag{5}$$

$$\eta = \frac{Fz\Delta N}{n_c It} \tag{6}$$

$$P = \frac{U\int Idt}{m} \tag{7}$$

where η = current efficiency of dilute (%), ΔN = $C_d^{N-1}V_d^{n-1} - C_d^{n}V_d^{n}$ (mole), C_d = concentration of dilute (M), V_d = volume of dilute (L), P = energy consumption of NaCl (KW h kg^{-1}), n_c = number of cell pair, I = current (A), U = applied potential (V), m = mass of removed salt (kg), and t = time (S).

3. Results and discussion

3.1. FTIR analysis and morphology

The successful synthesis of BPPO based AEMs was confirmed by FTIR analysis. Fig. 3 depicts the FTIR spectra of pristine as well as aminated BPPO membrane. The characteristic band at 750 cm^{-1} corresponds to the C-Br stretching in the pristine BPPO membrane [33,39]. After reaction with the TEA, the characteristic band at 1250 cm^{-1} appeared in the aminated BPPO membrane is due to the C-N stretching vibration which is absent in pristine BPPO membrane indicating the successful reaction between BPPO and TEA. The band at 1608 cm^{-1} is attributed to C=C stretching vibration in phenyl groups; the peak at 1190 cm^{-1} is the characteristic of C-O-C stretching [5,40]. The bands at 1446 cm^{-1} are due to stretching of –CH groups (V and δ) [5,41]. The adsorption peaks of symmetrical and asymmetrical stretching vibration of C-O are at 1200 cm^{-1} and 1306 cm^{-1} and those of phenyl group at 1470 cm^{-1} and 1600 cm^{-1} respectively. Moreover, the signal for C-Br stretching in bromobenzyl groups expected at 740 cm^{-1} to 750 cm^{-1} was not observed in FTIR spectra of aminated BPPO membranes [39,42].

To investigate the morphology of prepared membranes with varying concentration of TEA in the membrane matrix, the SEM images of surfaces and cross-sections were taken by scanning electron microscopy. Fig. 4 shows the SEM micro graphs of surface and cross-section of all prepared membranes. It shows that all the membranes possess dense and homogeneous morphology. It has been observed

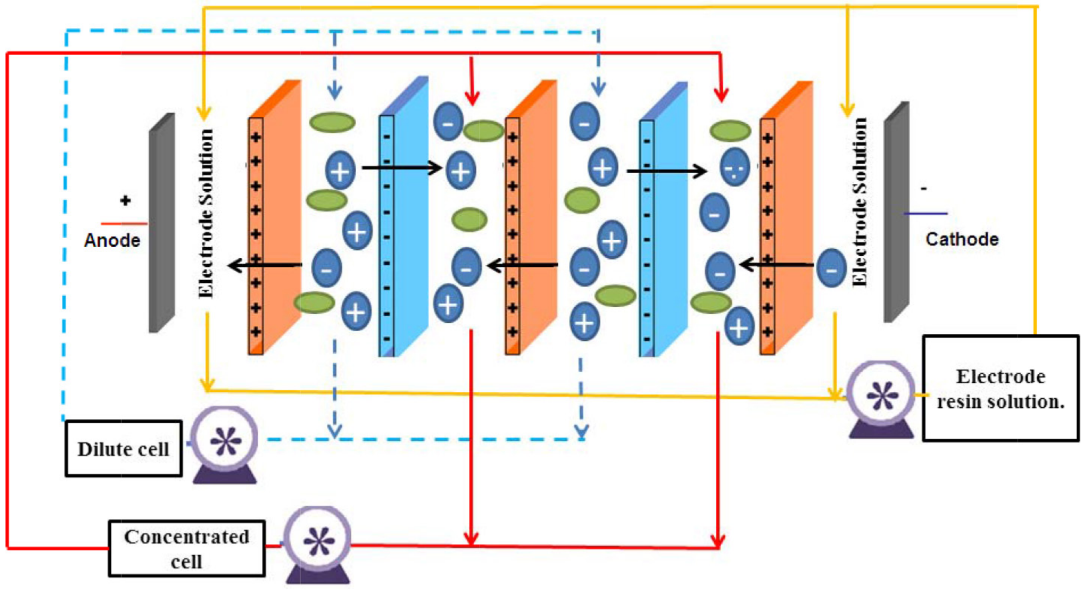

Fig. 2. The schematic setup of ED stack.

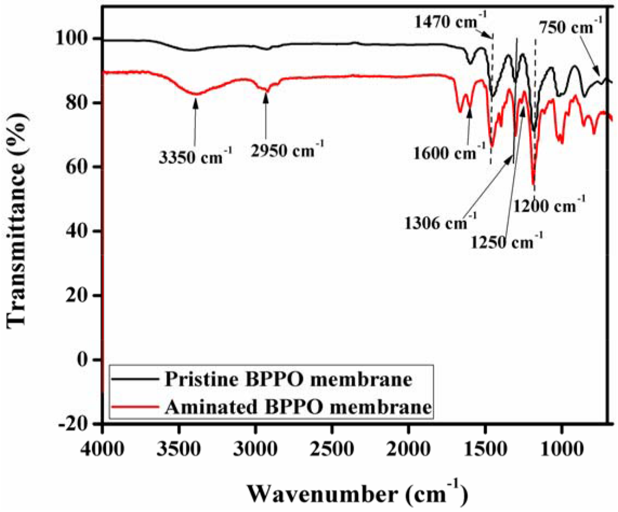

Fig. 3. IR spectrum of different anion exchange membranes.

from the membranes surface that better miscibility can be noticed with increasing the ion exchange contents the polymer matrix. The membrane homogeneity is found to be enhanced with increasing the concentration of TEA in the membrane matrix. The membrane M4 with higher concentration of TEA possess higher homogeneity as indicated in Fig. 4. This morphology of prepared membranes is useful for desalination application.

3.2. Ion exchange capacity

The ion exchange capacity (IEC), measured in meq/g, is an important parameter for determining the performances of AEMs and provides information about the density of ion conducting sites in the membrane [43]. It is related to the number of accessible and exchangeable hydrophilic groups grafted onto the polymer matrix, which are responsible for their charged nature. It was measured by Mohr's method and is shown in Fig. 5. It is clear that IEC values of prepared AEMs are found to be enhanced from 0.39 mmol/g to 1.98 mmol/g with increasing the concentration of TEA in the membrane matrix. Therefore, the prepared membranes are highly charged which are necessary for electro-membrane separation processes.

3.3. Water uptake and linear expansion ratio

Water uptake is an important endowment of AEMs because it provides ions and mass transport channels in the membranes matrix. Fig. 6 depicts the water uptake of prepared AEMs with varying the concentration of TEA in the membrane matrix. It is in the range of 6.90% to 18.27% for membrane M1 to M4. Membranes water uptake is found to be enhanced with increasing the concentration of TEA in the membrane matrix. It is associated to the increased hydrophilic region in the membrane matrix. Both IEC and W_R are important parameters of AEM to describe its hydrophilicity.

Fig. 6 depicts the linear expansion ratio of prepared AEMs in DI water at ambient temperature. It is found to be enhanced from 1.20–10.76% with increasing the TEA contents in the membrane matrix. It is indicated that prepared AEMs have excellent swelling resistance which is responsible for their long time running in desalination by ED process.

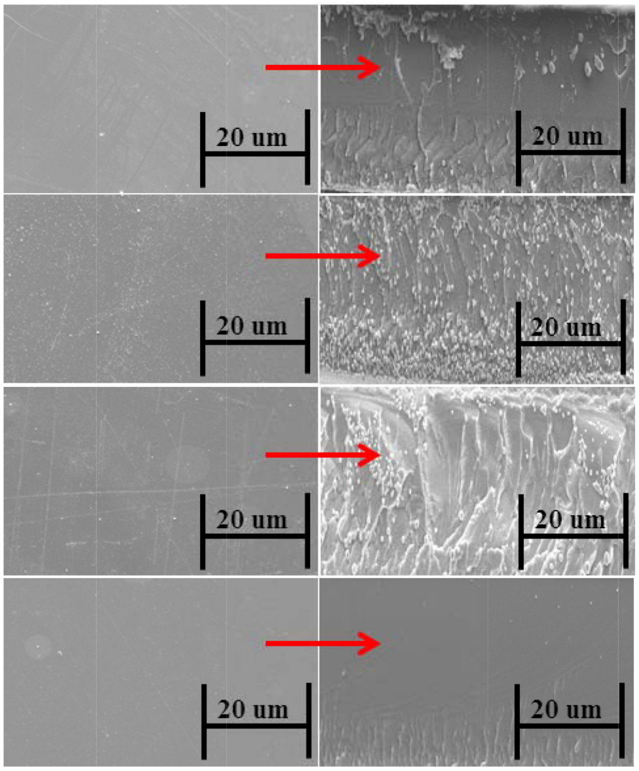

Fig. 4. Surface (left side) and cross-section (right side) of prepared AEMs.

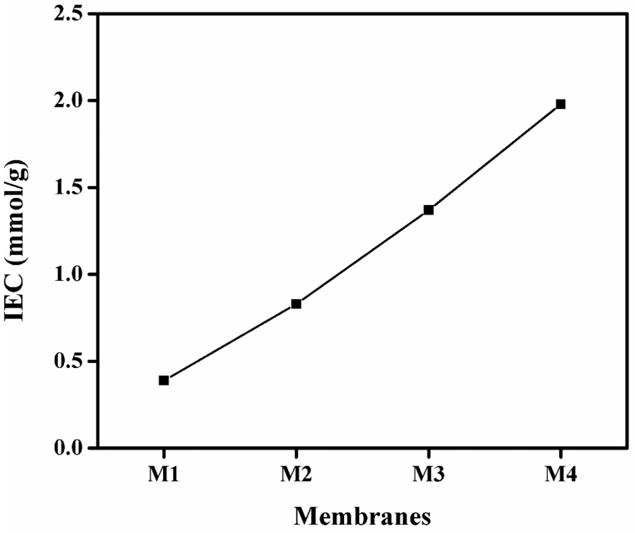

Fig. 5. Ion exchange capacity of prepared AEMs.

3.4. Thermal and mechanical stability

The thermal stability of the prepared AEMs was investigated by employing TGA under a nitrogen flow at a heating

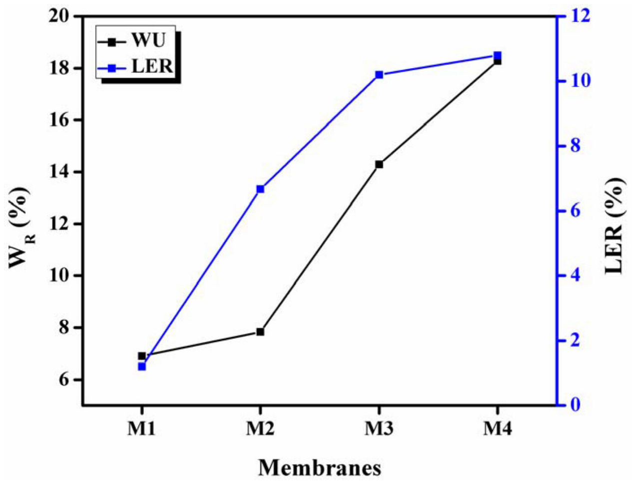

Fig. 6. Linear expansion ratio of prepared AEMs.

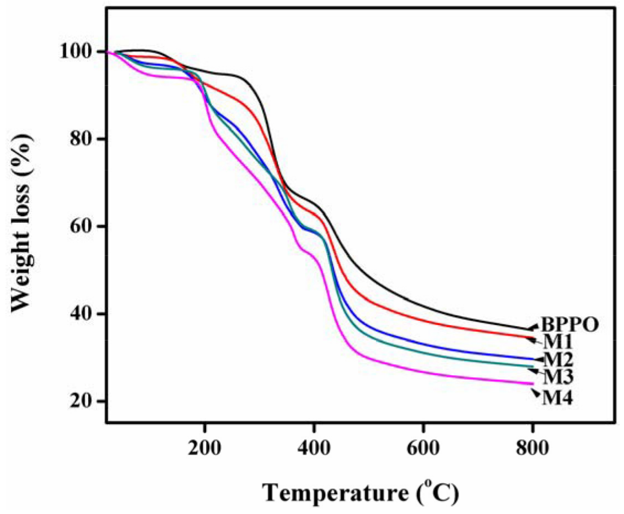

Fig. 7. TGA thermo grams for different anion exchange membranes.

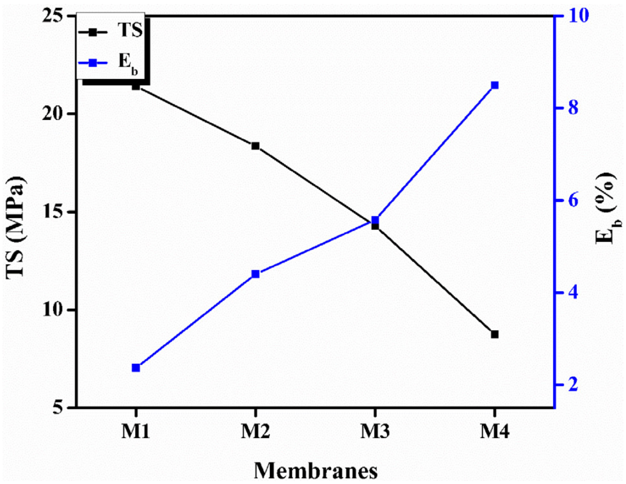

Fig. 8. Tensile strength and elongation at break of prepared AEMs.

rate of 10°C min^{-1} from 40ºC to 800ºC and obtained results are shown in Fig. 7. All of the membranes showed a similar degradation tendency. Three consecutive weight loss steps can be observed from the obtained thermo grams. In the first step around 80–140ºC, there is a small weight loss which is attributed to the evaporation of absorbed water and residual solvent (NMP) from the membrane matrix. This second weight loss around 250ºC is due to the decomposition of quaternary ammonium group in the membrane matrix. The final weight loss around 400ºC is ascribed to the degradation of main polymer chain. All the prepared membranes showed good thermal stability essential for electrodialysis applications.

Fig. 8 shows the mechanical properties including tensile strength (TS) and elongation at break of (E_b) the prepared membanes tested by DMA. All the membranes were investigated in wet state at room temperature. The values of TS are in the range of 8.76 MPa to 21.41 MPa whereas the values of E_b are in the range of 2.37% to 8.5% for the prepared membranes. These results can be explained on the bases of TEA concentration in the membrane matrix. The values of TS are found to be decreased with increasing the concentration of TEA into the polymer matrix which is similar to previously reported membranes [33,36]. On the other hand, the prepared membrane possess the E_b values in the range of 2.37% to 8.50% which are lower than previously reported anion exchange membranes [5]. The membrane with higher tensile strength possess lower elongation at break. It can be observed that prepared membrane M4 with lower tensile strength and higher elongation at break showed higher flexibility. Therefore, prepared AEMs possess good mechanical stability which is essential for desalination of NaCl Via ED process.

3.5. Transport number and membrane area resistance

The membrane transport number of ion exchange membrane is interpreted as the current by the counter-ions [5]. A higher transport number of membrane indicates higher perm-selectivity to counter-ions (anions here). Fig. 9 shows the transport numbers of the prepared AEMs. It is found to be enhanced with increasing the concentration of TEA in the polymer matrix. Prepared membrane M4 possesses highest transport number (0.96) among all the prepared AEMs. It could be associated to the maximum suppression of co-ions diffusion by higher concentration of TEA in the membrane M4. With increasing TEA in the polymer matrix, the hydrophilicity of prepared AEM is found to be increased. It results in the increased transport number of prepared AEMs which is important for better desalination performance. Hence these results indicate that the prepared AEMs are capable toward the chloride ions.

Membrane area resistance has profound effect on the desalination of NaCl Via electrodialysis especially on energy efficiency. It was measured in 0.5 M NaCl at room temperature. Fig. 9 shows the area resistance of prepared AEMs and commercial membrane *Neosepta AMX*. Membrane area resistance of prepared AEMs shows a decreasing trend with increasing the concentration TEA in the poly-

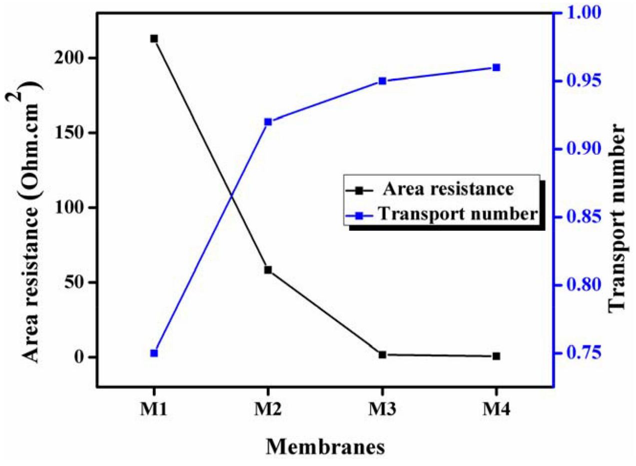

Fig. 9. Transport number and area resistance of prepared AEMs.

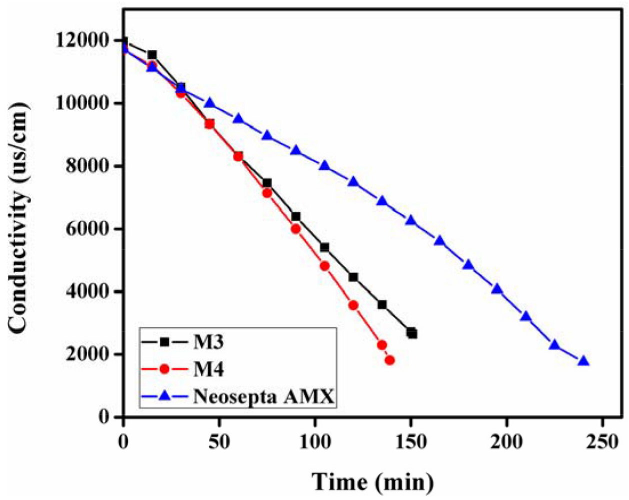

Fig. 10. The change in conductivity of NaCl with time.

mer matrix. This is because that hydrophilicity of prepared membranes increases with enhancing TEA in membrane matrix. Thus it depends on the hydrophilicity of prepared anion exchange membranes.

3.6. NaCl desalination by electrodialysis

Prepared AEMs for electro membrane applications were investigated in desalination of NaCl Via electrodialysis and compared with commercial membrane *Neosepta AMX* under the same experimental conditions. The ED experiments were conducted by flowing 0.1 M NaCl in the dilute cell (DC) with a flow rate of 25 mL/min under the direct current of 28 mA/cm^2. The conductivity of NaCl solution in DC was measured during the experiment. Fig. 10 depicts the change in conductivity of NaCl in DC with time for the selected membranes and commercial membrane *Neosepta AMX*. The conductivity of NaCl in DC is found to be decreased with the passage of time which is associated to the increased amount of ion exchange content in the polymer matrix. This increment in the ion exchange contents results in the increased hydrophilicity of AEMs which is responsible for higher desalination performance of prepared membranes. Hence, prepared membranes possess higher desalination efficiency than commercial membrane *Neosepta AMX* under identical experimental conditions. Moreover, prepared membranes (M3 and M4) exhibit higher desalination performance than previously reported membranes [5,44,45]. Table 2 summarizes the desalination performance of the prepared membrane (M3 and M4) and commercial membrane *Neosepta AMX* under identical experimental conditions. It can be seen that the values of current efficiency and flux of salts for prepared membrane (M3 and M4) are higher than commercial membranes Neosepta AMX under same experimental conditions. Therefore, prepared membranes (M3 and M4) indicated better performance than the commercial membrane *Neosepta AMX* under same conditions. Thus, it can be used for salt removal from water.

Table 2
The membranes ED performance compared to commercial membrane Neosepta AMX

Membranes	Operating current		
	0.2 A		
	η (%)	P (Kw h kg^{-1} salt)	Flux (mole m^{-2} h^{-1})
M3	43.20	64.24	4.61
M4	57	44.75	6.08
AMX	32.80	70.14	3.49

4. Conclusions

In this work, preparation of AEMs from BPPO and TEA has been reported by solution-casting method. The successful reaction between BPPO and TEA was confirmed by FTIR spectroscopy. The SEM micro graph showed the dense and homogeneous morphology of prepared membranes. Prepared AEMs have excellent thermal stability and mechanical stability essential of desalination by ED. These membranes possess ion exchange capacity of 0.39 mmol/g to 1.98 mmol/g, water uptake of 6.90% to 18.27%, linear expansion ratios of 1.20% to 10.76%, tensile strength of 8.76 MPa to 21.41 MPa, area resistance of 212.95 Ω. cm^2 to 0.72 Ω. cm^2 and transport number of 0.75 to 0.96. Compared with the commercial membrane *Neosepta AMX*, prepared membranes (M3 & M4) showed higher desalination efficiency and lower energy consumption under the same experimental conditions. Therefore, prepared membranes (M3 & M4) showed higher desalination performance than commercial membrane *Neosepta AMX* under the similar experiment conditions. This research proved that prepared membranes can be potentially applied for desalination Via ED.

Abbreviations

Code	—	Full name
AEM	—	Anion exchange membrane
AMX	—	Commercial anion exchange membrane

BPPO — Brominated poly(2,6-dimethyl-1,4-phenylene oxide)
CMX — Commercial cation exchange membrane
CC — Concentrated compartment
DC — Dilute compartment
E_b — Elongation at break
ED — Electrodialysis
IEC — Ion exchange capacity
IEM — Ion exchange membrane
LER — Linear expension ration
R — Gas constant
SEM — Scanning electron microscopy
TS — Tensile strength
TEA — Triethylamine
W_R — Water uptake

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