Research Paper

Supporting Polyvinylchloride Polymeric Blend Membrane with Coated Woven Fabric

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Article info

Keywords

Highlights

- New blend membranes were prepared using coated woven fabric.
- The prepared membranes have been characterized.
- Membranes performance results show high salt rejection and good flux.

Graphical abstract

High Flux and Salt rejection (for salty solution 35000 ppm)

Abstract

Blend reverse osmosis membranes were fabricated using polyvinyl chloride (PVC) with cellulose acetate (CA) as polymer blends. Tetrahydrofuran (THF) and N-Methyl-2-pyrrolidone (NMP) were used as solvents. The membrane polymer solution was cast on a coated woven fabric support material. The prepared membranes have been characterized by SEM and mechanical properties. SEM results prove that the prepared membranes are smooth and their pores are distributed throughout the entire surface and bulk body of the membrane without any visible cracks. The stress-strain mechanical test indicates an excellent mechanical behavior. The membranes performance results show that the salt rejection reached 98.4 % at a concentration of 20000 ppm with high flux under pressure of up to 50 bars. Thus, in turn, the prepared membranes can be applied for sea and brackish water desalination through reverse osmosis technology. Also, the prepared membrane was compared with membranes without support and membranes with commercial support.

1. Introduction

The technology of the artificial polymeric membrane has received much attention from both academia and industry. The advantages of membrane technology as compared to other operation technology depend on the separation principle, according to the membrane selectivity that can be performed isothermally with low energy consumption [1-4]. Membrane processes have high energy efficiency, simple operation, easy up and down scaling, and were also operating at ambient temperature, thus preventing any
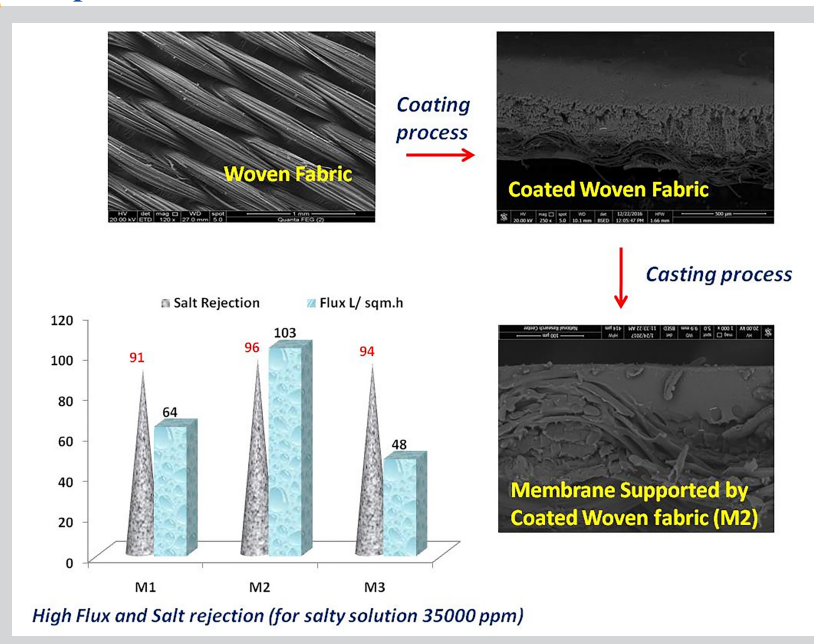
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change or degradation of products [5-10].

The simple definition of the membrane is a semi-permeable barrier that allows transfer of one or more compounds under a certain driving force such as molecules, particles, gases and /or liquid mixture or solution [11-14]. The driving force through the membrane can be found as pressure, concentration, and voltage difference depending on the driving force of the separation and the particle size of the separated species [15].

The most attractive process for desalination to produce fresh water from brackish or seawater is the reverse osmosis process. Desalination using RO membranes requires low energy compared with the other desalination processes, and hence the reverse osmosis process (RO) is more attractive. The dense membranes are used for the RO process under high-pressure for monovalent ion separation like sodium chloride salt. In principle, desalination using RO depends on the membranes and their structure [15-20]. For example, thin film composite membrane (TFC) is prepared by coating the polysulfone or polyether sulfone as a polymeric support by polyamide layer using interfacial polymerization process [21]. TFC membranes are the most commercial RO membranes and could be applied to different grades of saline water according to the polyamide layer on the surface of the membrane. For example, commercial TFC (Filmtec BW30) membranes provided salt rejection of 94.3% for feeding 2000 ppm saline water [22]. The blending method in membrane preparation using different kinds of polymers led to improvement in the membrane performance. However, mixing different blend percentages of cellulose acetate (CA) with polyvinyl chloride (PVC) also provided various grades of RO membranes that could be used in desalination [23]. Where using 3% CA of blending with PVC provided a blend membrane used for Red seawater desalination (38 528 ppm) with salt rejection of 99.95% [23].

Using polymer blending leads to the production of a new kind of membranes that gain the properties of the two polymers or improve the properties of one of them, according to hydrophilicity or hydrophobicity, performance and rejection percentage [24]. In previous studies, the fabrication of the asymmetric PES membrane by blending with manganese (III) acetylacetonate as a metal organic compound in the polymer solution mixture led to improving the hydrophilicity compared to pure PES [13]. Also, Abdallah et al. (2014) prepared a PES/TiO$_2$NTs blend membrane by blending the PES polymer with titanium dioxide nanotubes to produce the membrane applied in membrane distillation, which enhanced the productivity of water vapor to 99% with permeate flux reaching 18.2 kg/m^2.h for salty water of 8000 ppm [14, 16]. Moreover, the effect of blending polyethersulfone with cellulose acetate was studied; however, the results indicated improvement in the permeate flux was 21 kg/m^2.h and the salt rejection reached 99% for salty water concentration of 6000 ppm [17, 18].

Additives like polyvinylpyrrolidone, (PVP), poly(ethylene glycol) (PEG) improve the hydrophilicity and diffusion properties of membranes, due to make pore size distribution on the membrane surface. Therefore, using these additives by blending or addition on coagulation bath can enhancement membrane performance and antifouling properties [25].

To produce a polymeric flat sheet membrane, the polymer solution can be cast on a support such as a glass plate or on a supporting material (woven or nonwoven support) and then immersed into a coagulation bath for membrane formation to occur. Most flat sheet membranes are prepared in the lab without supporting materials that provided weak mechanical properties of prepared membranes compared with supported membranes. For example, the tensile strength of polyethersulfone membrane without support is 25.8 MPa, while the tensile strength of the supported polyethersulfone membrane with woven fabric is 148 MPa [26]. Also, using supporting materials in membrane preparation reduces the shrinkage that often occurs when the membranes are unsupported [26].

This work novelty is for improving the mechanical properties of prepared membranes using a new kind of supporting material, which is the coated woven fabric by PVC polymer on one side of the fabric that improves membrane performance and increases the lifetime of membranes.

In this work, blend membranes of PVC/CA were prepared using the phase inversion method and cast on the coated woven fabric. The produced membranes were characterized using SEM and the mechanical properties and the performance of the membranes were investigated.

2. Material and method

2.1. Material

Analytical grade N-methyl 2-pyrrolidone (NMP) and tetrahydrofuran (THF) as solvents, polyvinyl chloride (PVC), polyethylene glycol 400 (PEG) and cellulose acetate (CA) were supplied by Sigma-Aldrich Company. Textile composite materials from woven fabric were purchased from the Egyptian local market. Commercial non-woven support was purchased from Holykem China Company.

2.2. Preparation of coated woven fabric

Woven fabric on the front side and polyvinyl chloride on the backside was used in this work. Direct Roll Coating was used to make a polymeric coating layer of PVC on the woven fabrics. In this process, the coating liquid was rolled onto the fabric by a roller suspended in the coating solution. A blade was positioned close to the roller to ensure that there was not too much coating solution applied as shown in Figure 1 [27].
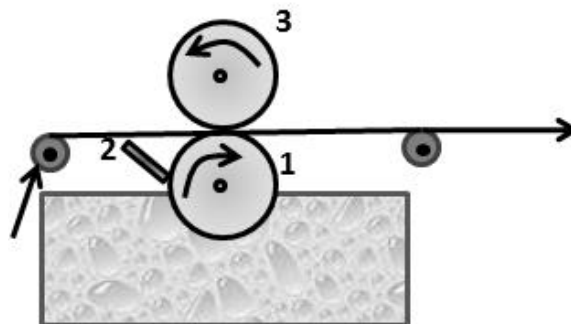

Fig 1. Direct Roll Coating, where (1) suspended roller, (2) blade, and (3) Top roller.

2.2.1. Characterization of coated woven fabric

The characterization of the coated woven fabric depends on some standard specifications like ASTM D-1682 for measuring fabric tensile strength and elongation at break, where this test method covers the raveled strip and cut strip test procedures to determine the breaking force and elongation of most textile fabrics. The rate of air permeability for the support woven fabrics was measured as presented in ASTM D737 - 04(2008) as a measure of its porosity and so its application in water filtration system. The air flow passing perpendicularly at a suitable rate through a known area of fabric was adjusted to obtain a prescribed air pressure differential between the two fabric surfaces. Depending on the rate of airflow, the air permeability of the fabric was determined. Diaphragm bursting strength (ASTM D3786 / D3786M | Developed by Subcommittee: D13.59) was used to characterize the woven fabric support. This test method describes the measurement of the resistance of textile fabrics to bursting using the hydraulic diaphragm-bursting tester. In this test, a 25 mm diameter steel ball is pushed through the stretched fabric and the required force was recorded. The merits of this test were that it could be carried out on a standard universal strength tester with a suitable attachment. In this test, the fabric sample was clamped over a rubber diaphragm by means of an annular clamping ring. An increasing fluid pressure was applied to the underside of the diaphragm until the specimen bursts. The operating fluid, in this case, was a gas [27].

2.3. Preparation of blend membranes

15% of PVC and 3% CA were used to prepare blend membranes which were dissolved in a mixture of solvents with a ratio 1:1 of THF/NMP (81%) and 1% PEG. The polymeric solution was prepared at 600 RPM mixing speed at a room temperature of 25°C and for 18 h. PEG was added in a polymeric solution during mixing as a pore former and as a plasticizer for a polymeric solution [25]. The prepared solution was cast onto in three ways; one on a glass plate without any support (M1), the second one was cast on the coated woven fabric (M2), and the third one was cast on the commercial non-woven support (M3) and all of them were cast with 150 µm thickness and fixed on a glass support. Subsequently, the glass support was immersed in distilled water at room temperature. After membrane solidification, the membranes were stored in fresh distilled water for 24 h until use. Figure 2 indicates the chemical structure for using polymers and solvents to prepare blend PVC/CA membranes.
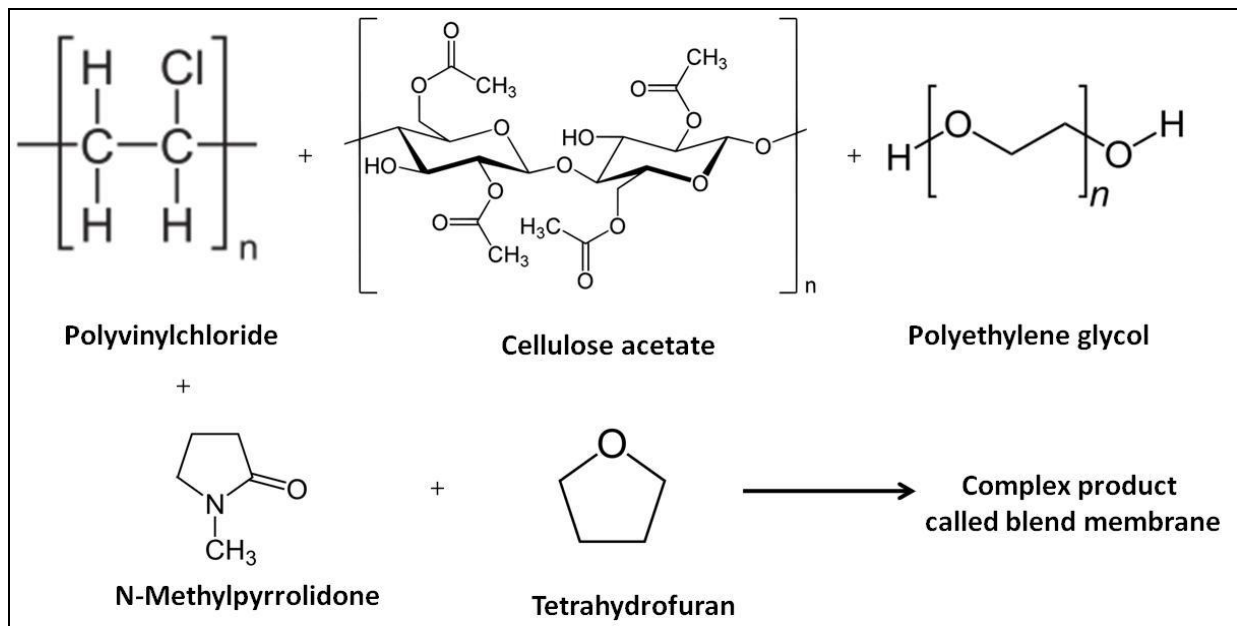
Fig.2. Chemical structure for using polymers and solvents to prepare blend PVC/CA membrane.

2.4. Membrane characterization

2.4.1. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) was used to describe the morphology of the prepared membranes. The samples for the cross-sectional view were coated with gold to provide electrical conductivity using a JEOL 5410 scanning electron microscope (SEM) and conducted at 20 kV.

2.4.2. Mechanical testing

The mechanical properties of blend membranes were studied to determine the effect of supporting materials on the membrane mechanical test. The tensile strength and elongation of the membranes were measured using a mechanical testing system (INSTRON-5500R) with an accuracy of measurement within ± 5%.

2.4.3. Membrane porosity and BET surface area

The inner surface of the prepared blend membranes supported and unsupported was determined using the Brunauer-Emmett-Teller (BET) method. Samples of known weights of the membrane were cut into long strips and placed in a glass column of the apparatus, dried and degassed by heating at 80 °C for 3 hrs. The average area was determined using the BET single point.

The contact angle was measured using a Compact video microscope (CVM) manufactured by SDL-UK. The contact time was 10 s with average drop volume of 10 µl and each value was averaged from 10 measurements. The test method was according to ASTM D724-99 standard for the surface wettability of paper [28].

2.4.4. Determining the membrane structural parameter model

Membrane structural parameter (S) is given by the following equation that depends on the support layer thickness (x), tortuosity (τ) and its porosity (ε) [29]:

$$S = \frac{x\tau}{\varepsilon} \tag{1}$$

The solution-diffusion model was used in this case. The most general description of the mass transport through a membrane was based on irreversible thermodynamics [29].

$$J_A = -D_{AM} K_A \frac{\Delta C_A}{l} \tag{2}$$

where J_A is the permeate flux of solute (L/m²h), D_{AM} is the diffusion coefficient (m²/h) of solute on the membrane, C_A is the concentration of solute (ppm), K_A is the distribution constant and l is the membrane thickness (m).

The osmotic pressure of a feed may be estimated by van't Hoff's equation [29]:

$$\pi = R(T + 273) \sum M_i \tag{3}$$

where T is the temperature (°C) and ΣM_i is the sum of molarities of ions and non-ionic compounds (mol/L) and R is equal to 0.08206 atm L/K mol. Therefore, the fluxes of the water solution can be described by Eq. (4):

$$J_B = -\frac{C_{BM} D_{BM} v_B}{RTl} (\Delta P - \Delta \pi) \tag{4}$$

where, D_{Bm} is the diffusion coefficient (m²/h) of water in the membrane, C_{Bm} is the concentration of water in the membrane (ppm), and v_B is the molar volume of water.

Other than the flux of water, the process is also evaluated in terms of solute rejection, R_J, defined as:

$$R_J = 1 - \frac{C_{AP}}{C_{AF}} \tag{5}$$

where, C_{AP} is the concentration of solute in the permeate (ppm) and C_{AF} is the concentration of solute in the feed (ppm) [29].

Accordingly, the salt rejection can be estimated from arranged equation (6):

$$R_J = \left[1 - \frac{D_{AM} K_A RT C_{BP}}{C_{BM} D_{BM} v_B (\Delta P - \Delta \pi)}\right]^{-1} \tag{6}$$

where, $C_{BM} D_{BM} = 2.7 *10^{-8}$ kg/m.s and $D_{AM} K_A = 4.2*10^{-14}$ m²/s [29]. The model was solved using Matlab R2014b, Simulink.

2.3. Membrane performance measurements

The experiments were performed on the lab desalination unit as shown in Figure 3. This system has a flat sheet membrane module of three openings for feeding, concentrate and permeate. The feed continuously goes through the membrane module from a closed feeding tank (50 liters) using a high-pressure pump. The permeate is collected from a downstream of the membrane module. Fabricated membranes were located in a stainless steel plate module 12 cm in diameter. In all experiments, different synthetic solutions of concentration 20000 ppm, 35000 ppm, and 45,000 ppm were fed continuously to the membrane module up to 50 bar at a temperature of 25°C.
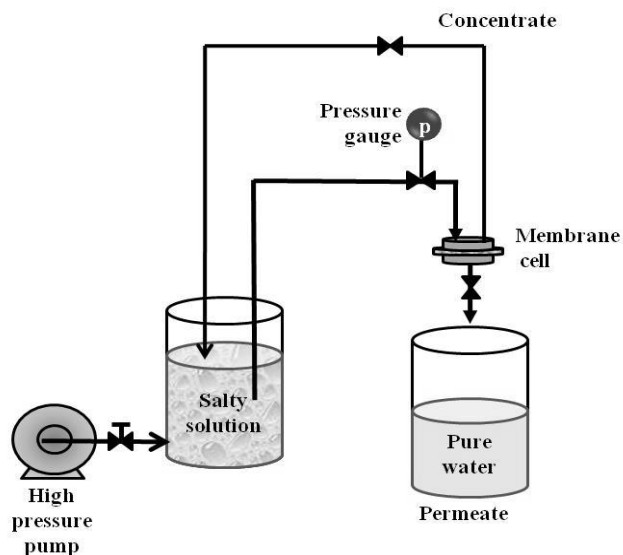
Fig.3. Schematic drawing of laboratory desalination unit.

For this system, the experimental water flux J_{BE} (L/m² h) is given by the following equation:

$$J_{BE} = \frac{V}{A.t} \tag{7}$$

where V is the volume of the pure water permeate (L), A is the effective area of the membrane (m²) and t is the permeation time (h).

The total dissolved solids (TDS) of the produced water were determined using a conductivity meter called Adwa (AD 310), EC/temp meter made in Romania. It has an electrical conductivity range from 19.99 µS/cm to 199.9 mS/cm, where 1mS/cm is equal to approximately 670 ppm.

In addition, the salt rejection (SR%) was conducted in triplicates for each membrane and the average result was calculated using the following equation:

$$R_J \% = 1 - \frac{C_p}{C_f} *100 \tag{8}$$

where, C_f and C_p are concentrations (ppm) at feed bulk and permeate, respectively.

3. Result and discussion

3.1. Characterization of coated woven support

The coated polyester 3/1 woven fabrics were subjected to full physical and mechanical characterization. The thickness of the sample was found at 0.21 mm (210 microns), with a coated fabric weight of 204gm /m². The air permeability measured in cm³/cm².sec proved zero permeation for the tested coated support. The coated fabric was found to bear pressure by measuring its bursting pressure, which gives 10.2 kg$_f$ high values when compared with woven non-coated fabrics.

The warp tension test was found to be 59 kg/5cm of the fabric, while the weft tension gives a higher value of 80 kg/5cm. The same increase in wet elongation percentage of 59% was compared with only 28% of the warp elongation percentage.

3.2. Characterization of prepared membranes on different supporting material

3.2.1 SEM

The SEM views of the prepared PVC/CA membranes using two solvents THF/NMP, which were made by the phase inversion (wet method), revealed that the corresponding structures gave the impression to be pore-free on the top (airside) but highly porous on the bottom (glass side) surfaces. Figure 4 (a) indicates the prepared membrane without any supporting material (M1), which indicates that the dense top layer and finger-like structure in the middle due to using PEG as a pore former. This leads to an improvement in the pore

size distribution [25], and formation of an asymmetric membrane. Figure 4 (b) indicates the cross-section of supporting material, where the woven layer is the bottom layer and the coated top layer is porous. Figure 4 (c) indicates that the SEM snapshot shows the cross section of the blend membrane on coated woven fabric (M2), which has a dense top layer and the bottom layer appears as a fabric woven. Figure 4 (d) illustrates the prepared blend membrane on a commercial nonwoven support (M3), where the dense layer appears on the top of the membrane, with the finger-like structure on the middle and nonwoven support on the bottom. It is clear from Figure 4 (c) that the penetration of polymer solution closed the pores of the coated support and produced a highly dense membrane compared with the membrane supported on the nonwoven fabric, where the visible penetration is low making a membrane consisting of three layers; top dense layer, porous middle layer, and support bottom layer. However, the appearance of a finger-like structure in the membrane is due to the use of lower polymer concentrations of CA and PEG. Also, complete polymer solution penetration depends on the viscosity of the solution [1, 26]. The low viscosity of the polymeric solution leads to rapid penetration of the fabric or support and therefore the surface is not covered with a dense layer of polymer, but the fabric or support pores are clear, visible and not covered. The high viscosity of the polymer helps to completely penetrate the fabric. Thus, a dense layer of polymer covering the surface of the fabric is formed; hence entirely covering the fabric pores [1, 26].

3.2.2 Mechanical properties

The mechanical properties of the prepared blend membranes were studied for the coated woven fabric without membrane layer, the membrane without support, the membrane with commercial support and the membrane on the coated woven fabric. The results indicate that the highest tensile strength was 159.48 kg/m² with elongation of 63.3 mm for the prepared membrane on the coated woven fabric (M2) compared with the lowest tensile of 2.7 kg/m² with elongation of 9.9 mm for the membrane without support (M1) as shown in Figure 5. The results indicated that using coated support reduces membrane shrinkage and wrinkling in addition to the mechanical strength enhancement [1]. Casting on support leads to penetration of the polymer solution into void spaces along the support thickness, which occurs during the interval time between casting and membrane formation in the coagulation bath. According to the surface tension of the top membrane film and interfacial tension of the membrane after coagulation, the support acts as an anchor of the formed membrane leading to limitation in membrane shrinkage and providing high mechanical properties of the membrane [1, 23, 26].

3.2.3. Membrane porosity, contact angle and BET area

Porosity and BET area of the blend membranes are supported and unsupported, the mean pore diameter is in range of the reverse osmosis pore membrane size. The mean pore diameter and porosity are listed in Table 1. It can be found that the pore size of the blend membrane with coated support was in the nano-size due to a coated layer of support, which decreased the pores size of the support. Also, using cellulose acetate produces a dense top layer, which was in agreement with the analysis of membrane morphology. The prepared membrane with coated support provides a porosity of 19.1%, which is the lowest one and lowest BET area according to using coating woven support, which is attributed to reducing the size of pores and provides the best pore distribution on the whole membrane surface. Low porosity of the membrane based on the development of the top dense layer of the membrane due to good interconnectivity occurred between the top layer of the membrane and other layers during membrane formation, which means good interconnectivity occurred between the polymeric membrane and support [26]. Also, solvents affected the membrane structure during the coagulation process. Using Tetrahydrofuran as a solvent only for PVC can provide a non-porous membrane, while the addition of NMP provides porosity for the formed membrane. For example, using THF more than NMP in a polymeric solution led to a decrease in microvoids and the number of pores decreased. Accordingly, we use NMP to THF of 1:1 to get suitable porosity of the prepared membranes [30]. Contact angle measurement is used to indicate the membrane hydrophilicity. The results indicated that using 3% CA leads to improvement in the membrane hydrophilicity. The contact angle for the prepared membranes is close to each other due to the use of similar polymeric solution, where the difference in this work is the support of membranes. The M1 membrane without support has a contact angle of 67°±2, M2; the membrane with coated support has a contact angle of 65°±2 and M3; and membrane with commercial non-woven support has a contact angle of 68°±2, as shown in Table 1. Improvement in hydrophilicity of the membranes is attributed to the acetyl group in cellulose acetate [23]. In addition, the addition of PEG in the polymeric solution increases the hydrophilicity and the diffusive transport properties of membranes, which leads to improvement in the membrane performance [17, 31].
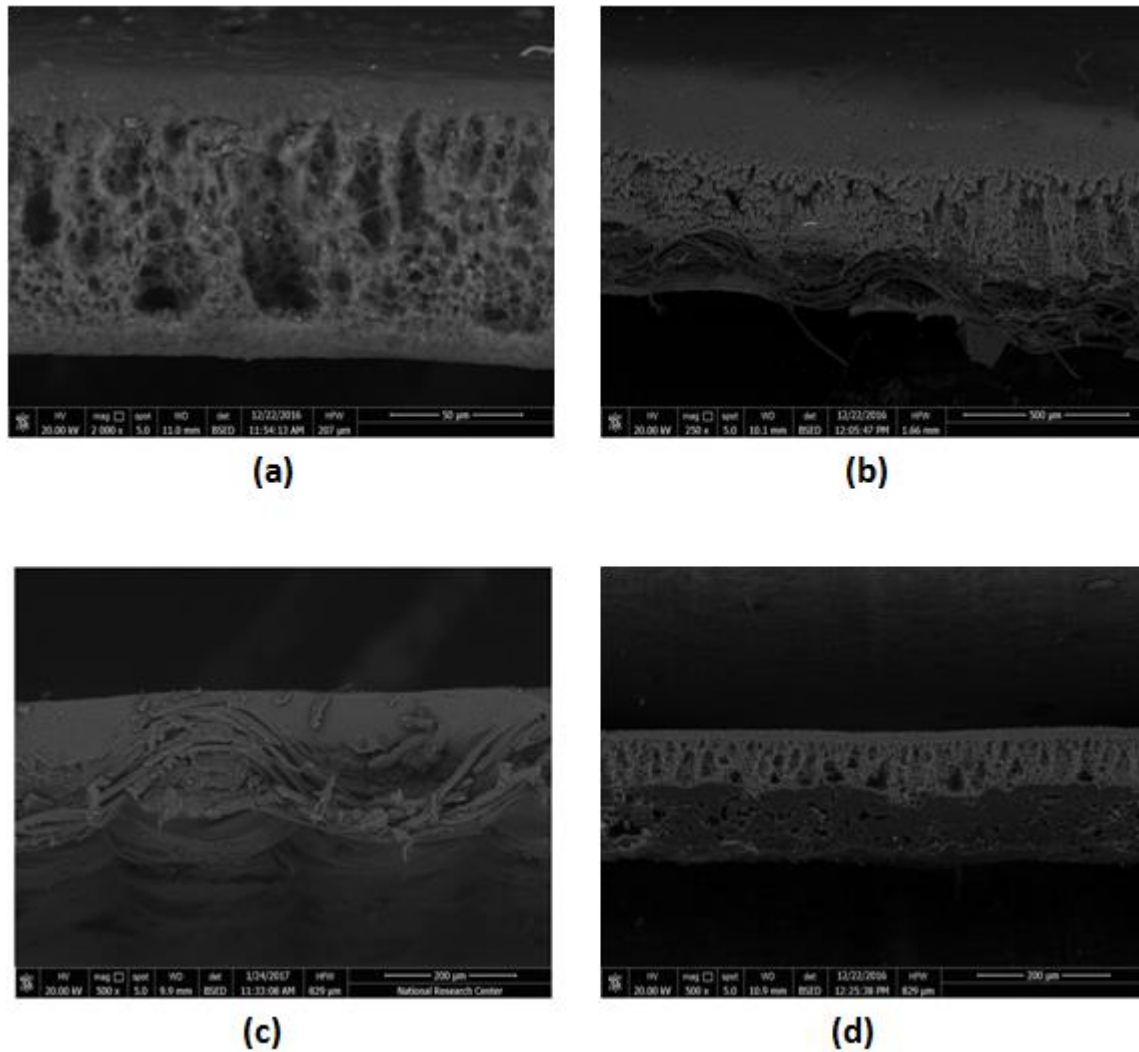
Fig 4. SEM characterization of (a) Cross section of prepared blend membrane without support (M1); (b) Cross section of coated fabric support; (c) Cross section of prepared blend membrane on coated fabric support (M2); (d) Cross section of prepared blend membrane on commercial support (M3).

Table 1
Measurement of BET area and pores characterization.

Membrane Type	BET area m²/g	Mean pore diameter (nm)	Porosity%	Contact angle
Support	19.8	230	89.17	-
M1	5.6E-2	0.27	31.8	67°
M2	0.456	0.342	20	65°
M3	6.2	0.45	23.16	68°

3.3. Determining the membrane structural parameter model

The membrane structural parameter (S) determines the resistivity of the membrane support layer to the diffusion of the salt solution. The low value of the structural parameter is required for any osmotic membranes to a decrease in the concentration polarization phenomena [26].

The results presented in Table 2 show that the S values for support are 0.015 mm, which is the lowest one, meaning that the support can provide the highest permeate flux. Blend membrane without support has 0.33 mm S value, which is also considered a low value that is due to the finer like pore structure of the membrane which improves porosity, while the highest S value is 1.79 mm at blend membrane cast on the coating support due to the condensing dense top layer and lowest porosity, which is 19.1%. Accordingly, this membrane is a reverse osmosis membrane. On the other hand, the prepared membrane with commercial support has 0.96 mm S value, where the commercial support may be more porous than the coated support. Table 2 illustrates the predicted results from the model solution for different

feed salt solution concentrations that indicates high permeate water flux of J_B due to the use of a high operating pressure of 50 bar. The results also indicate that a reduction in flux and increasing rejection at the feed salt solution concentration matched with most desalination research [21, 23].
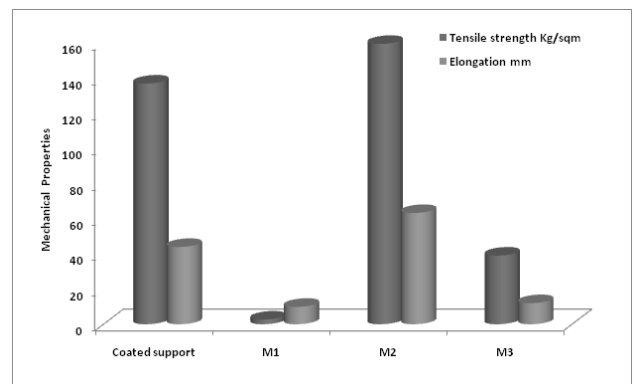
Fig. 5. Mechanical properties of prepared membranes, M1; prepared blend membrane without support, M2; blend membrane on coated fabric support and M3; prepared blend membrane on commercial support.

Table 2
Membrane structural parameter model results.

Membrane Type	S (mm)	JA model L/m².h			JB model L/m².h			R_J % model		
		20000 ppm	35000 ppm	45000 ppm	20000 ppm	35000 ppm	45000 ppm	20000 ppm	35000 ppm	45000 ppm
Support	0.015447	143.3	104.3	98.3	203.3	185.3	156.3	10.5	8.2	5.5
M1	0.334541	34.4	27.5	15.6	87.8	74.6	55.6	98.6	97.3	95.4
M2	1.792356	39.3	30.4	21.2	75.8	55.8	46.2	98.4	97.6	95.8
M3	0.961838	36.6	29.13	18.7	79.8	67.8	49.7	97.9	96.8	95.7
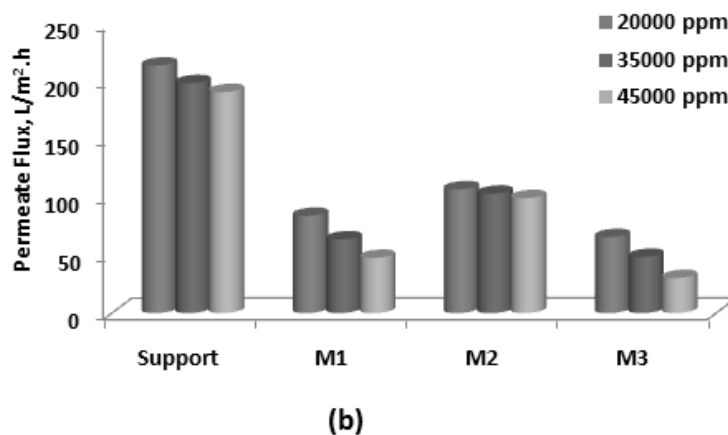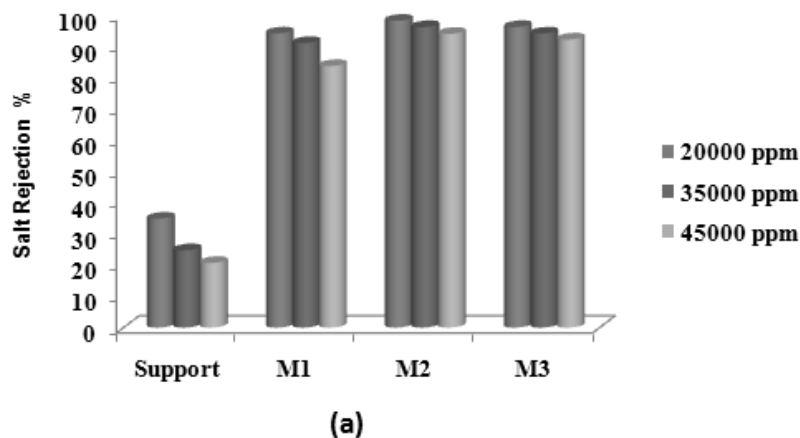
Fig. 6. Membrane Performance test; (a) Effect of feed concentration on the salt rejection of prepared membranes; (b) Effect of feed concentration on permeate flux of prepared membranes, where M1; prepared blend membrane without support, M2; blend membrane on coated fabric support and M3; prepared blend membrane on a commercial support.

3.4. Membranes performance

Effect of different synthetic solution concentrations of sodium chloride (20,000 ppm, 35000 ppm, and 45000 ppm) under 50 bar applied pressure at 25 °C were studied to determine the performance of the prepared blend membranes. The performance of the membranes was studied using support only, membrane without support (M1), membrane with coated woven fabric support (M2) and membrane with commercial support (M3). The results indicate the highest salt rejection and good permeate flux using a membrane with coated woven support. Where the salt rejection reached 98.4 % at a concentration of 20,000 ppm, 96.4% at 35,000 ppm and 94.2% at 45,000 ppm as shown in Figure 6 (a).

Increasing feed concentration leads to a decrease in permeate flux according to the high concentration of salt on the membrane surface, which needs high pressure to facilitate the desalination process depending on Darcy's law, which considers that there is a proportional relationship between the direct discharge rate through a membrane, and also the pressure drop is

taking place according to the viscosity and concentration of the feed [23], that appears at a feed concentration of 45000 ppm, where the rejection reached 94.2% and permeate flux reached 99.3 L/m².h at an operating pressure of 50 bar.

Figure 6 (b) illustrates that the permeate flux of the membrane with coated woven fabric is considered the best flux compared to the membrane without support, while it is natural that the highest flux was obtained in using support only. The permeate flux reached 107 L/m².h at a concentration of 20000 ppm, 103.2L/m².h at 35000 ppm and 99.2 L/m².h at 45000 ppm. The permeate flux and salt rejection experimental results are matched with the predicted data from the model, where the increase in salt concentration provides a decrease in permeate flux and salt rejection percentage.

However, the structure of the prepared membranes has a top dense layer, which is a selective layer that provides good selectivity for salt. On the other hand, increasing salt concentration on the membrane surface leads to a decline in the permeate flux due to clogging of pores and concentration polarization. Also, the low porosity of the membranes can facilitate the

clogging of pores. This problem can be eliminated through membrane surface washing or backwash after getting the decline in permeate flux [32, 33].

4. Conclusions

Polyvinylchloride (PVC)/ cellulose acetate (CA) blend membranes were prepared using different supports coated woven support, commercial nonwoven support and without support. The following conclusions can be illustrated by this work:

- The prepared blend membranes were characterized by SEM and mechanical properties and the results of SEM indicate that the membranes contain a dense top layer and an intermediate spongy like structure and the bottom layer appears as a fabric woven for M2, while M1 (membrane without support) SEM indicates the dense top layer and finger-like structure in the intermediate layer of the membrane. SEM images of the prepared blend membrane on a commercial nonwoven support (M3) indicate a dense layer on the top of the membrane, with the finger-like structure in the middle and nonwoven support on the bottom.
- The mechanical properties of prepared supporting blend membranes provide the highest tensile strength of 159.48 kg/m^2 with elongation of 63.3 mm for M2, while the lowest tensile was 2.7 kg/m^2 with elongation of 9.9 mm for the membrane without support (M1).
- The prepared membrane on the coated support (M2) provides the lowest porosity of 20%, and lowest BET area due to using coating woven support that leads to reducing the size of pores and provides the best pore distribution on the whole membrane surface.
- The performance of prepared membranes was carried out using various salt concentration, and the results indicate that the supporting blend membrane by coated woven fabric provided the highest salt rejection of 98.4 % at a concentration of 20000 ppm and good permeate flux of 107 $L/m^2.h$.
- The solution-diffusion model was studied and the experimental results matched the predicted data from the model.

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