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Investigation of Antifouling Properties of Modified Reverse Osmosis Membrane Containing Natural Extract

D. E. Abd-El-Khalek [1*], S. R. Ramadan [2], A. Morsy [3], B. A. Abd-El-Nabey [4]

[1] *National Institute of Oceanography and Fisheries, NIOF, Egypt,*
[2] *Quality Control & Environment Department, Water Company, Holding Company of Water & Wastewater, Alexandria, Egypt.*
[3] *Materials Science Department, Institute of Graduate Studies & Research, Alexandria, Egypt*
[4] *Faculty of Science, Department of Chemistry, Alexandria University, Alexandria, Egypt,*
**Corresponding author, Email address: dalia1282002@yahoo.com.*

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**Corresponding author, Email address: dalia1282002@yahoo.com.*

Abstract

Biofouling is the major challenge in reverse osmosis membrane technology. In this work, cellulose acetate (CA) composite membrane was prepared by adding extract of halfabarr (Cymbopogon Proximus) as a novel environmental friendly additive to improve the performance and anti-biofouling properties of the membranes. Modified membrane was characterized by Fourier transform infrared spectroscopy, water contact-angle measurements, water content measurements and scanning electron microscopy, while the anti-biofouling properties were studied by static protein adsorption. The effect of modification of the membrane on the salt rejection and water flux was studied using a cross flow RO unit. The results indicated that modified membrane has lower contact angle accomplished with high water content. The extract exhibited pore-forming ability and enhanced hydrophilicity of prepared membrane, as well as increased salt rejection and water flux values. Static protein adsorption results showed that modified membrane resists effectively protein adsorption and microbial growth.

1. Introduction

Membrane technology is one of the most promising ways to produce high quality water [1-4]. Desalination and water reuse has been long acknowledged as a feasible mainstay to address this grand challenge by offering safe and clean water in many arid areas, coastal regions or remote locations. Particularly for water-scarce countries such as Middle Eastern and North Africa Countries that have attempted and implemented all other measures to secure fresh water, desalination may serve as the most viable approach to supply fresh water.

Membranes and other nanoporous materials have been considered as the essential technologies to address global water shortage problem [5]. Extensive product line of water and wastewater filtration systems such as conventional pressure driven seawater and brackish water reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF) and microfiltration (MF) as well as osmotically driven forward osmosis (FO) and pressure retarded osmosis (PRO) have been introduced in the market [6]. Up to now, only cellulose triacetate (CTA)-based and polyamide (PA)-based membranes have been successfully used in the SWRO plants for reliable water securing since the first finding of an outstanding semipermeable membrane of cellulose-based polymers in 1957 by Reid [7, 8]. Cellulose acetate membranes (CAMs) are commonly used in the reverse osmosis (RO) desalination of brackish. The

advantage of using CAMs for this process is that they have favorable chemical and materials properties [9]. CAMs are relatively stable and chlorine tolerant, they have a neutral surface charge, and due to available surface hydroxyl groups they can be readily derivative. Furthermore, they are reproducible, biodegradable, biocompatible, and are relatively low cost because they derive from naturally occurring cellulose [10]. Application of CAMs is restricted to some extent due to the buildup of biological matter at the membrane surface; a phenomenon known as biofouling [11].

Biofouling on membranes can be described as an irreversible deposition and accumulation of bioorganic matter onto the surface of the membranes. This type of fouling is caused by the attachment of microorganisms to the membrane surface and the subsequent growth of colonies on the surface. During the last years, plant extracts were employed as new sources for functional groups in industrial applications such as antiscalants [12-15] and corrosion inhibitors [16] in order to develop new green chemicals for safe environment. Also, it was found that the addition of sunflower (*Helianthus annuus*) seeds extract to cellulose acetate composite increased the hydrophilicity of the memberane and improved its performance [17]. As known, plant extracts can be considered as a rich source of natural chemical compounds that can be extracted by simple methods [12,13].

The genus *Cymbopogon proximus* (halfabarr) of the family Gramineae, locally known as Halfa-bar, is an aromatic, densely tufted grass growing wildly in Upper Egypt [18]. The *Cymbopogon proximusis* highly reputed in folk medicine as an antispasmodic and urolithiasis (renal stone removal), and diuretic agent, and for gout. The plant is used in the treatment of prostate inflammation, kidney disease, inhibition of kidney shrinkages, anthelminthic and for stomach pains. El-Nezhawy et al, 2014 reported that proximol has antioxidant activity [19]. This antioxidant activity of proximol was attributed the plant contents of flavenoids, ruten and quericetine that are well known antioxidant so it have efficacy against the deterioration in renal functions. Aqueous extract of *Cymbopogon proximusis* was investigated as inhibitor of steel [20] and Zinc corrosion [21].

The main objective of this work is to improve the performance and antifouling properties of CA-RO membrane by adding extract of *Cymbopogon Proximus* during membrane preparation process using phase-inversion technique. The modified membranes will be characterized by Fourier transform infrared (FTIR), contact angle, and scanning electron microscopy (SEM) techniques. The performance of the pristine and modified membranes will be assessed for water desalination using water flux and salt rejection measurements.

2. Methodology

2.1. Materials:

CA (molecular weight of 100,000 g mol–1 and 39.8 wt.% acetyl) was supplied by Aldrich and 1,4 dioxane (supplied by Panreac Quimica S.A , Barcelona, Spain). Methanol (purity = 99.5%) and acetone (purity = 99%) were received from Labsolve (Lisbon, Portugal). Acetic acid (purity = 99.8%) was supplied by BDH Anala R (England), and NaCl was purchased from Merck, Aldrich, and the Egyptian Petrochemical Company, Egypt. Egg Albumin powder (purity = 99%) was supplied from Raheja Center, Mumbai.

2.2. Preparation of *Cymbopogon Proximus* extracts:

The leaves were grinding to powdery form after harvesting for 2 h in oven at 70°C. A 5 g of the powder was refluxed in 100 mL distilled water for 1 h to prepare stock solution of extract. The refluxed solution was filtered to remove any contamination. The stock solution was evaporated to obtain extract residue.

2.3.Membranes Preparation:

CA-RO membranes were prepared using the phase-inversion method [22] in which mixture of acetone (13.5 g), dioxane (27 g) and acetic acid (5 g) were used as solvents for CA (8.45 g), while methanol (10.7 g) was used as a nonsolvent. Modified CA-RO membranes were prepared by the addition of 0.3g of Cp extract to this mixture. The thickness of RO membranes was previously selected (250μm) and spread at a constant speed (10 mm s^{-1} using a knife of an automatic applicator (Zehntner 2300-Swiss, Switzerland)). The prepared CA-RO was then post-treated for 10 min at about 80°C–85°C, then soaked in deionized water for 24 h. Each membrane was prepared three times for reproducibility.

2.4.Membranes Characterization:

2.4.1. Chemical structure

FTIR spectrometer (FTIR LX 18-5255 Perkin Elmer) was used for characterizing the membranes. The spectra were recorded in the wave number range of 4,000–400 cm–1. The CA membrane samples had been grinded with KBr in a ratio of 1:10 in the powder form to reduce the particle size.

2.4.2. Morphological investigation

Morphology of membranes was observed using SEM (XL 30 JEOL). The membranes were sputter coated with a thin film of gold under vacuum prior to morphological examination. Furthermore, membranes were fractured in liquid nitrogen for cross-sectional images.

2.4.3. Hydrophilicity measurements

Contact angle

The contact angle of the prepared CA-RO membrane surfaces was measured using Rame hart, Instrument, France. A drop of distilled water (2 μL) was placed on the membrane surface (3 cm× 2 cm) using a microsyringe (Hamilton Company, Reno, NV, USA). The contact angle was measured directly within 10 s, at five different positions.

Water content

Water content can be defined as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of the membrane to the mass of the dry membrane [23]. Water content of the membranes was obtained after soaking membrane in water for 24 h, and the membranes were weighed followed by mopping them with blotting paper. The wet membranes were placed in an oven at 85°C for 24 h, after that they were placed in oven under vacuum, and the dry weights of the membranes were determined.

The percentage of water content (WC%) was calculated using the following equation

$$\textbf{Water content \%} = (W_w - W_d) * 100 / W_w \quad (1)$$

Where W_w and W_d represent the weights of wet and dry samples, respectively.

2.5. Membrane performance

The performance tests (salt rejection and water flux) for membrane sample (area 42 cm^2) were done using a cross flow RO unit (CF042, Sterling, USA). Saline salt solutions of NaCl of 10,000 ppm and pH7 were used. The determination of the total dissolved salt of the permeate water was measured with a pH

and conductivitymeter (430 portable, Jenway, England). The water flux (F) and salt rejection (R) values were obtained using Eqs. (2) and (3):

$$F = V/A*t \quad (2)$$

where V is the total volume of water passing through the membrane (L), A is the membrane area (m^2), and t is the time (h).

$$R = (C_o - C_{memb})x\ 100/C_o \quad (3)$$

where C_o is salt concentration in the feed water side and $C_{memb.}$ is the salt concentration in the permeate side of the membrane [24].

2.6.Antifouling properties measurements:

Static protein adsorption of the membrane:

Adsorptive fouling was evaluated by immersing the membrane in egg albumin as protein feed solution [25]. As described in previous works [17], the amount of adsorbed protein was determined by measuring the concentration of the protein solution before and after adsorption. The concentration of protein was measured using UV spectrophotometer (UV-1601, Shimadzu, Japan). The relative protein adsorbed (RP%) was used to identify the extent of adsorptive fouling.

$$RP\% = C_o - C_a / C_o\ x\ 100$$

where C_o and C_a are protein concentrations before and after membrane soaking, respectively.

Fouling attachment monitoring:

For fouling investigation, different Membrane samples, 2.5 cm x 2.5 cm in size, submerged in Mediterranean seawater for 15 days [17]. The dried membrane surface was observed by scanning electron microscope (SEM) after coating with a gold layer. The density of the microbes on the membrane surface was investigated from the SEM images.

3. Results and Discussion

3.1. Membrane characterization:

Figure 1 presents the cross-section micrograph of prepared membranes showing non-porous structure of pristine membrane which consisted of a skin-layer on the top surface and finger-like pores under this layer. The top skin layer of cellulose acetate membrane was generated by solvent evaporation while finger-shaped voids were formed after annealing treatment [26]. On other hand, cross-section micrograph of modified membrane showing mini-pores in the skin layer which are responsible for rejecting the salt. These mini-pores gradually enlarge from the top surface to the underside of the membrane in which very large channels are apparent. This unique morphology of this modified membrane indicates that extract exhibited the pore-forming effect during membrane formation.

The FTIR spectra of pristine and modified CA membrane are illustrated in figure 2. As seen, the modified membrane has OH functional group (3000–3750 m-1) which makes the membrane more hydrophilic. Moreover, the additional peak at 1640–1630 m_1clarifies the adsorption of water, which not only assists in the make and break of hydrogen bonding of water but also causes easy permeation of water across the membrane [27]. Also, the band at 1,731 cm^{-1} is corresponding to the C=O bond of the carboxylic group. In addition, the peaks at 2,932 cm^{-1} is assigned to the C–H bending, while the band at

1,049 cm–1 probably corresponds to the stretching modes of C–O ether. It is reported that studied extract is rich with flavonoids, triterpenoids, which contain many functional groups in addition to phenolic structures [28].

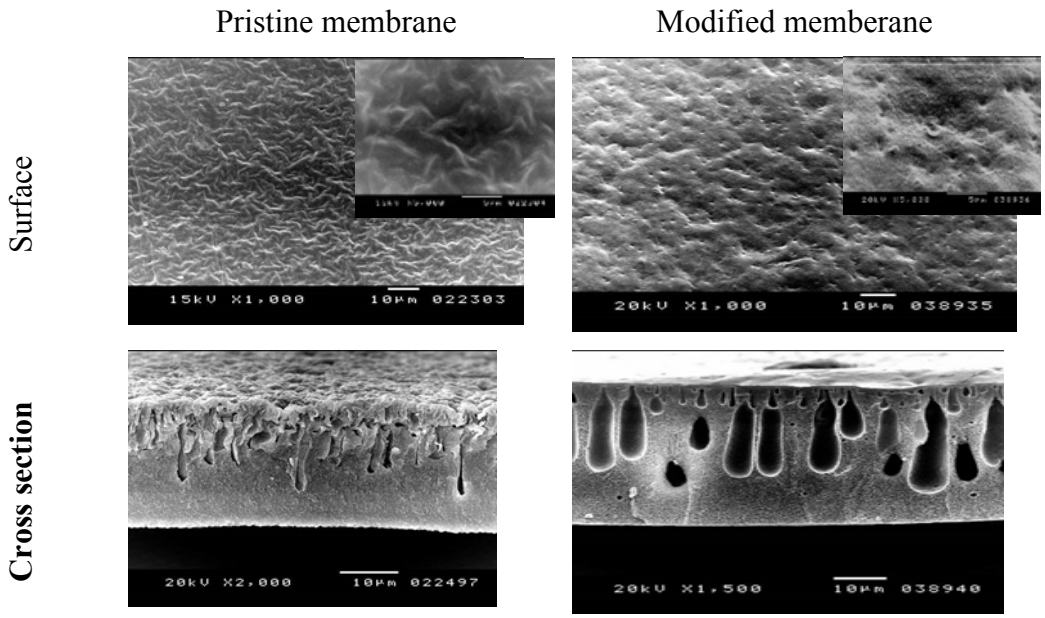


Fig. 1. SEM images of surface, bottom, and cross section of pristine CA-RO membrane and modified membrane containing Cymbopogon *proximus* extract.

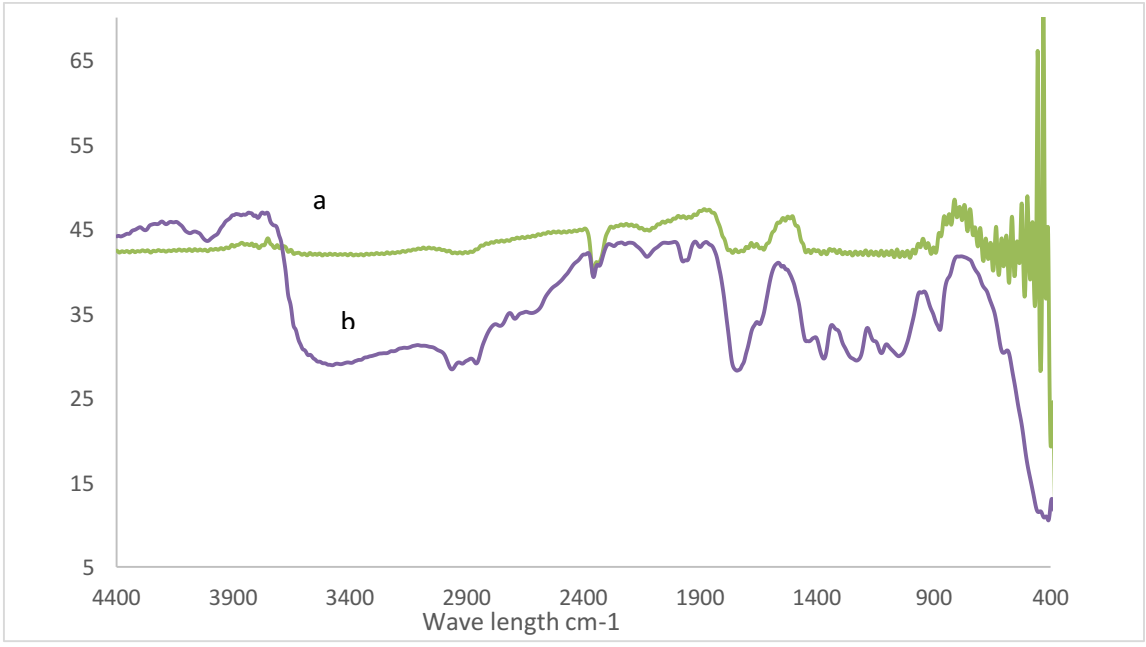


Fig. 2: FTIR spectra of (a) pristine CA-RO membrane and(b) modified membrane containing Cymbopogon proximus extract.

3.2. Hydrophilicity Examinations:

In order to evaluate the surface hydrophilicity of prepared membranes, the water contact angles and water content of these membranes were measured and presented in figure 3 and Table 1. It is clear that the addition of the extract decreases the values of the contact angle from 65.4 to 55.8 (which means 14.7

% decreasing rate) which indicates hydrophylicity improvement, on other hand, water content of modified membrane increased than pristine one (from 71.3 to 73.6 %). It is reported that contact angle and water content are affected by pore size and wettability of internal pore channels [29]. The above results can be discussed on the basis that the existence of hydrophilic functional groups in the extract leads to the improvement of hydrophilicity of the modified membrane which plays an important role in improving the resistance of the fouling [30-32].

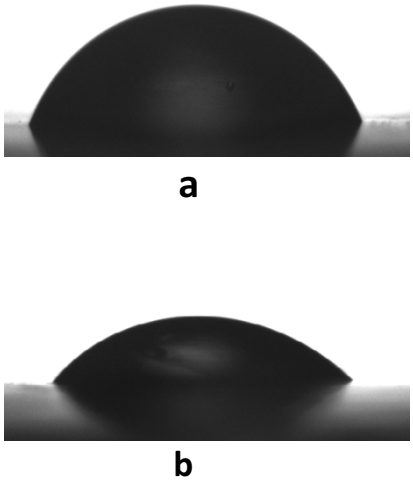


Fig. 3. Contact angles of pristine membrane (a), and modified membrane (b).

Table 1: Water content and contact angle for studied membranes.

Membrane	Water content%	Contact angle
pristine membrane	71.30 %	65.40
Modified membrane	73.6%	55.84

3.3. Anti-fouling properties of the membranes:

Static protein adsorption

Figure 4 shows that the antifouling performance against protein fouling on pristine and modified celluluce acetat memberane. As seen, the addition of Cymbopogon Proximus extract to cellulose acetate composite decreases the relative protein adsorption (RP%) from 74.9 to 10.8, and consequently, increases the resistivity of the membrane towards adsorptive fouling. This result can be discussed on the basis that the extract act as hydrophylicity modifier with more hydrophilic surface which are insusceptible to hydrophobic protein molecules. As known, , protein fouling is caused by adsorption of protein molecules on the membrane surface and the adsorptive interaction between foulants and the membrane surface is one of the most significant elements for membrane antifouling property.

The modified membranes surface contributed to the formation of hydration layers via ionic solvation of the charged groups and hydrogen bonds between the amide groups and the water molecules [33]. The hydration layers led to a strong repulsive force to protein at a specific distance, and made the protein in contact with the membrane surface in a reverse manner [34].

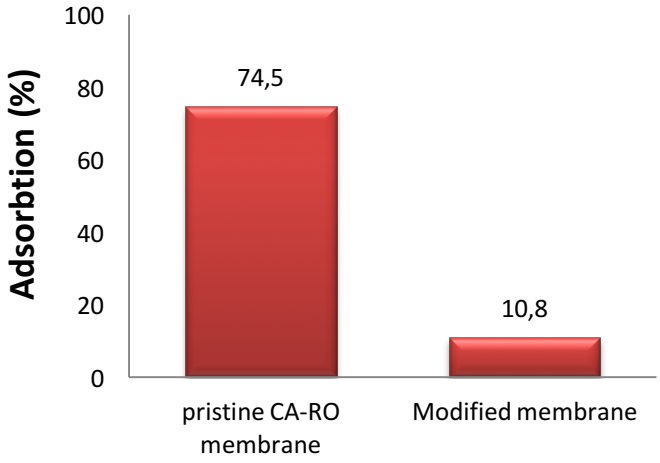
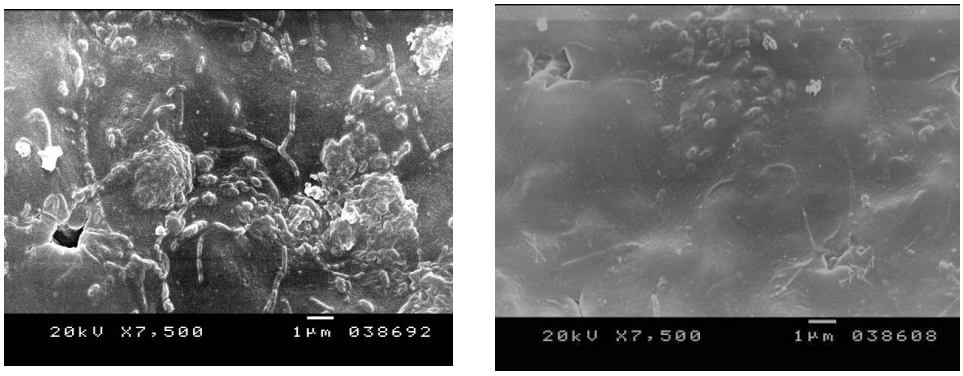


Fig. 4. Static adsorption of protein on pristine and modified membranes.

Fouling attachment on membrane:

In order to further identify the anti-fouling properties of pristine and modified cellulose acetate membrane, their surface morphology after immersion in seawater for 15 days was characterized with SEM. As shown in Fig. 5, the microbe's density on modified membrane was substantially lower than that of pristine one. For the membrane containing the extract, microbial attachment decreased on its surface, indicating that it has anti-adhesive properties. This phenomenon may be ascribed to the presence of functional groups of plant extract. It was reported that electrostatic interaction between the functional groups and cell membrane results in cell death [22].



a **b**

Fig.5. SEM photograph of pristine membrane (a)and modified membrane (b) after immersion in seawater for 15 d.

3.4. Effect of extract addition on membrane performance:

The relation between water flux, salt rejection and applied pressure was plotted during the desalination process and illustrated in figures 6 and 7. The results in the figures show that modified membrane with Cp extract additive has higher water flux values. Also, the salt rejection increased to 96.7% at 14 bar instead of 92.3% for the pristine membrane. Increasing the pressure over 16 par decreases the salt rejection. The decrease of salt rejection with the increase of applied pressure can be attributed to the corresponding high fluxes may lead to concentration polarization in the measuring cell, which causing a drop in the salt retention [23].

The increase in salt rejection and water flux values in presence of the additive can be attributed to the formation of mini pores and enlarged channels in the dense layer (as previously seen in section 3.1). The results well agree with hydrophylicity and surface morphology data which indicated that extract increases hydrophylicity of the membrane in addition to acting as pore former. It is reported that the change in permeability of the membranes may be attributed to two possible reasons [25]. One is the increase in hydrophilicity due to the addition of the plant extract, and or the other is the formation of a porous membrane structure.

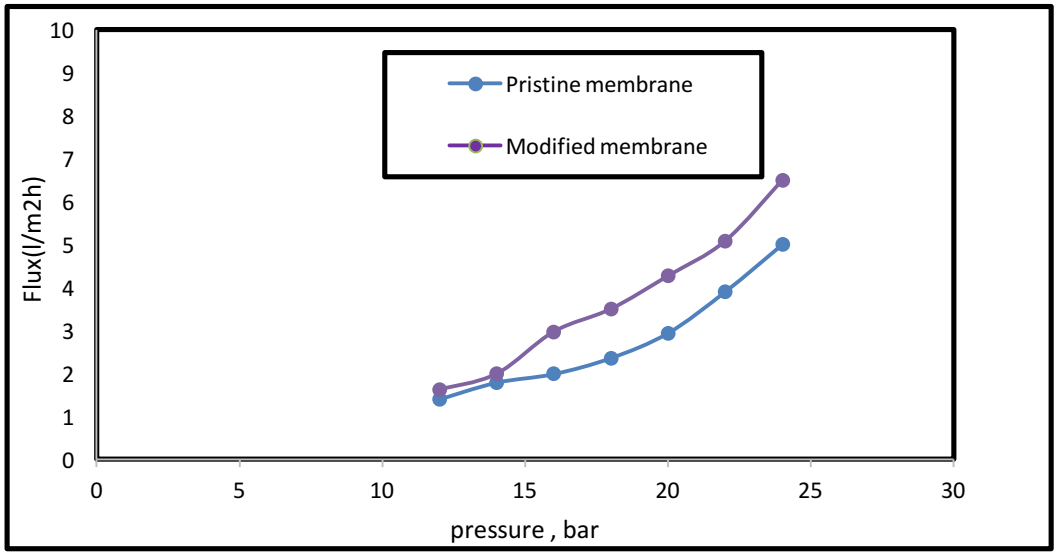


Fig. 6. Variation of permeate water flux of pristine and modified membranes versus feed pressure.

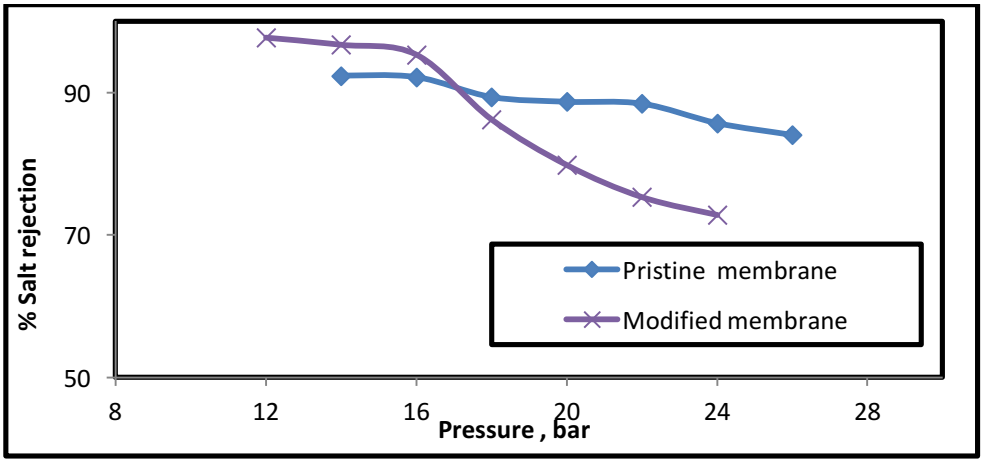


Fig 7: Variation of the percentage of salt rejection of pristine and modified membranes versus feed pressure.

Conclusion

In this work an improved, and relatively low-cost composite CA membrane was fabricated by a facile method using the phase inversion technique. This new composite contains *cymbopogon proximus* extract which is rich in many functionalized groups as proved in FTIR examinations. The SEM micrographs proved that this membrane has a unique morphology which shows mini-pores in the skin layer, these

mini-pores gradually enlarged from the top surface to the underside of the membrane where very large channels are apparent. Contact angle and water content measurements illustrated that addition of the extract to membrane composite enhanced its hydrophilicity, and its resistivity to protein adsorption and microbial growth. Furthermore, performance tests indicated that the modified membrane showed best performance where the salt rejection increased to 96.7% at 14 bar instead of 92.3% for pristine one with improved water flux properties.

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