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Adapting the performance and physico-chemical properties of PES nanofiltration membrane by using of magnesium oxide nanoparticles

Sayed Mohsen Hosseini†, Ehsan Bagheripour, and Mohsen Ansari

Department of Chemical Engineering, Faculty of Engineering, Arak University, Arak 38156-8-8349, Iran

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Abstract−A new mixed matrix Polyethersulfone (PES)-co-Magnesium oxide nanoparticles (MGO) nanocomposite nanofiltration membrane was prepared through phase inversion method by using polyvinylpyrrolidone (PVP) as pore former and N, N dimethylacetamide (DMAc) as solvent. The influence of MGO nanoparticles concentration in the membrane matrix on the separation performance and physico-chemical characteristics of prepared membrane was studied by scanning electron microscopy, surface analysis, porosity measurement, water contact angle, permeability flux, salt rejection, antifouling property, and tensile strength. SEM images exhibited situating of MGO nanoparticles on the top surface of mixed matrix prepared membranes. SEM analysis also showed formation of a dense nanoparticle layer on the surface of prepared membrane at high additive concentration. Surface analysis results that revealed membrane surface roughness was increased initially by addition of MGO and then was decreased. Measured porosity showed reduction behavior for all prepared membranes filled with MGO nanoparticles. The membrane surface hydrophilicity was enhanced 35% by incorporating MGO nanoparticles into the membrane matrix. Results showed that membrane permeation flux was improved 32% by utilizing of MgO nanoparticles into the membrane matrix. Salt rejection was also improved 49% by using MGO nanoparticles in the membrane matrix relatively. The modified membranes filled with different concentrations of MGO nanoparticles showed higher antifouling properties and tensile strength compared to the neat PES membrane.

Keywords: Nanofiltration, Magnesium Oxide Nanoparticles, Hydrophilicity, Separation Performance, Antifouling Behavior

INTRODUCTION

To improve the NF physico-chemical properties, recently extensive research has been done with various modification methods such as supplying different polymeric materials, blending of polymers, surface modifications and loading of different nanoparticles to obtain better membranes [1,2]. Utilizing of inorganic nanoparticles or fillers in polymeric membranes have been examined in many applications to enhance the thermal, chemical stability, mechanical properties and to improve the separation properties, performance and anti-fouling characteristic of polymeric membranes based on synergism between the nanoparticles and polymeric matrix properties [3-6]. Meanwhile, metal oxide nanoparticles were widely used as filler agents into the membrane matrix order to optimize membrane performance.

In the current study, the matrix modification of NF membrane with MGO nanoparticles was carried out to obtain efficient properties/performance, which can be useful in water and wastewater treatment. To prepare a modified NF membrane with enhanced of hydrophilicity/antifouling properties, polyethersulfone (PES), and magnesium oxide nanoparticles (MGO) were used with the below consideration.

One of the most important polymeric materials which is widely used for preparation of membranes is PES. The prepared membranes based on the PES have good mechanical property, thermal and outstanding oxidative stability. They have some flaws that need to be overcome, such as its hydrophobicity in nature resulting in easily fouling with reduction of permeate flux during filtration process. So, its modification and increase of its hydrophilicity can improve its antifouling properties [7]. MgO is an important inorganic material with a wide band-gap with large specific surface area [8-10]. MGO nanoparticles are safe materials to humans. They have been used in many applications, such as catalysis, catalyst supports, toxic waste remediation, refractory materials and adsorbents, additive in heavy fuel oils, reflecting and anti-reflecting coatings, superconducting and ferroelectric thin films as the substrate, super- conductors and lithium ion batteries [11-13]. Magnesium is IIA group element with atomic number 12 and oxygen is VIA group element with atomic number 8. The compound MgO has boiling and melting points as 3,600 °C and 2,852 °C [14].

To our knowledge, MGO nanoparticles have not been used for modification of PES based nanofiltration membranes. So, the main goal of the current research is to investigate MGO nanoparticle impacts with different concentrations on the performance and properties of PES based NF membrane.

In this research, the mixed matrix PES/MGO nanoparticles were prepared by solution casing technique. The effect of MGO nanoparticles concentration in the membrane matrix on the separation performance and physico-chemical characteristics of prepared membrane was studied by scanning electron microscopy, surface analy-

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†To whom correspondence should be addressed.
E-mail: S-Hosseini@araku.ac.ir, Sayedmohsen_Hosseini@yahoo.com

sis, porosity measurement, water contact angle, permeability flux, salt rejection, antifouling property, and tensile strength.

EXPERIMENTAL

1. Materials

The materials used in the current study were all analytical grade without further purification. Polyethersulfone (PES Ultrason E6020P with MW=58,000 g/mol) was supplied by BASF Company (Germany). N,N-Dimethyl acetamide and polyvinyl pyrrolidone (PVP) with 87.12 and 25,000 g/mol molecular weight were obtained from Merck, Germany. Magnesium oxide was obtained from US NANO Company with 20 nm average particle size (MW: 40.304). All other chemicals were supplied from Merck. Throughout the experiment, distilled water was used.

2. Preparation of the NF Membranes

The mixed matrix nanofiltration membranes were prepared by casting solution method. The preparation was followed by dissolving distinct amounts of PES (18 wt%), PVP (1 wt%) into the solvent (DMAC) in glassy reactors by a mechanical stirrer (VelpSientifica Multi 6 stirrer) for more than 4 h. This was followed by dispersion of MGO nanoparticles with different concentrations into the casting solutions. Casting solutions were then sonicated around 30 min for desired dispersion of nanoparticles. Solution was left at room temperature for a period of one day for the removal of dissolved air bubbles. Polymeric solutions were then cast into clean and dry glass plates at room temperature using a film applicator with a constant thickness (150 μm). Then, they were dipped immediately in deionized water (as non-solvent). The prepared membranes were kept in fresh deionized water for 24 h and then dried between two filter paper sheets at room temperature for one day before testing. The composition of different polymeric solutions is shown in Table 1.

3. Membrane Characterization

To investigate morphology and changes of membrane surface before and after MGO nanoparticles incorporation into the membrane matrix, scanning electron microscopy was applied (Seron Technology Inc. Korea). For this aim, the membranes were kept in the nitrogen liquid for 5 min and then were fractured. After sputtering the surface with a very thin gold film the SEM investigation was performed. For investigation roughness of prepared membranes, surface analysis was employed (FemtoScan method).

By using contact angle measuring instrument, contact angle (θ) of the prepared membranes was studied to analyze the surface hydrophilicity after MGO nanoparticles incorporation into the membranes. In this method, the probe liquid was deionized water. The contact angle was measured in three random locations in the ambient temperature and the average value was reported.

Table 1. Composition of casting solutions (% wt)

Membrane no	Solvent (DMAc) wt%	Nanoparticles (MgO) wt%
M (1)	81	0
M (2)	80.95	0.05
M (3)	80.9	0.1
M (4)	80.5	0.5
M (5)	80	1

[a]Constant concentration of PES and PVP (18 : 1 wt/wt%)

4. Porosity

Following equation was used for the overall porosity (ε) calculation [15,16]:

$$\varepsilon\,(\%)=\left(\frac{W_w-W_d}{\rho_f V_m}\right)\times 100 \tag{1}$$

where W_w, W_d, ρ_f and V_m are the wet and dry samples weight (g), water density (g/cm^3) and membrane small size volume (cm^3), respectively. All experiments were done three times and the reported results are mean values.

5. Filtration Tests

To investigate the performance of the prepared membranes, a dead-end filtration cell with effective filtration area of 11.94 cm^2 was used. Prepared membranes were first compacted with deionized water at 0.5 MPa for 20 min prior to filtration to reach a steady flux during filtration experiments, then operating pressure was reduced to 0.45 MPa for filtration test. The permeability flux j_v (L/m^2 h) was calculated using the following equation:

$$J_V=\frac{V}{A\Delta t} \tag{2}$$

where V is volume of permeated flux (L), A is membrane area (m^2) and Δt is sampling time (h).

Na_2SO_4 aqueous solution (0.01 mol/L) was used as feed solution for nanofiltration performance estimation. For salt rejection calculation, the following equation was used:

$$R\%=\left(\frac{C_f-C_p}{C_f}\right)\times 100 \tag{3}$$

where, C_p and C_f are salt concentration in permeate and feed, respectively, and were measured by conductivity meter (Ohaus Corporation, S/N B143385306, U.S.A.).

6. Antifouling Ability of the Membranes

Flux decreased ratio measuring through the continuous filtration experiment was used for determination of the antifouling properties of the prepared membranes. Following equation was applied to calculate flux decreased ratio [17]:

$$M\%=\frac{J_0-J_1}{J_0}\times 100 \tag{4}$$

where J_0 is the initial permeation flux, J_1 is the permeation flux after continuously filtrating for 60 min.

7. Mechanical Strength

ASTM1922-03 standard was applied to determine mechanical strength of the mixed matrix NF membranes [18,19]. The membranes were cut into standard shapes and the maximum tolerable load of membranes was measured.

RESULTS AND DISCUSSION

1. Morphology PES/MGO NF Membranes

The surface SEM images of prepared mixed matrix membranes filled with different concentrations of MGO nanoparticles are

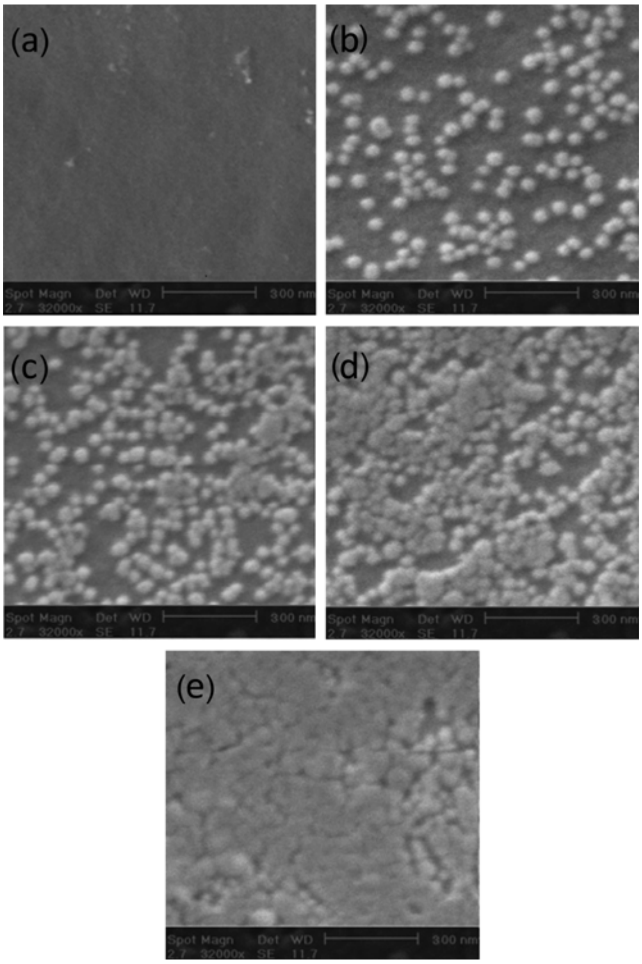

Fig. 1. Surface SEM images of prepared membranes with different concentration of MGO nanoparticles (a) M1, (b) M2, (c) M3, (d) M4, (e) M5.

shown in Fig. 1. As can be seen, the surface of pure PES membrane is clearly without any particles and it looks smooth. The surface of mixed matrix NF membranes filled with MGO is rife of nanoparticles placement and higher nanoparticles loading into the membrane surface was observed with increase of nanoparticles concentration into the casting solution. The presence of these particles in the membrane surface will lead to a rougher surface. In this condition a dense and hydrophilic surface will form, which can result in the lower foulants deposition on the membrane surface.

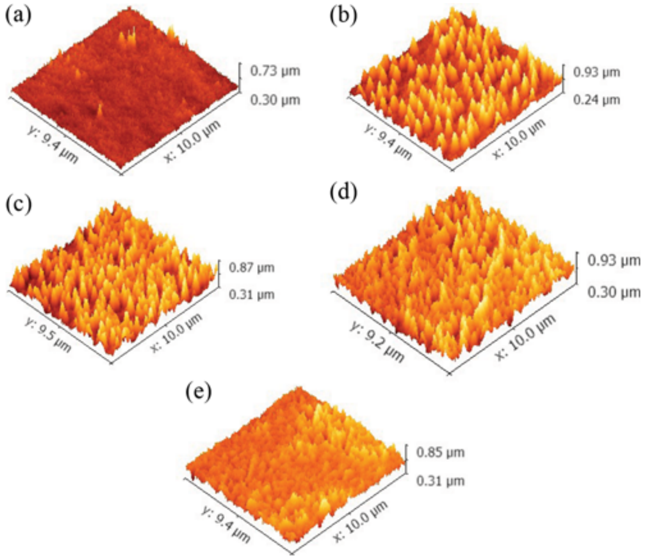

Fig. 2. The Surface analysis images of membranes (a) M1, (b) M2, (c) M3, (d) M4, (e) M5.

The presence of MGO nanoparticles in the membrane surface happens during the phase inversion process. The hydrophilic MGO nanoparticles migrate spontaneously to the membrane/water interface to decrease the interface energy [20].

As shown in Fig. 1, the content of circular MGO nanoparticles in the membrane top surface increased when concentration of MGO nanoparticles in PES matrix was increased and it covers uniformly PES surface in M5.

Surface analysis was also used to investigate the surface roughness of the PES nanofiltration membrane before and after modification process. The surface images and roughness parameters of prepared membranes are demonstrated in Fig. 2 and Table 2, respectively. According to the results, Ra (roughness average) is 14.4 for neat PES membrane without nanoparticles, which is the lowest value compared to PES/MGO mixed matrix membranes. The surface images (Fig. 2) show that the membrane surface is very smooth for neat PES (M1) and many peaks and valleys have appeared for M2 containing 0.05 wt% MGO nanoparticles. The Ra for M2 is 58.8 (Table 2), which is the highest compared prepared membranes. As seen in the SEM images (Fig. 1), migration of MGO nanoparticles to the surface during phase inversion process can form sharp peaks on the membrane surface, which should

Table 2. Surface roughness parameters of mixed matrix membranes filled with different concentrations of MGO nanoparticles

Ra (roughness average)	Rq (root mean square roughness)	Rt (maximum height of the roughness)	Rv (maximum roughness valley height)	Rp (maximum roughness peak height)
14.4	18.5	115.6	56.5	59.0
58.8	75.9	439.1	215.8	223.3
53.2	65.8	356.2	171.4	184.8
48.4	60.6	317.1	152.2	164.7
29	36.7	238.7	124.2	114.5

increase the roughness of membranes. As can be seen in the surface analysis images (Fig. 2), after increasing concentration of MGO nanoparticles into the PES matrix, membrane surface roughness was changed obviously and tended to a much smoother surface compared to M2. The Ra was decreased from 58.8 for M2 to 29 for M5 containing of 1 wt% nanoparticles. As mentioned in SEM images, in the higher MGO nanoparticle loading rate, the number of particles migrating to the surface increased (see SEM images Fig. 1(e) and a uniform thin/dense layer formed by nanoparticles on the PES membrane, leading to a smoother surface compared to M2. The results presented in Fig. 2 for roughness are very well consistent with SEM surface images. Changing of membrane roughness by various membrane modification techniques such as coating on membrane surface or mixed matrix modification with nanoparticles was also found by other researchers [21-24].

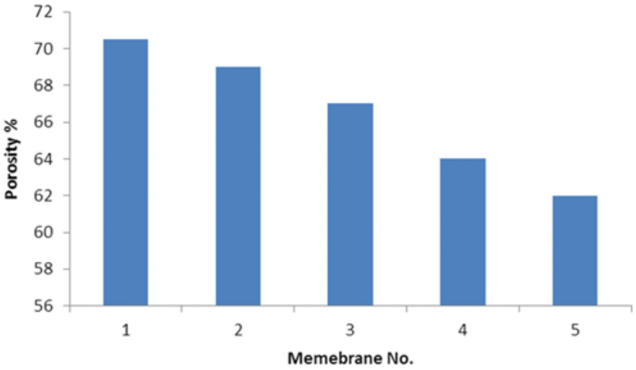

Fig. 3. The effect of nanoparticles concentration on overall porosity of the prepared membranes.

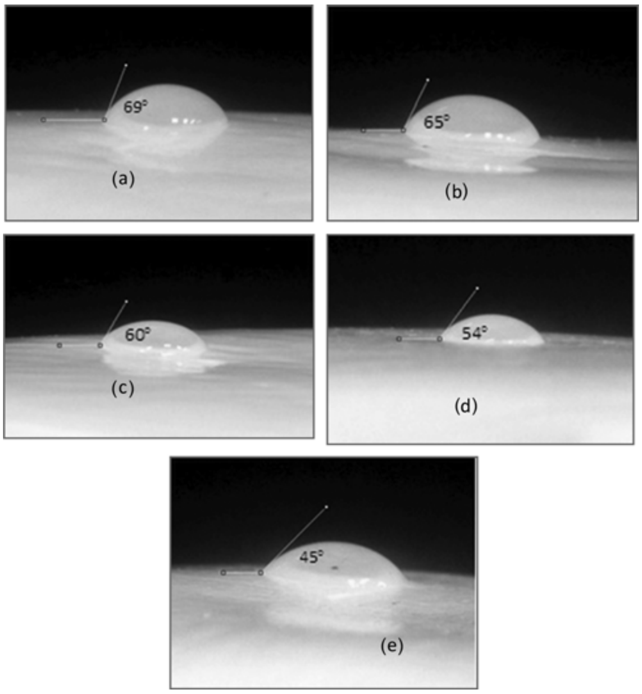

Fig. 4. The influence of MGO nanoparticles content ratio on contact angle.

Calculated membrane porosity is presented in Fig. 3. The results revealed that the highest porosity is for unfilled PES membrane compared to the filled ones. MGO nanoparticle migration and placement onto the membrane surface causes a significant reduction of surface pore size, which can result to the declined in porosity.

2. Contact Angle Characterization

To investigate membrane hydrophilicity, water contact angle measurement widely has been used. Generally, a higher hydrophilicity is related to the smaller contact angle. Higher surface hydrophilicity prevents foulant deposition due to the compact water layer on to the membrane surface, which can improve antifouling properties of the membrane during filtration [25]. The results of contact angle between water droplets and membrane surface are shown in Fig. 4.

The results revealed that contact angle significantly was decreased from 69° for unfilled PES membrane to 45° for membrane filled with 1 wt% MGO nanoparticles (M5).

The largest contact angle belonged to M1, which is related to the hydrophobic nature of PES. After incorporation of MGO nanoparticles into the membrane matrix, an improvement in membrane hydrophilicity was observed by slight reduction of contact angle. The migration and presence of MGO nanoparticles to the top surface of prepared membrane increases the membrane hydrophilicity in comparison of unfilled PES membrane. A similar result was reported in the published literature with iron-oxide nanoparticles [26].

3. Membrane Filtration Performance

The permeability flux of prepared mixed matrix membranes, which was measured at 0.45 MPa, is shown in Fig. 5 measured at the pressure of 0.1 MPa. Membrane hydrophilicity and membrane morphology are the main factors affecting permeability flux and rejection [27]. As demonstrated in Fig. 1(b), the presence of nanoparticles in the top surface of membrane significantly affected the surface structure compared to M1 (neat PES). The nanoparticle migration to the top surface during phase inversion, largely will decrease the surface pore size and porosity (see Fig. 3), which may lead to a decrease in the flux of the membrane. So, the flux in M2 was affected with pore blocking caused by MGO nanoparticles. The permeability flux increased from 4.5 ($L/m^2 \cdot h$) for M2 to 69.8 ($L/m^2 \cdot h$) for M4 filled with 0.5 wt% MGO nanoparticles, which is

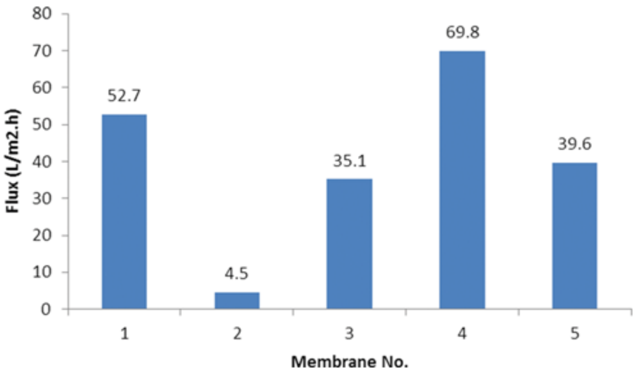

Fig. 5. Permeability flux of prepared mixed matrix membranes at various MGO concentration.

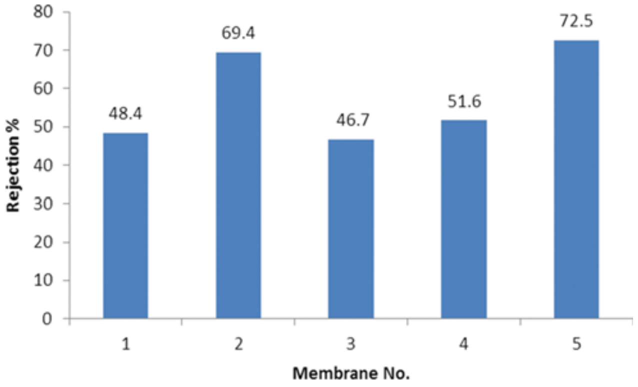

Fig. 6. Salt rejection performance for the prepared mixed matrix membranes.

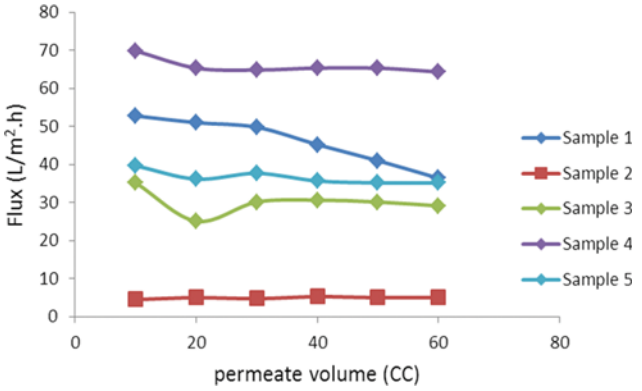

Fig. 7. Flux versus time for the prepared nanofiltration membranes with different MGO nanoparticles concentration.

14.5-times more than M2. The main factor for this flux improvement can be related to the synergetic effects between hydrophilic MGO nanoparticles placed in the top surface and water and improvement of the surface hydrophilicity. Therefore, M3 and M4 demonstrate better permeability flux compared with M2. After incorporation of 1 wt% MGO, again permeability flux was decreased from 69.8 ($L/m^2{\cdot}h$) for M4 to 39.6 ($L/m^2{\cdot}h$) for M5. In this condition, although contact angle was decreased for M5 and its surface has the highest hydrophilicity properties, but the cross linking force between much numbers of nanoparticles migrated to the top surface which led to formation of a very dense and uniform nanoparticle layer onto the PES surface. In this condition, blocking of surface pores can reach the highest value (see Fig. 1(e)), leading to the reduction. This result is consistent with the report in the literature for membrane surface modification [28]. The influence of MGO nanoparticle concentrations on salt rejection is shown in Fig. 6. The results reveal that rejection was improved initially from 48.4% for neat PES membrane to 69.4% for M2 containing 0.05 wt% MgO nanoparticles. As mentioned, pore blockage phenomenon, because of placement of nanoparticles in the top surface of membrane and reduction of membrane porosity, decreases the amount of water traffic through the membrane, which is associated with reduction in salt passage through the membrane matrix. This enhances the salt rejection. In addition, the results of contact angle measurements (Fig. 4) also confirmed higher hydrophilicity for M2 in comparison with net PES membrane. It's widely accepted that hydrophilic surface can cause to lower foulant deposition on membrane surface, which leads to better rejection [20].

The salt rejection was decreased again for M3 and M4, which may be due to rougher surface for these membranes compared to pristine membrane (M1). It is well known that a membrane with rough surface has more ability to trap the salt molecules on membrane surface. Moreover, establishment of concentrated stagnant layer on membrane surface due to increase of membrane roughness improves the possibility of slat percolation through the membrane matrix and decrease the salt rejection. [29]. The rejection was increased again from 51.6 to 71.5% by more increase of nanoparticles loading ratio up to 1 wt% in membrane matrix. This can be attributed to nanoparticle accommodation on membrane surface at high additive concentration, which forms a dense resistance layer onto the membrane surface (SEM images; Fig. 1(e)) and so increases the salt rejection.

The time dependency of the prepared NF membranes permeate flux is shown in Fig. 7. The results revealed that all mixed matrix membranes filled with MGO nanoparticles have more steady state flux during filtration process compared to unfilled membrane. Due to the membrane blocking with solutes deposition on the surface of membranes, the permeation flux decreases by increasing of filtration time [30].

To determine the antifouling performance of prepared membranes, the difference between initial flux and final flux was calculated as decreased flux ratio. The membrane fouling tendency is higher with higher value of decreased flux ratio [31]. The results of decreased flux ratio calculated with Eq. (4) are shown in Fig. 8. The results reveal that the decreased flux ratio of all mixed matrix membranes filled with various amounts of MGO nanoparticles is lower than that of unfilled PES one. In this condition, higher antifouling properties can be expected from prepared mixed matrix membranes. Thus, it can be concluded that the presence of MGO nanoparticles into the membrane structure and surface, due to the hydrophilicity improvement of membranes, improved antifouling performance of PES membrane.

4. Membrane Tensile Strength

The influence of MGO nanoparticles concentration on tensile

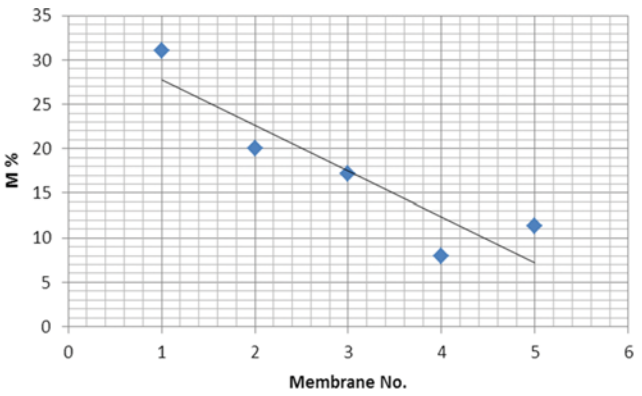

Fig. 8. The effect of MGO nanoparticles on the decreased flux ratio (Anti fouling ability).

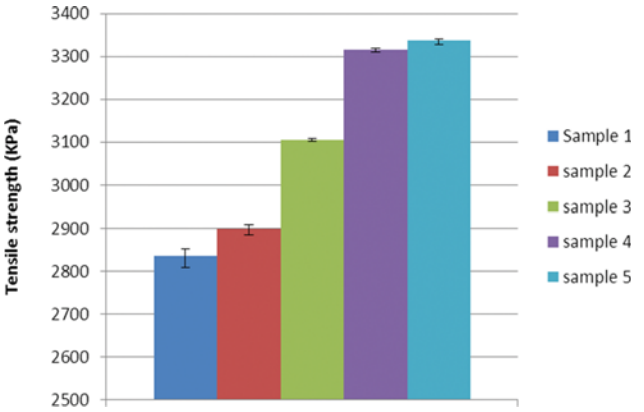

Fig. 9. The effect of MGO nanoparticles content ratio on membranes' tensile strength.

strength of PES membrane is shown in Fig. 9. Obtained results (Fig. 9) reveal that tensile strength was increased slightly by increase of MGO nanoparticle concentration loaded into the PES. Formation of strong interfacial bonding between polymer and nanoparticles can lead to the improvement of the mechanical strength [32,33].

The results indicated that incorporation of MGO nanoparticles into the PES matrix can act as a physical cross linking agent in membrane structure to link the polymer chain leading to the increase of rigidity. In this condition, higher load stress will be tolerated with the membranes filled by higher loading rates of MGO nanoparticles.

CONCLUSION

PES nanofiltration membrane was filled with different concentrations of MGO nanoparticles. Surface SEM images of mixed matrix membranes clearly showed migration of MGO nanoparticles to the top surface during phase inversion process. The surface analysis images indicated initially an increase of membrane surface roughness for membrane filled with 0.05 wt% nanoparticles, and again formation of a smoother surface for higher nanoparticles loading rates. Addition of MGO nanoparticles into the casting solution led to the lower porosity in all membranes. Membranes contact angle was decreased from 69° for unfilled PES membrane to 45° for M5, which confirmed significantly improved membrane hydrophilicity. Permeability flux was reduced initially for M2 and then was increased for M3 and M4. It was decreased again in M5 related to formation a very dense nanoparticles layer in top surface. Rejection results showed an improvement in M2 and M5. Rejection was decreased for M3 and M4 due to the rougher surface compared to M1. The mixed matrix membranes filled with different concentrations of MGO revealed higher antifouling ability compared to PES membrane. Additionally, tensile strength was improved straightly by different concentrations of MGO nanoparticles.

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