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High efficient removal of lead(II) and nickel(II) from aqueous solution by novel polysulfone/Fe_3O_4–talc nanocomposite mixed matrix membrane

Pourya Moradihamedani[a,b], Katayoon Kalantari[c,d], Abdul Halim Abdullah[a,b,*], Noor Azian Morad[c,d]

[a]*Materials Science and Characterization Laboratory, Institute of Advanced Technology, University Putra Malaysia, 43400 Serdang, Selangor, Malaysia, Tel. +60 1125588994; emails: pouryamoradi999@gmail.com (P. Moradihamedani), halim@upm.edu.my (A.H. Abdullah)*
[b]*Faculty of Science, Chemistry Department, University Putra Malaysia, 43400 Serdang, Selangor, Malaysia*
[c]*Shizen Conversion & Separation Technology (SHIZEN), Universiti Teknologi Malaysia, Jalan Sultan Yahya Ahmad Petra, 54100 Kuala Lumpur, Malaysia, emails: ka_upm@yahoo.com (K. Kalantari), azian.morad@gmail.com (N.A. Morad)*
[d]*Malaysia–Japan International Institute of Technology (MJIIT), Universiti Teknologi Malaysia, Jalan Sultan Yahya Ahmad Petra, Kuala Lumpur, Malaysia*

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ABSTRACT

Novel mixed matrix membranes were prepared from polysulfone (PSf), talc and Fe_3O_4–talc nanocomposite by phase inversion technique. PSf/Fe_3O_4–talc membranes showed higher lead and nickel rejections compared to PSf/talc membranes, which reaches to 99.4 and 96.2% for lead and nickel ions, respectively, at feed pH 5 and lower rejections at pH 3.5 and 2. Higher surface area in Fe_3O_4–talc nanocomposite than talc could be considered as a main reason of higher heavy metals removal in PSf/Fe_3O_4–talc membranes. The heavy metals removal enhances with the increase in nanocomposite content from 7 to 9 wt.% because higher numbers of vacant sites in membrane morphology were available for adsorption. Heavy metals rejection reduces in higher nanocomposite concentrations (11 and 13 wt.%) due to the formation of macrovoids in membrane substructure. In addition, a reduction in the metal ion rejection was recognized by enhancement in feed solution concentration and applied pressure. This is caused by difficulty for the remaining vacant sites to be filled with the heavy metal ions because of repulsive forces between the adsorbed solute molecules on the surface and solute in bulk phase. The interactions of lead and nickel ions with nanocomposite placed on the membrane top layer were revealed by the scanning electron microscopy with energy dispersive X-rays.

Keywords: Polysulfone; Nanocomposite; Mixed matrix membranes; Heavy metals removal

1. Introduction

Heavy metals are elements having atomic weights between 63.5 and 200.6, and a particular gravity more than 5.0. With the fast advancement of commercial ventures, for example, mining operations, metal plating facilities, tanneries, fertilizer industries, paper industries, batteries and pesticides, heavy metals wastewaters are directly or indirectly released into the environment increasingly, especially in developing countries. Unlike organic contaminants, heavy metals

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*Corresponding author.

are not biodegradable and have a tendency to accumulate in living organisms and many heavy metal ions are known to be toxic or carcinogenic. Toxic heavy metals such as zinc, copper, nickel, mercury, cadmium, lead, and chromium are the particular concern in the treatment of industrial and municipal wastewaters [1]. Among these hazardous materials, nickel surpassing its critical level might bring about serious kidney and lung sickness beside pulmonary fibrosis, gastrointestinal distress, and skin dermatitis and it is recognized that nickel is human cancer-causing agent [2]. Lead can cause central nervous system damage. Lead can also harm the liver, kidney and reproductive system, essential cellular procedures, and brain capacities. The toxic symptoms are insomnia, anemia, dizziness, headache, irritability, muscles weakness [3]. Diverse treatment methods have been developed for heavy metal removal from aqueous solutions, for example, oxidation, reduction, precipitation, ion exchange, membrane filtration, and adsorption. Among these methods, the membrane separation technique and adsorption are the most favorable process, economically and technically, for removing heavy metals from aqueous solutions [4,5]. The technologies of membrane processes used to remove metals from the wastewater are ultrafiltration (UF), reverse osmosis (RO), nanofiltration (NF) and electrodialysis. UF is a membrane technique working at low transmembrane pressures for the removal of dissolved and colloidal material [5].

Recently, Fe_3O_4 nanoparticles were shown to be highly efficient materials for heavy metal ion removal by adsorption. Metal ion adsorption by magnetite was demonstrated through a combination of electrostatic attraction and ligand exchange [6–8]. However, there are some challenges that Fe_3O_4 nanoparticles present. The first one is that Fe_3O_4 nanoparticles oxidize and dissolve easily. In addition, the recycling process is difficult due to the small size of nanoparticles. Finally, the nanoparticles tend to co-aggregate and thus their effective surface area decreases and it causes their reaction activity. Several techniques have been developed to minimize the co-aggregation of the Fe_3O_4 nanoparticles and improve their manipulation, for example using polymers and clays [9]. Moreover, clay soils are widely used as adsorbents that isolate hazardous and other waste materials from surrounding environments. There are few studies on the adsorption characteristics of heavy metal ions in talc. Talc is known by the chemical formula $Mg_3Si_4O_{10}(OH)_2$. It consists of a magnesium hydroxide layer ($MgO{\cdot}H_2O$) sandwiched between two silicate layers (SiO_2), forming a three-layer structure [10]. Talc is commonly used as a filler, coating, and dusting agent in paints, lubricants, plastics, cosmetics, pharmaceuticals, and ceramics manufacture [11].

In this work, a novel combination of membrane filtration and adsorption techniques was applied by preparation of PSf/talc and PSf/Fe_3O_4–talc nanocomposite mixed matrix membranes to remove Ni(II) and Pb(II) from aqueous solution efficiently. Accordingly, in order to find an optimum concentration of additives, mixed matrix membranes containing 7, 9, 11, and 13 wt.% of talc and Fe_3O_4–talc were prepared. Moreover, the effect of initial concentration of heavy metals in feed solution as well as pH of feed solution on the performance of prepared mixed matrix membranes were investigated.

2. Experimental

2.1. Materials and instrumentation

PSf ($M_w = 22.000$ g/mol) from Sigma–Aldrich Co. (USA) was selected as a membrane material because of its superior selectivity, high mechanical strength, and notable thermal resistance. N-methyl-2-pyrrolidone (NMP) was used as a solvent. In order to adjust the pH environment of the bulk solution into acidic or basic condition, 1 M hydrochloric acid (HCl) and sodium hydroxide (NaOH) were added. Ferric chloride hexahydrate ($FeCl_3{\cdot}6H_2O$) and ferrous chloride tetrahydrate ($FeCl_2{\cdot}4H_2O$) with purity of 96% were used as the iron precursor and talc powder (10 μ, $3MgO\ 4SiO_2{\cdot}H_2O$) was obtained from Sigma–Aldrich (St Louis, MO, USA). NaOH with purity of 99% was obtained from Merck KGaA (Darmstadt, Germany). Deionized water was used for all experiments.

2.2. Synthesis of Fe_3O_4–talc nanocomposites

In order to prepare Fe_3O_4–talc nanocomposites, 2.0 g of talc was suspended in 70 mL deionized water, and then the molar ratio in $FeCl_3$ solution was adjusted to 2:1 by adding 2.07 g of Fe^{3+} and 0.75 g of Fe^{2+}. The ion solution suspended with talc composites was stirred for 1 hour for impregnation by the external surface of talc layers to prepare talc/Fe^{3+}–Fe^{2+} composites. The 15 mL freshly prepared NaOH (2.0 M) was added to talc/Fe^{3+}–Fe^{2+} composites suspension under continuous stirring. The suspensions were finally centrifuged, washed twice with ethanol and double-distilled water and kept in a stove at 100°C for 2 h. All the experiments were conducted at an ambient temperature and under a non-oxidizing (oxygen-free) environment through the flow of nitrogen gas. A schematic representation of Fe_3O_4–talc nanocomposite is shown in Fig. 1.

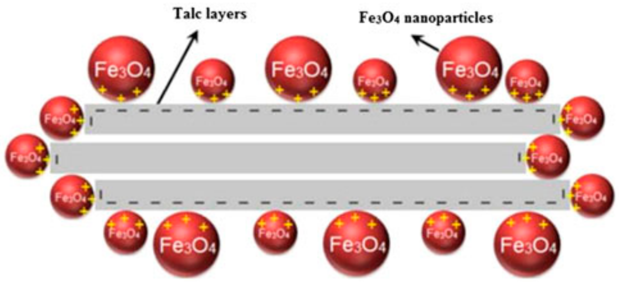

Fig. 1. Schematic representation of Fe_3O_4-talc nanocomposite.

Fig. 2 demonstrates the powder X-ray diffraction of talc and Fe_3O_4–talc. As shown in Fig 2(b), the PXRD peaks in the wide-angle range of ($2\theta = 30–80^\circ$) ascertained that the peaks at 30.35˚, 35.47˚, 43.29˚, 59.30˚, 62.10˚, and 74.11˚ related to the 2 2 0, 3 1 1, 4 0 0, 5 1 1, 4 4 0, and 6 2 2 crystalline structures of the Fe_3O_4 cubic nanocrystal with a spinel structure (Ref. Code Fe_3O_4: 01-088-0315). It is obvious that the formation of Fe_3O_4 nanoparticles led to increase in peaks' intensity [12,13].

Specific surface area of the talc powder and Fe_3O_4–talc nanocomposite were determined by the Brunauer–Emmett–Teller (BET) method (acquisition and reduction). The BET isotherm is the basis for determining the extent of nitrogen adsorption on a given surface. A Quantachrom AS1Win 2008 (Quantachrome Instruments, Boynton Beach, FL, USA) was used in this work for measuring the surface area of samples. The systematic sorption and desorption of nitrogen provided the fundamental information on the surface characteristics. The surface areas of talc powder and Fe_3O_4/talc nanocomposite were found to be 6.675 and 37.079 m^2/g, respectively.

2.3. Mixed matrix membrane preparation

Polysulfone/talc (PT) and Polysulfone/Fe_3O_4–talc (PFT) mixed matrix membranes were prepared by wet phase inversion technique. Homogeneous solution of the PSf dissolved in NMP was prepared in the presence of four different concentrations of talc and Fe_3O_4–talc nanocomposite (7, 9, 11, and 13 wt.%) by stirring with 200 rpm for 24 h at room temperature. The mass ratio of PSf to total solution was 18 wt.%. The solution was sprinkled and cast on a glass plate by a film applicator with 80 μm thicknesses. The wet film was immersed into the coagulation water bath for 24 h at room temperature. At the final stage, the membranes were dried at room temperature for a day. The composition of prepared membranes and their designation is listed in Table 1.

2.4. Removal of heavy metal ions

Heavy metals removal was carried out with 50, 70, and 90 mg/L aqueous solution of Pb(II) and Ni(II) at solution pH of 5.0 (initial pH), 3.5, and 2. All experiments were carried out twice at room temperature (25 ± 2℃) in a batch type and dead-end UF cell (Merck) with an effective membrane filtration area of 13.8 cm^2. Fig. 3 shows the schematic diagram of the dead-end UF system used in this experiment [14].

The performance of the membrane in the removal process was evaluated based on the flux measurement and the rejection percentage. The flux of feed solution was measured at different feed pressures of 1–3 bar. The following equation was used for calculation of the feed flux:

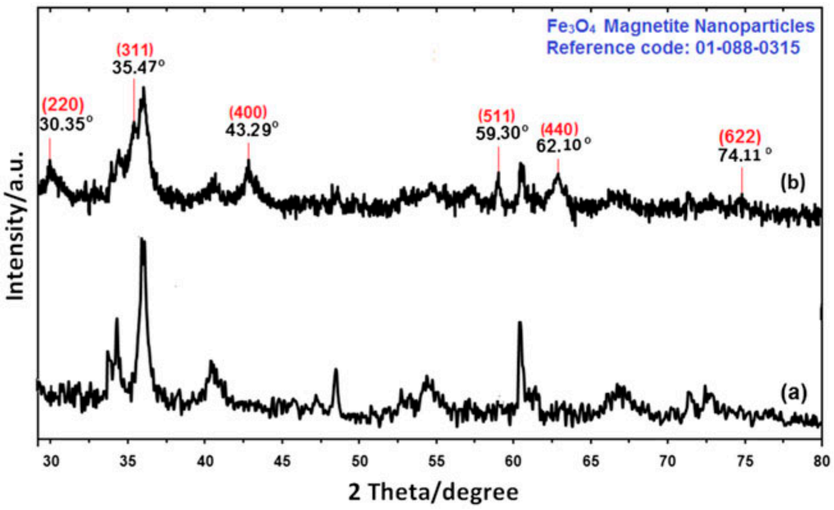

Fig. 2. Powder X-ray diffraction patterns of talc (a) and Fe_3O_4-talc (b) for determination of Fe_3O_4 crystal structure.

Table 1
Preparation conditions of mixed matrix membranes

Casting solutions (wt.%)				
Membrane	PSf	NMP	Talc	Fe_3O_4–talc
Neat PSf	18	82	0	0
PT7	18	75	7	0
PT9	18	73	9	0
PT11	18	71	11	0
PT13	18	69	13	0
PFT7	18	75	0	7
PFT9	18	73	0	9
PFT11	18	71	0	11
PFT13	18	69	0	13

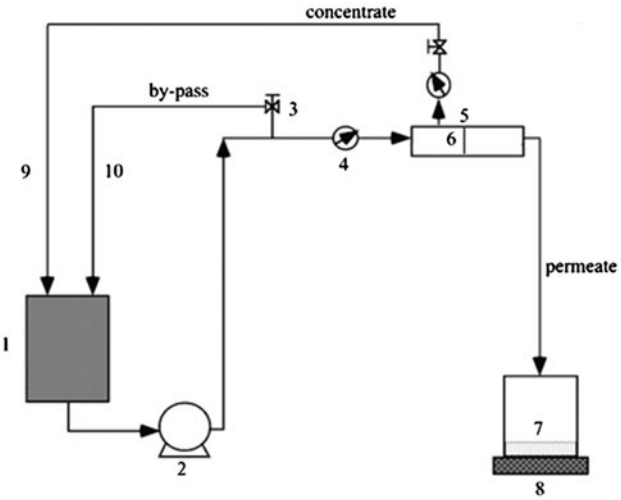

Fig. 3. Schematic diagram of the dead-end ultrafiltration system for heavy metals removal from water.
Notes: (1) feed tank, (2) pump, (3) valve, (4) pressure gauge, (5) dead end cell, (6) membrane, (7) permeate, (8) balance, (9) concentrate, (10) bypass.

$$J_w = \frac{Q}{A \cdot \Delta t} \tag{1}$$

where J_w is the flux (L/m^2 h), Q is the amount of collected permeate (L) and Δt is the sampling time (h).

The metal ion rejection of prepared membranes was calculated using the following equation:

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100 \tag{2}$$

where C_p and C_f are the heavy metal concentrations in permeate and the feed, respectively [14].

2.5. Characterization of the membrane

The surface functional groups of the neat PSf and the PSf/talc-Fe_3O_4 membranes were analyzed by Fourier transform infrared spectroscopy (Series100 PerkinElmer FT-IR 1650) in the scanning range of 280–1,380 cm^{-1} while the morphology of the membranes were observed using the scanning electron microscope (Philips XL-30).

The membrane water content was measured by soaking the membrane in water for 24 h and weighing it after wiping off with tissue paper. The membranes were dried in oven, at 40°C for 24 h and weighted. Based on wet and dry weights of the membranes, the water content was calculated using the following equation:

$$\%\ \text{Water content} = \frac{W_w - W_d}{W_w} \times 100 \tag{3}$$

where W_w and W_d are the weights of wet and dry membranes, respectively.

The porosity of prepared membranes was calculated using the following formula:

$$\%\ \text{Porosity} = \frac{W_w - W_d}{dAL} \times 100 \tag{4}$$

where W_w is the weight of wet membrane, W_d is the weight of dry membrane, d is density of water (g/cm^3), A is the membrane effective area (cm^2), and L is the thickness of membrane (cm) [14]. The contact angle of membranes was measured using FTA-200 dynamic contact angle analyzer according to the sessile droplet method. In order to minimize the experimental error, the average of the three different locations is reported.

3. Results and discussion

The permeability of the prepared membranes at different feed pressures (1–3 bar) was evaluated in term of flux. The variation in permeate flux passing through the membranes with different talc and Fe_3O_4–talc nanocomposite loadings is shown in Fig. 4. Neat PSf membrane did not have any permeability even until 3 bar feed pressure. The impermeability can be due to the formation of thick and dense top layer of the membrane. The membrane permeability increased with the addition of talc and Fe_3O_4–talc to the casting solution and reached a maximum of 25 L m^{-2} h^{-1} (Pb solution) and 29.5 L m^{-2} h^{-1} (Ni solution) for PFT13 at feed pressure of 3 bar. The results presented that PSf/Fe_3O_4–talc

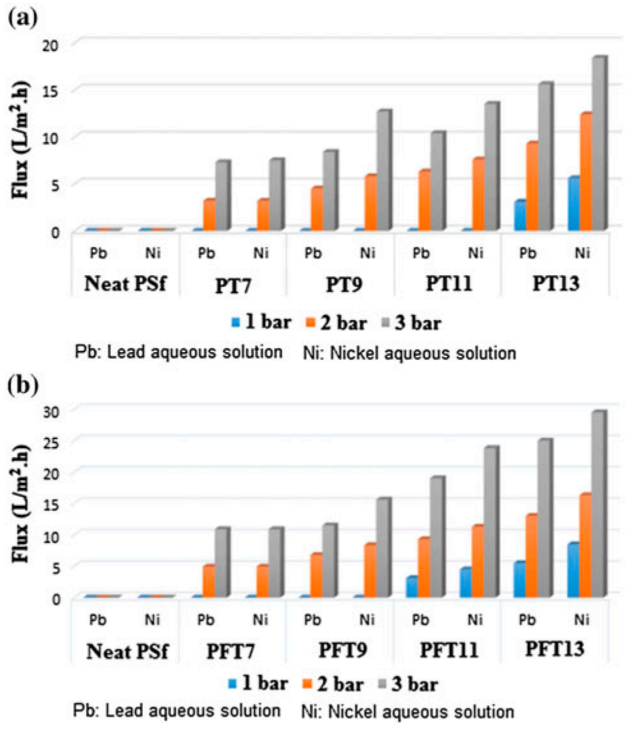

Fig. 4. The flux recorded for (a) PSf/talc and (b) PSf/Fe_3O_4–talc membranes containing different talc and Fe_3O_4–talc concentrations at different feed pressures.

membranes had higher permeability than PSf/Talc membranes. This could be as a result of the presence of Fe_3O_4 nanoparticles in casting solutions that produces membrane with higher porosity.

The performance of prepared mixed matrix membranes for heavy metals removal at different feed pressure and pH is shown in Table 2. The results disclosed that the PSf/Fe_3O_4–talc membranes exhibited higher rejection of heavy metals than PSf/talc membranes. This could be explained by higher surface area of Fe_3O_4–talc nanocomposites compared to pristine talc [15]. When talc sheets are broken, two different layers were produced. One layer, resulting from cleavage of the layer, is known as “faces.” It comprises completely compensated oxygen atoms, possesses very low electrical charge, and is nonpolar in water and hydrophobic. Another layer, arising from the breaking of the ionic bonds within the layers, is named “edges.” It comprises hydroxyl ions, siloxane groups (–Si–O–Si–), oxygen, and magnesium ions that can undergo hydrolysis. This layer has a comparatively high electrical charge and the presence of functional hydroxyl groups such as –SiOH, and –MgOH causes hydrophilic effect [16]. The negatively charged hydrophilic sites of talc particles are $\equiv SiO^-$. Due to electrostatic attraction between positively charged metal ions (Pb(II) and Ni(II)) and negatively charged $\equiv SiO^-$, talc is capable of adsorbing the heavy metal ions with strong affinity.

A reduction in rejection of lead and nickel ions was observed as the concentration of the talc and Fe_3O_4–talc nanocomposite increased beyond 9 wt.%. High content of nanocomposite in casting solution can increase the hydrophilicity of prepared membrane. Hence, instantaneous phase inversion between solvent (NMP) and non-solvent (water) produce porous membranes with macrovoids in the structure. Consequently, more metal ions were able to pass through the membranes.

The effect of pH of the feed solution on the heavy metals removal was examined. The variations in metal ions removal at feed pH 5 (initial pH), 3.5 and 2 are indicated in Table 2. Since the zeta potential of polysulfone membranes is near zero at pH 3, the surface of the membrane is positively charged at pH < 3 and

Table 2
Rejection of heavy metals in different talc and Fe_3O_4–talc concentrations and feed solution pH

	Pb(II) rejection %						Ni(II) rejection %					
	(pH 5.0)		(pH 3.5)		(pH 2)		(pH 5.0)		(pH 3.5)		(pH 2.0)	
Membrane	2 bar	3 bar	2 bar	3 bar	2 bar	3 bar	2 bar	3 bar	2 bar	3 bar	2 bar	3 bar
PT7	88.5	86.6	84.0	82.5	79.3	77.0	85.0	83.2	82.7	80.0	82.5	80.0
PT9	90.0	89.4	85.0	83.0	80.6	78.2	86.1	85.5	83.0	81.9	83.0	81.0
PT11	86.3	83.8	84.1	81.2	76.0	74.0	83.0	82.2	80.0	79.0	80.0	78.3
PT13	83.3	80.0	81.9	78.8	73.7	72.0	81.0	78.3	78.4	76.6	78.0	75.0
PFT7	99.0	98.5	95.4	90.0	91.3	88.5	95.7	93.6	91.5	87.3	89.4	85.4
PFT9	99.4	99.0	96.5	93.8	93.0	90.3	96.2	94.7	93.0	90.7	91.3	86.6
PFT11	97.2	97.0	93.0	88.2	88.4	83.6	85.3	82.5	83.5	80.6	82.2	78.3
PFT13	95.4	95.0	88.6	85.3	85.7	80.0	83.1	80.0	81.0	77. 3	79.7	75.1

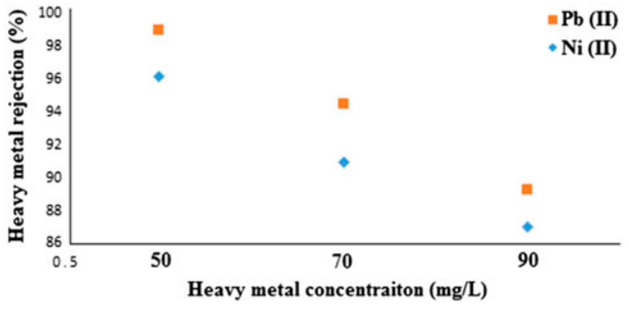

Fig. 5. Effect of metal ions concentration on the membrane performance.

negatively charged at pH > 3 [17,18]. Accordingly, repulsion force between positively charged membrane top layer and heavy metal ions at pH 2 caused a reduction in lead and nickel ions removal by prepared mixed matrix membrane.

From another point of view, the Fe_3O_4 nanoparticles may also react with the added HCl at feed pH < 3.5 and converted to $FeCl_2$ and $FeCl_3$ (Eq. (5)):

$$Fe_3O_4 + 8HCl \rightarrow FeCl_2 + 2FeCl_3 + 4H_2O \quad (5)$$

Subsequently, the surface area of Fe_3O_4-talc nanocomposite was decreased leading to a reduction in the heavy metal ions removal at lower pH.

As shown in Table 2, the removal efficiency of Pb (II) is higher than that of Ni(II) ions. The difference could be justified through the differences in the radius of the ions. Since the radius of Ni(II) (0.69 Å) is significantly smaller than Pb(II) (1.32 Å), nickel ions are easier to hydrate, therefore producing a larger layer of water on the surface. As a result, Ni(II) ions are more mobile in bulk solution and would have a lesser tendency to interact with nanocomposites [19].

Since PFT9 membrane was shown the highest rejection for both heavy metal ions, it was chosen to investigate the effect of metal ions concentration on the rejection performance of the membrane. Fig. 5 shows that metal ions removal decreased at higher concentrations of heavy metals in feed solution (70 and 90 mg/L). This phenomenon could be related to the difficulty of remaining vacant sites to be filled with the heavy metal ions because of repulsive forces between the adsorbed solute molecules on the surface and solute in bulk phase.

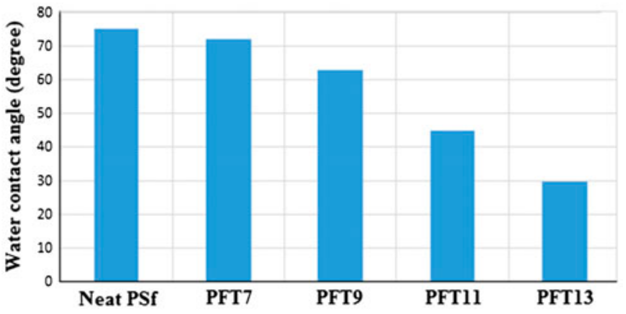

Fig. 7. Water contact angle of prepared membranes with different Fe_3O_4–talc concentrations.

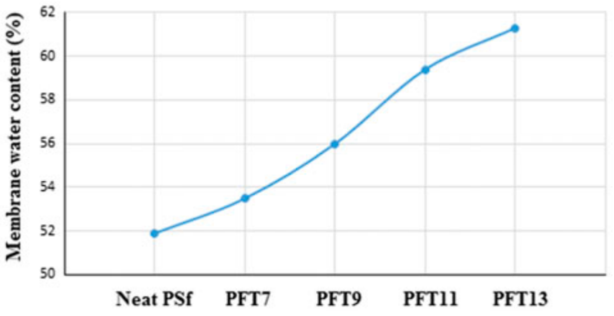

Fig. 8. Membrane water content in different Fe_3O_4–talc concentrations.

Fourier transform infrared (FTIR) was used to investigate the immobilization of Fe_3O_4–talc in PSf networks. Fig. 6 shows the spectra of PSf and PSf/Fe_3O_4–talc mixed matrix membranes in the range of 280–1880 cm^{-1}. A sharp peak at 1,240 cm^{-1} and two peaks at the range 1,300–1,350 cm^{-1} were

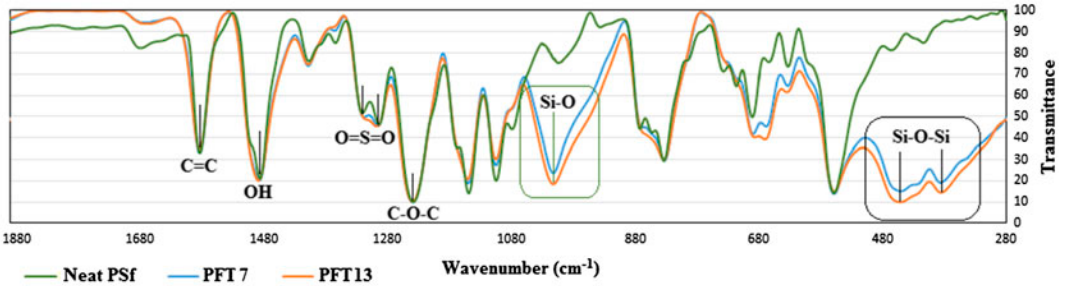

Fig. 6. FTIR spectrum of neat PSf, PFT7, and PFT13 membranes.

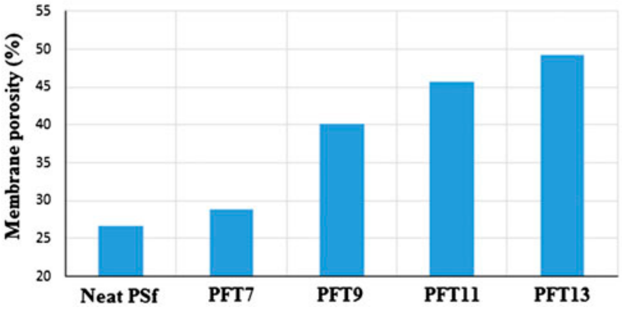

Fig. 9. Variation in membrane porosity with different Fe_3O_4–talc contents.

assigned to C–O–C group and the symmetric O=S=O stretching vibration in PSf [20,21]. High intensity peaks at 1,490 and 1,590 cm^{-1} were assigned to vibration of OH and C=C conjugation system of the benzene ring in PSf [22,23]. There are new peaks at 390 and 450 cm^{-1} in the PFT7 and PFT13 membranes spectra that could be assigned to the symmetric stretching vibration of O–Si–O group in talc. Strong bands at approximately 1,000 cm^{-1} were associated with stretching of Si–OH in talc [12]. These new peaks found in PFT7 and PFT13 spectrum indicate the presence of Fe_3O_4–talc nanocomposite in prepared mixed matrix membranes.

The hydrophilicity of the top layer of neat PSf and PSf/Fe_3O_4–talc mixed matrix membranes was examined based on water contact angle measurement. The smaller contact angle shows increase in the hydrophilicity of membrane surface. The water contact angle of neat PSf membrane was around 75.0°. As illustrated in Fig. 7, the water contact angle of the membrane decreased with the increase in the Fe_3O_4–talc concentration. Accordingly, PFT13 membrane had the lowest water contact angle. Hence, inclusion of Fe_3O_4–talc nanocomposite increased the hydrophilicity of the mixed matrix membranes thereby giving smaller contact angle.

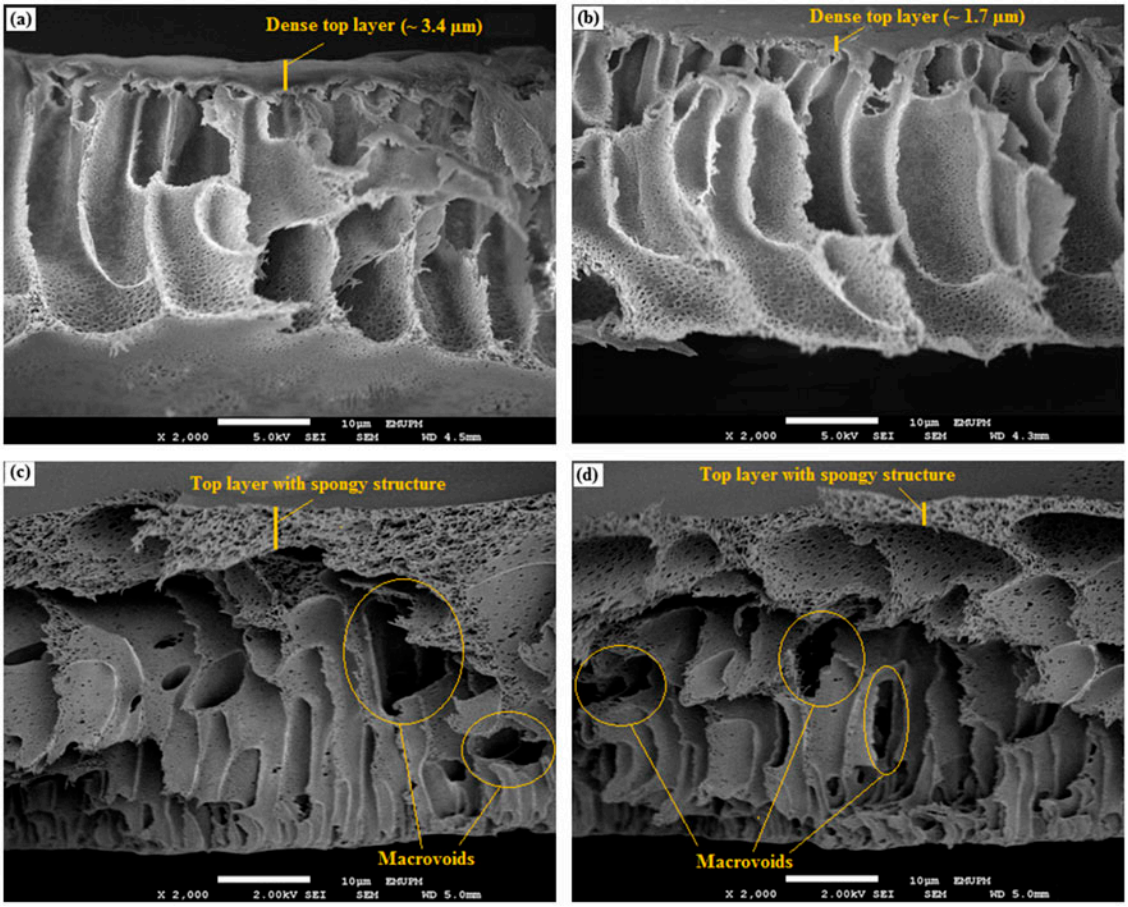

Fig. 10. SEM micrograph of membranes with different Fe_3O_4–talc contents; (a) Neat PSf, (b) PFT7, (c) PFT11, and (d) PFT13.

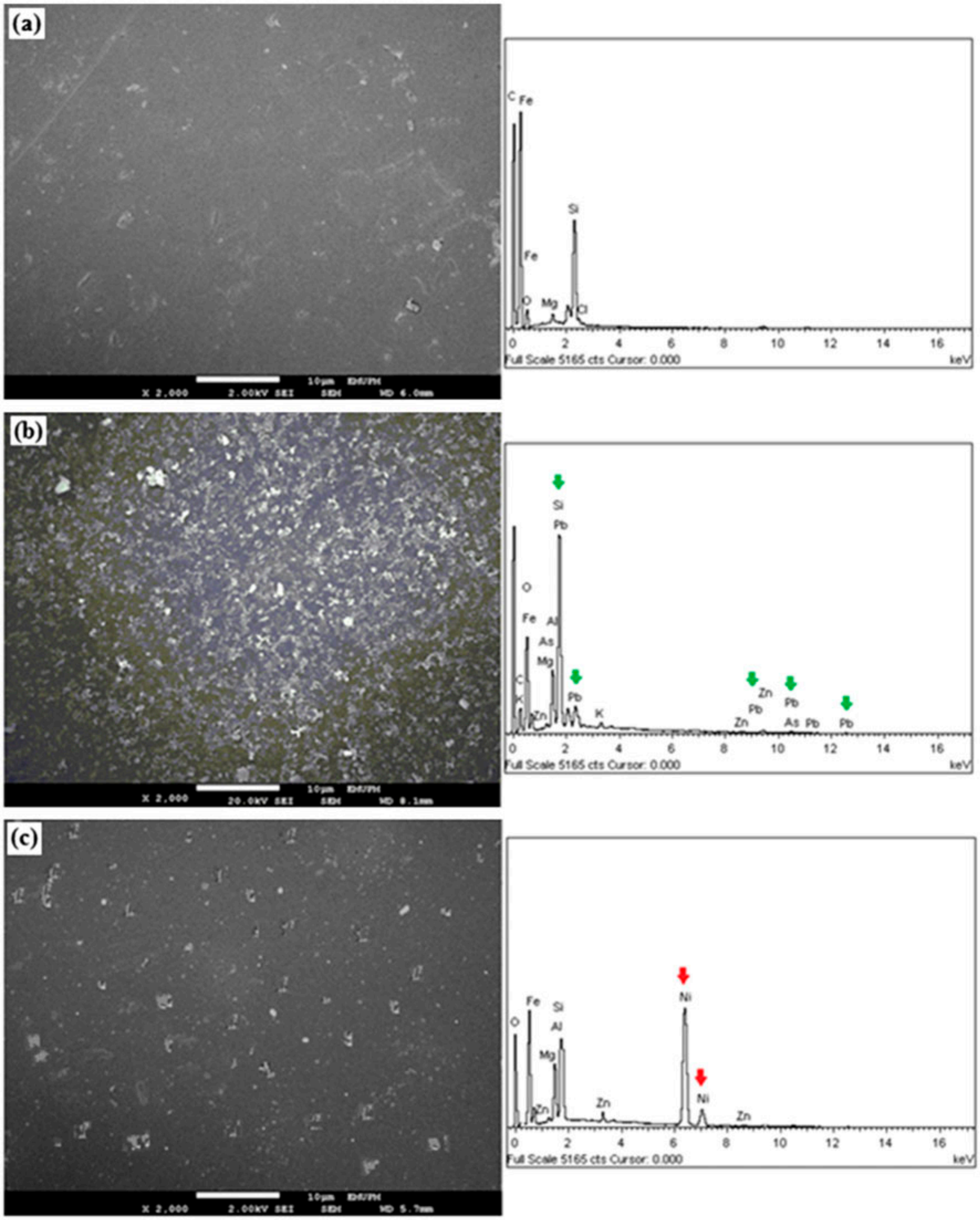

Fig. 11. The SEM-EDX images of PFT9 mixed matrix membrane (a) before removal test, (b) after lead ion removal, and (c) after nickel ion removal.

The water content of prepared membranes was analyzed to explore the variety in porosity of the membranes in different Fe_3O_4–talc concentrations. As indicated in Figs. 8 and 9, the membrane water content and porosity increased significantly in higher content of Fe_3O_4–talc nanocomposite. The highest water content and porosity were 61.3 and 49.3%, respectively, for PFT13. Membrane with the highest content of Fe_3O_4–talc exhibits the greatest water content. This phenomenon proves that the increment of

Fe_3O_4–talc concentration in the membrane structure led to the higher porosity of the membrane.

The morphology of the membranes containing different Fe_3O_4–talc concentrations was analyzed using scanning electron microscopy (SEM). Fig. 10 demonstrates the cross-section view of neat PSf, PFT7, PFT11, and PFT13 membranes. As indicated in SEM micrographs, neat PSf membrane has finger-like pores in substructure and a thick top layer (~3.4 μm) which is a main reason of the impermeability of the membrane. The thickness of top layer decreased in PFT7 (~1.7 μm) and longer finger-like pores were created in substructure. Membranes with top layer of spongy structure and support layer containing macrovoids are produced in higher concentrations of Fe_3O_4–talc nanocomposite (PFT11 and PFT13). Since hydrophilicity of membranes was improved in higher Fe_3O_4–talc concentrations, the water diffusion velocity through the membrane structure was enhanced during phase inversion. Then, instantaneous phase inversion has occurred between solvent (NMP) and non-solvent (distillated water) in the coagulation bath, which attributed to the membranes with thin surface layer and large finger-like pores and macrovoids in support layer. Hence, the size of pores and membrane porosity increased with non-solvent diffusion speed [23,24].

Scanning electron microscopy with an energy dispersive X-ray (SEM-EDX) can provide spatial and composition distribution of elements in solid materials. Heavy Elements (high atomic number) backscatter electrons more strongly than light elements, and thus appear brighter in SEM-EDX images [25,26]. SEM-EDX topographic images of the top surface of fresh PFT9 membrane and after the ultrafiltration of Pb(II) and Ni (II) ions are illustrated in Fig. 11. Bright zones on the surface of the membrane after heavy metal removal correspond to the presence of complexed lead and nickel ions (Fig. 11(b) and (c)). This phenomenon discloses that the metal ions rejection in membrane filtration process occurred through the fixation of lead and nickel ions on the membrane by interactions with the Fe_3O_4–talc nanocomposite [27].

4. Conclusions

Novel mixed matrix membrane was prepared by incorporation of Fe_3O_4–talc nanocomposites into the PSf casting solution. The BET results proved that the surface area of talc powder is 6.675 m^2/g and this number increased to 37.079 m^2/g for Fe_3O_4–talc nanocomposite. Lead and nickel ions removal was tested and the results revealed that proper amount of nanocomposite in casting solution and feed concentration are responsible for efficient metal ions elimination. The membrane containing 9.0 wt.% of Fe_3O_4–talc nanocomposite (PFT9) showed the highest lead rejection (99.4%) and nickel rejection (96.2%) for 50 mg/l Pb(II) and Ni(II) aqueous solutions. Presenting the high metal ions rejection and permeate flux at 3 bar feed pressure, the PFT9 membrane was selected as the well-performed prepared membrane in this study. The SEM-EDX images demonstrated that the adsorption mechanism improves ion rejection in the prepared mixed matrix membranes.

Acknowledgments

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