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TECHNICAL NOTE

Fabrication and Characterization of Novel Mixed Matrix Polyethersulfone Based Nanofiltration Membrane Modified by Ilmenite

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A B S T R A C T

This study is focused on fabrication and characterization of mixed matrix poly ether sulfone based nanofiltration membrane by phase inversion method. The effect of different amounts of Ilmenite ($FeTiO_3$) particles as inorganic filler additive in the casting solution on physic-chemical characteristics of membranes was studied. Scanning electron microscopy, surface analysis, water content, contact angle, permeation, rejection and tensile strength measurements were carried out in membrane characterization. SEM images showed asymmetric structures with a dense top layer and porous sub-layer for the membranes. Images revealed that membrane sub-layer porosity increased and top-layer thickness decreased by $FeTiO_3$ utilizing in membrane matrix. SEM images showed smooth surface for modified membranes compared to pristine one. Membrane water content improved initially by increase of additive concentration up to 0.05 wt% into the casting solution and then decreased. The contact angle measurements showed that membrane surface hydrophilicity improved using Ilmenite in the membrane matrix. The membrane tensile strength improved initially by increasing additive concentration into the casting solution and then decreased. Utilizing of Ilmenite into the membrane matrix caused increase of water flux and salt rejection from 10 to 172.4 $L/m^2 \cdot h$ and 53.9 to 88.5%, respectively.

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1. INTRODUCTION

Nowadays, membrane process is a promising, simple and energy-efficient technology not only in various industries but also in humans' life [1]. Among the various types of membranes, nanofiltration (NF) is being greatly developed because of its advantages such as low operating pressure, high water flux, high rejection of multivalent anion salts and organic solutes. Therefore, it has attracted worldwide interest. NF process has been used in many applications such as wastewater reclamation, industrial water production, water softening and in the separation of compounds having different molecular weights [2-7].

Polyethersulfone (PES) is one of the most important polymeric materials which have been widely used in nanofiltration preparation. PES based membranes showed good chemical and thermal stabilities as well as appropriate mechanical property [8]. The PES based membranes show hydrophobic property which leads to intense restriction in permeate flux [9].

For improving hydrophilicity and flux of hydrophobic polymeric membranes, there are several practical methods such as interfacial polymerization with hydrophilic monomers, coating of substrate membrane with hydrophilic polymer or nanoparticles and grafting and blending with nanoparticles or hydrophilic polymers [10].

In case of membranes modification methods, employing different inorganic nanoparticles such as silver, zeolite, titanium oxide, silica oxide and zirconium oxide embedded into the membrane matrix is very well known/interesting method. The utilization of inorganic materials particles into the polymeric matrix can lead to the achievement of unique physic/chemical properties and capacities such as hydrophilicity and mechanical, thermal and oxidative stabilities and also to

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improve the separation characteristics of membranes based on the synergism between the organic-inorganic components properties [11].

Ilmenite ($FeTiO_3$) is new class of advanced materials with very interesting features and capacity such as anti-ferromagnetic insulators and adsorption characteristics because of valence states of action's (Ti, Fe) in $FeTiO_3$ which provides unique physicochemical properties [12]. A few researches have considered $FeTiO_3$ particles in the membrane matrix for water desalination [13, 14].

According to the earlier reported study [15], a novel mixed matrix polyvinylchloride/ $FeTiO_3$ electrodialysis heterogeneous cation-exchange membrane was prepared by casting solution technique and the effect of $FeTiO_3$ concentration as additive in membrane matrix on electrochemical properties and performance of membrane was studied. Generally, the results revealed better performance/properties in comparison to bare polyvinylchloride membrane.

No report was found by our literature survey for application of $FeTiO_3$ particles into the mixed matrix PES based NF membranes to improve the performance and properties of membranes. So, preparing a novel mixed matrix NF membrane with suitable physicochemical properties for the application in water treatment was the main goal of the current research. For this aim, mixed matrix PES based NF membranes were prepared by solution casting technique. $FeTiO_3$ particles were utilized as inorganic filler additives into the membrane matrix to investigate its effect on the PES NF membrane performance and physicochemical properties.

Scanning electron microscopy, surface analysis, water content, contact angle, permeation, rejection and tensile strength measurements were carried out in membrane characterization. The results are valuable for NF membrane processes for water recovery and treatment.

2. EXPERIMENTAL

2. 1. Materials N, N Dimethylacetamide (DMAC, Mw = 87.12 g/mol) and polyvinylpyrrolidone (PVP, Mw = 25,000 g/mol) from Merck Inc., were used as solvent and pore former, respectively. Polyethersulfone (PES) provided by BASF (Ultrason E6020P, MW= 58,000 g/mol) was employed as membrane based binder. Ilmenite ($FeTiO_3$, powder, average particle size < 37 μm) from Iran, was utilized as inorganic filler additives. Distilled water was also used as non-solvent (coagulation bath) throughout the experiments. Sodium sulfate (Na_2SO_4) was also supplied from Merck.

2. 2. Membrane Preparation The PES-co-$FeTiO_3$ mixed matrix membranes were prepared by phase inversion method and casting solution technique.

TABLE 1. Composition of used casting solutions in membrane preparation

Membranes no.	DMAC (wt. %)	$FeTiO_3$ (wt. %)
M1	79	0
M2	78.95	0.05
M3	78.9	0.1
M4	78.5	0.5
M5	78	1

Constant concentration of PES and PVP (20: 1 w/w %)

The preparation proceeded by dissolving desired amount of polymer binder (PES) and the pore former (PVP) into the solvent (DMAC) in glass reactors equipped with a mechanical stirrer for more than 4 h. This was followed by dispersing different amounts of $FeTiO_3$ particles as inorganic filler additive into the polymeric solutions. The mixture was mixed vigorously at room temperature to obtain uniform particle distribution into the polymeric solutions. For better dispersion of particles and breaking up their aggregates, the solutions were then sonicated for 30 min using an ultrasonic instrument. After the sonication, the mixing process was repeated for another 10 min using the mechanical stirrer. Also the prepared solutions were kept for one day to completely remove air bubbles dissolved in solutions and then were casted on dry and clean glass plates using casting knife with 150 μm thickness. The casted polymeric films were immediately immersed into deionized water at room temperature for 24 h until solvent was extracted and solidification was done completely. The composition of casting solutions are depicted in Table 1.

2. 3. Characterization of the Membranes

2. 3. 1. Morphological Studies Cross sectional structures of the prepared membranes were monitored using scanning electron microscope (SEM, Philips, Model XL30, and The Netherlands). Before scanning the samples by SEM apparatus, the samples were frozen in liquid nitrogen and fractured. After sputtering with gold, they were observed by the electron microscope. Also for the evaluation of membrane roughness, 3D image metrology software was used.

2. 3. 2. Water Content and Contact Angle The water content was measured as the weight difference between the dried membranes and swollen ones. The wet membranes were weighed initially (OHAUS, Pioneer™, readability: 10^{-4} g, OHAUS Corp., USA) and then dried in an oven (Behdad Co., Model: O5, Iran) at 50 ^{o}C for 24 h until constant weight was obtained as dry-membrane. The following equation was used for water content calculation [15-17]:

$$Water\ content\% = (\frac{W_w - W_d}{W_w}) \times 100 \quad (1)$$

where W_w and W_d are the wet and dry membranes' weight (g), respectively. For minimizing the experimental errors, all measurements were performed three times for each membrane and their average values were reported.

Also water contact angle was used to evaluate the changes in the hydrophilicity and surface wetting characteristic of the prepared membranes. De-ionized water was used as the probe liquid in all measurements. To minimize the experimental error, the contact angle was measured in five random locations for each sample and then their average was reported. All experiments were carried out in the ambient conditions. Imaging of water droplets on the membranes surface was taken using digital camera (Model: Canon, EOS 1300D 18-55mm DC III) [18]. Then the angle between membrane surface and water droplet was measured with Image J software.

2. 3. 3. Mechanical Properties The tear resistance as a mechanical property of the prepared membranes was tested according to ASTM1922-03. All samples were cut in the standard shapes in the ambient conditions before testing. For each test, three samples were used and average values were reported [19].

2. 3. 4. Flux and Rejection The membrane permeability flux and salt rejection measurements were carried out using stirred dead-end cell filtration system with 11.94 cm^2 effective surface area. All of the experiments were done at 0.5 MPa as driving force which was created by nitrogen gas (99.9 % purity). The permeability flux was calculated as follows [20]:

$$J = (\frac{V}{A * \Delta t}) \quad (2)$$

where J, V, A and Δt are permeability flux (L/m^2h), volume of permeate (L), membrane surface area (m^2) and filtration time (h), respectively.

Also the following equation was used for calculation of membrane salt rejection [21, 22]:

$$Rejection(\%) = (\frac{C_1 - C_2}{C_1}) * 100 \quad (3)$$

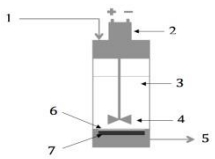

Figure 1. Schematic diagram of used dead-end stirred cell: (1) N_2 gas input, (2) electrical motor, (3) feed solution, (4) blades, (5) permeate (dilute solution), (6) membrane and (7) supported layer.

where, C_1 and C_2 are the Na_2SO_4 concentrations in permeate and feed measured by conductivity-meter (OHAUS, ST3100C, OHAUS Corp., USA).

3. RESULTS AND DISCUSSION

3. 1. Morphological Characterizations The cross-sectional SEM images of prepared membranes are shown in Figure 2. As it is seen, all of the prepared membranes have asymmetric structures with a thin and dense selective layer and a thick and porous sub layer. As can be seen in the images, utilization of additive particles into the membrane matrix caused obvious changes in membrane structure. Presence of $FeTiO_3$ particles into the casting solution also led to decrease of membranes top layer thickness obviously from 1602 nm for M1 (neat PES membrane) to 1129 nm for M4 containing 0.5 wt% $FeTiO_3$ particles. Additionally, the sub layer porosity was increased by embedding additive particles into the matrix. The reason can be explained with respect to instantaneous demixing phenomenon occurred during phase inversion process due to thermodynamic instability of casting solution which is assigned to addition of $FeTiO_3$ particles into the casting solution. So, increase of exchanging rate between solvent and non-solvent leads to formation of membrane with larger macro voids in sub-layer and thinner top layer (Figures 2c, d and e) compared to the neat PES. Decreasing membrane top layer thickness and increase of porosity in the sub layer can strongly offer the higher permeability for prepared membranes [23]. Additionally, Figure 3 exhibited the changes of roughness in the membranes surface caused by addition of different amounts of $FeTiO_3$ particles into the casting solution. The bright areas show the peaks on the membrane surface and dark areas illustrate the valleys areas. According to Figure 3, addition of $FeTiO_3$ particles into the membrane matrix has significant effect on roughness. As can be seen, the surface of neat PES membrane (Figure 3a) is rougher than that of membranes filled with $FeTiO_3$ particles (Figures 3b, c, d). This can lead to the higher hydrophobic surface for the PES membrane. As shown, the mixed matrix membranes (M2, M3 and M4) filled with $FeTiO_3$ particles is clearly smoother than that of the neat PES membrane. The smoother surface provides more hydrophilic characteristic for the mixed matrix membranes.

The roughness is an important parameter which affects the adsorption/desorption of foulants on the membranes surface. Decrease of roughness introduces more hydrophilic surface which can result in the lower adsorption of foulants on the surface of membranes leading to the higher rejection [24]. Subsequently, it is expected that the mixed matrix membranes (M2, M3 and M4) display higher water content and permeability

flux with lower contact angle compared to the pristine PES one. As shown in Figure 3e, the roughness increased again by embedding 1 wt% $FeTiO_3$ particles in the casting solution (M5). This may be attributed to the excessive additive particles placed in the membrane surface at high $FeTiO_3$ particles loading rate (1 wt%) which can increase the surface roughness [25].

3. 2. Water Content and Contact Angle The water content and contact angle studies have been employed to evaluate the effect of $FeTiO_3$ particles embedded into the membrane matrix on hydrophilicity and wettability of PES based membrane. Obtained results (Figure 4) revealed that membrane water content was enhanced initially by increase of $FeTiO_3$ particles content up to 0.05 wt% in the casting solution. This may be related to hydrophilic characteristic of $FeTiO_3$ particles which increase the exchange rate between solvent and non-solvent in coagulation bath [26] and causes the formation of larger pores and channels in membrane matrix (see SEM images). This provides more free spaces, voids and cavities in the membrane matrix and increases the possibility of water accommodation which results in more water content for the membranes. The membrane water content decreased again by more increase of additive content (1 wt%). This may be due to the pores and channels filling phenomenon (pore blockage) which is assigned to excessive particles at high additive loading ratio (see Figure 2c, d and e) and causes less water content for the membrane. Also the effect of $FeTiO_3$ particles loading ratio into the casting solution on the membrane surface hydrophilicity was examined by contact angle measurement. The photographic images of contact angle are shown in Figure 5. The results (Figure 5 and Table 2) indicated that the contact angle decreased from 66° for bare PES membrane to 43° for the filled membrane containing 0.5 wt% $FeTiO_3$ particles. The reason is due to increase of membrane surface hydrophilicity by utilizing hydrophilic $FeTiO_3$ particles in the membrane matrix which leads to the higher affinity between membrane surface and water droplets. As mentioned, for membranes surface roughness presented at Figure 3, decrease of surface roughness for M2, M3 and M4 compared to M1, which introduces hydrophilic characteristic to the membrane surface, can be another reason for lower contact angle at this range of additive loading ratio (0.05 - 0.5 wt%). The water contact angle increased again with addition of 1 wt% $FeTiO_3$ particles in the casting solution. This may be related to the higher roughness in the membrane surface at 1 wt% additive concentration (see Figure 3e) which introduces more hydrophobic area on membrane surface which leads to higher water contact angle (50) [24].

3. 3. Mechanical Properties of Prepared Membranes The effect of $FeTiO_3$ particles concentration on tensile stress of PES membrane was studied according to ASTM1922-03.

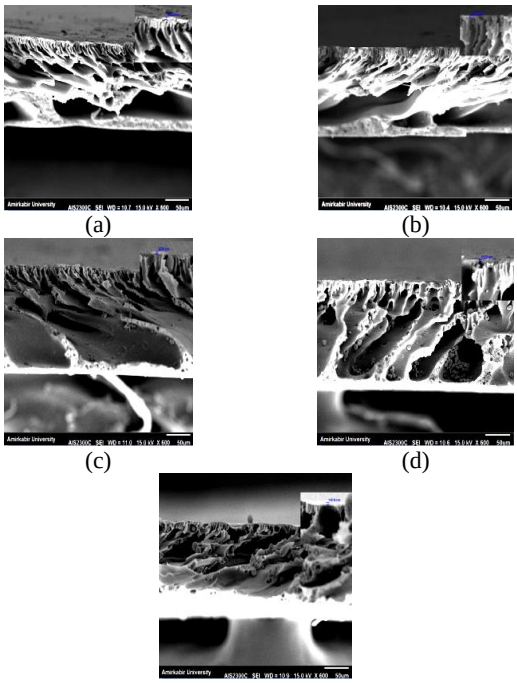

Figure 2. Cross-sectional SEM images of prepared membranes with different contents of $FeTiO_3$ particles: (a) 0.0 wt.%; (b) 0.05 wt.%; (c) 0.1 wt.%; (d) 0.5 wt.% and (e) 1 wt.%.

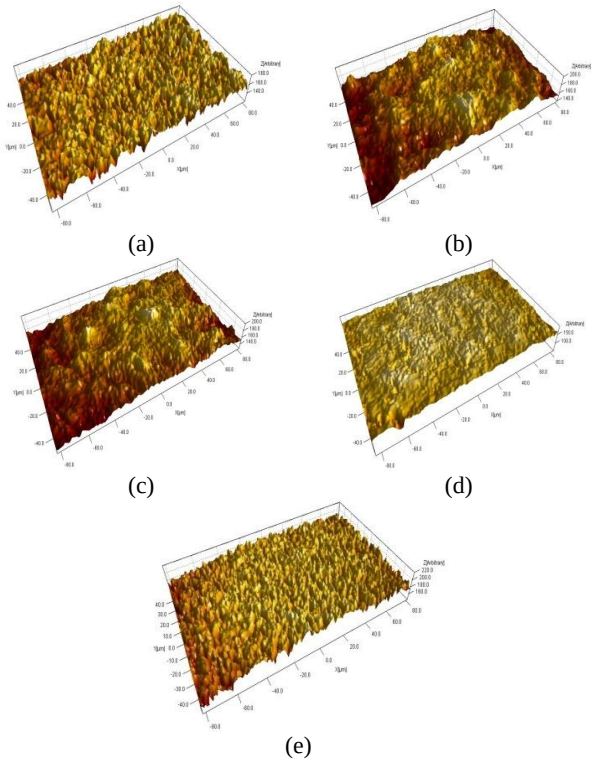

Figure 3. 3D surface images of prepared membranes with different concentrations of $FeTiO_3$ particles: (a) 0.0 wt.%; (b) 0.05 wt.%; (c) 0.1 wt.%; (d) 0.5 wt.% and (e) 1 wt.%.

Obtained results (Figure 6) showed that tensile strength of prepared membranes improved initially from 2120 to 3670 kPa by addition of 0.1 wt% $FeTiO_3$ particles into the membrane matrix and then decreased from 3670 to 3150 kPa by more addition loading ratios. The improvement of tensile strength for the prepared membrane may be related to the strong interfacial bonding formed between the polymers and additive particles which tend to improve the mechanical properties.

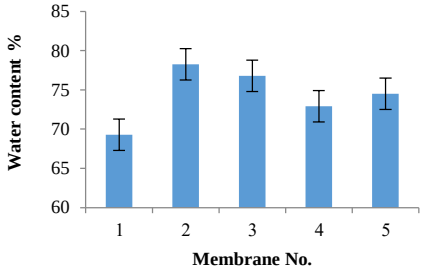

Figure 4. The effect of $FeTiO_3$ content ratio on membrane water content.

TABLE 2. The effect of $FeTiO_3$ particles concentration in membrane matrix on contact angle

Membrane no.	Contact angle(°)
1	66
2	59
3	55
4	43
5	50

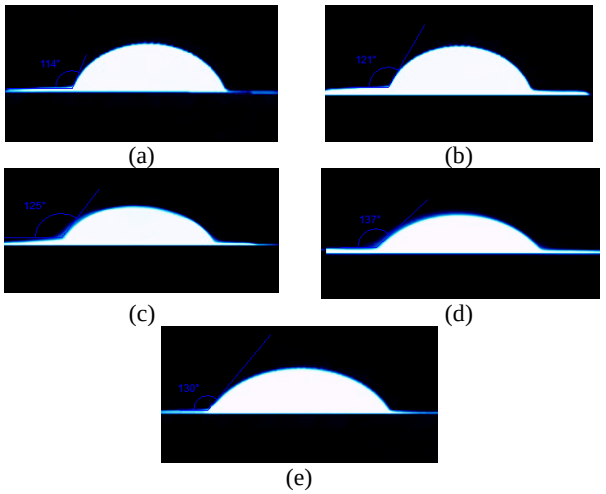

Figure 5. Contact angle images of membranes with different contents of $FeTiO_3$ particles: (a) 0.0 wt.%; (b) 0.05 wt.%; (c) 0.1 wt.%; (d) 0.5 wt.% and (e) 1 wt.%.

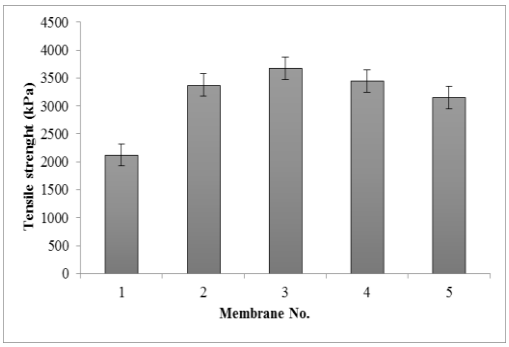

Figure 6. The effect of $FeTiO_3$ particles concentration on membrane tensile strength.

The particles agglomeration at higher additive concentrations (0.5 and 1 wt%) reduced the favorable molecular interactions between the additive particles and polymer binder [27, 28] which can be the main reason for the reduction of membrane tensile strength for M4 and M5.

3. 4. Membrane Filtration Performance The membranes permeability flux (Figure 7) initially enhanced from 10 to 172.4 ($L/m^2.h$) by increase of the additive concentration up to 0.05 wt% into the casting solution. This may be attributed to thinner top layer (Figure 2) and also higher water content for M2, M3 and M4. Moreover, as shown in Figure 3 (surface roughness), decrease of membrane roughness by embedding of additive particles (0.05 wt%) leading to the improvement of membrane surface hydrophilicity (see Figure 5) which can accelerate the water flux. The permeability flux decreased again by increase of additive concentration from 0.05 to 1 wt% into the casting solution. The reduction of flux at this range of additive incorporation may be related to pore blocking phenomenon occurred by excessive particles [24]. The SEM images illustrated in Figure 2 (c, d, e), confirms pore blocking by particles. Figure 8 shows the effect of $FeTiO_3$ particles concentration in the membrane matrix on salt rejection.

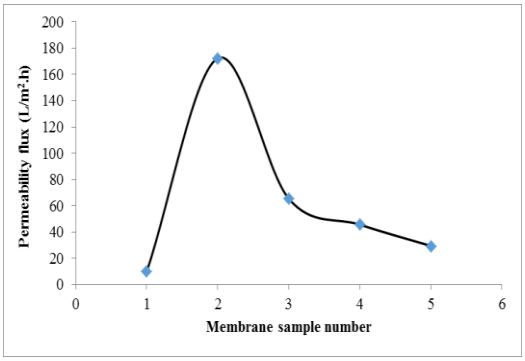

Figure 7. The effect of $FeTiO_3$ concentration in membrane matrix on permeability/flux.

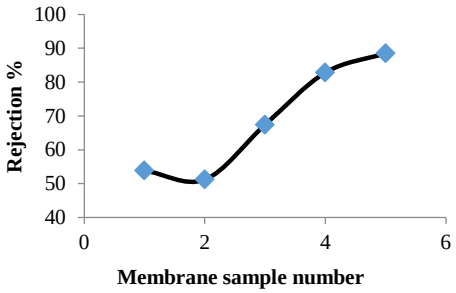

Figure 8. The effect of $FeTiO_3$ particles concentration on membrane salt rejection.

The salt rejection capability of prepared mixed membranes increased strongly from 53.9% for neat PES membrane to 88.55% for the modified membrane filled with 1 wt% particles. Increase of rejection at the range of 0.05- 0.5 wt% particles loading ratio also can be due to the reduction of roughness and smoother surface for M2, M3 and M4 (see Figure 3). At this condition, as seen in Figure 5, higher hydrophilicity was observed at the membranes surface and so salt rejection increased. As mentioned before, decrease of surface roughness leads to higher hydrophilicity and so results in lower aggregation of foulants on the membrane surface which improves the rejection [29, 30]. This means that the membranes' performance is strongly affected by the physical properties of membranes.

As can be seen in Figures 3 and 5, surface roughness and also water contact angle increased for M5 which resulted in low rejection for the membrane. But the obtained results in Figure 8 clearly revealed the increase of rejection even for M5. The reason can be explained by pore blocking by additive particles which reduce the membrane pore size. In this situation entrance of ions into the membranes pores occurs and rejection increases.

4. CONCLUSION

In the current research novel mixed matrix nanofiltration membranes were prepared by directly incorporation of $FeTiO_3$ particles into the casting solution. The effect of different contents of $FeTiO_3$ particles in the casting solution on the membrane physicochemical properties was investigated. SEM images showed asymmetric structures with a dense top layer and porous sub-layer for the membranes. Images revealed that membrane sub-layer porosity increased and top-layer thickness decreased by incorporation of $FeTiO_3$ in membrane matrix. SEM images also showed smooth surface for the modified membranes compared to pristine ones. Membrane water content improved by increase of additive concentration up to 0.05 wt% and then decreased. The contact angle measurements showed that membrane surface hydrophilicity improved by utilizing Ilmenite in membrane matrix. The membrane tensile strength initially improved by increase of additive concentration in the casting solution and then decreased. Utilizing Ilmenite in the membrane matrix caused sharp increase of water flux and salt rejection from 10 to 172.4 $L/m^2 \cdot h$ and 53.9 to 88.5%.

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Fabrication and Characterization of Novel Mixed Matrix Polyethersulfone Based Nanofiltration Membrane Modified by Ilmenite

TECHNICAL NOTE

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چکیده

در این پژوهش غشاهای شبکه آمیخته نانوفیلتراسیون بر پایه پلی اتر سولفون به روش تغییر فاز ساخته شده و مورد ارزیابی قرار گرفت. اثر ذرات المنیت با غلظت های متفاوت به عنوان ماده پر کننده در ساختار غشاهای تهیه شده، بر خواص شیمی/فیزیکی و جداسازی آنها مورد بررسی قرار گرفت. آنالیز میکروسکوپ الکترونی، آنالیز سطح، محتوای آب، زاویه تماس، میزان عبوردهی، پس دهی و تست مقاومت مکانیکی جهت بررسی عملکرد غشاها مورد استفاده قرار گرفت. عکس های میکروسکوپ الکترونی نشان داد که غشاهایی نامتقارن با لایه بالایی متراکم و لایه پایینی متخلخل شکل گرفته است. همچنین تصاویر نشان داد که استفاده از ذرات المنیت در ساختار غشاها سبب افزایش میزان تخلخل لایه نگهدارنده و کاهش ضخامت لایه فعال گشته است. تصاویر تهیه شده حاکی از ایجاد غشاهای شبکه آمیخته با سطح صاف در مقایسه با نمونه غشای اصلاح نشده است. نتایج نشان داد که میزان محتوای آب غشاها در ابتدا با افزایش میزان غلظت ذرات المنیت تا ۰/۰۵ درصد وزنی افزایش یافت و سپس با افزایش بیشتر میزان غلظت آنها روندی کاهشی نشان داد. همچنین نتایج زاویه تماسی نشان داد که آبدوستی سطحی غشاها در اثر به کار گرفتن ذرات المنیت در ساختار غشاها بهبود یافته است. مقاومت مکانیکی غشاها نیز در ابتدا با افزایش میزان غلظت ماده پرکننده افزایش یافته و سپس مجددا کاهش یافت. استفاده از ذرات المنیت در بدنه غشاها سبب افزایش فلاکس از ۱۰ تا ۱۷۲/۴ (لیتر/مترمربع.ساعت) و میزان پس دهی از ۵۳/۹ تا ۸۸/۵ درصد گردید.

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