

Research Paper

Evaluation of a Nanohybrid Membrane (PSF/ZnO) Efficiency in Natural Organic Matter Removal From Water



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ABSTRACT

Background: Natural organic matter (NOM) in drinking water sources has always been regarded as a precursor for the formation of trihalomethanes (THMs), haloacetic acids (HAAs), and carcinogenic properties. This study aimed to fabricate and characterize a nanohybrid ultrafiltration membrane (PSF/ZnO) to evaluate its efficiency in NOM removal from water.

Methods: Nanohybrid membranes with ratios of 0, 1, 2, 3 and 4% w/w of ZnO nanoparticles (NPs) were fabricated using the phase inversion method and characterized by the contact angle, AFM, FTIR, and SEM analyses. In this study, the effects of initial humic acid (HA) concentration, operating pressure, pH, and filtration time were examined on the HA removal efficiency and pure water flux through the membrane.

Results: The results showed that addition of the ZnO NPs to the PSF membrane reduced the contact angle on the PSF/ZnO nanohybrid membrane's surface. According to FE-SEM images, increasing the ZnO concentration changed the porous structure of the membranes from a spongy, teardrop shape to a finger-like channel structure. The FTIR analysis revealed an increase in the hydrophilicity of the membrane due to the presence of hydroxyl functional groups in ZnO. AFM images indicated an increase in the surface roughness of nanoparticle-containing membranes. It was found that an increase in the concentration of the ZnO NPs (0-4% w/w) increased HA removal efficiency (43.63-83.4%).

Conclusion: This study demonstrated that the use of the PSF/ZnO nanohybrid membranes increased HA removal efficiency and pure water flux passing through the membrane.

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1. Introduction

According to the standards of the World Health Organization (WHO), safe water is not available to a significant percentage of the world population, and this issue is exacerbated by climate change and the spread of pollutants [1]. Among the pollutants in water, natural organic matter (NOM) is considered an important factor that has always been a problem in water treatment plants [2]. NOM is a non-uniform mixture of organic compounds that are derived from plants, animals, microorganisms, and their waste products, which enter the water by the decomposition of these resources [2, 3]. The molecules of NOM generally contain many functional groups that affect their chemical behaviors, and therefore these materials are different in reactivity, structure, and color properties [4]. In addition to color, odor, and taste creation in water, the presence of these substances can interfere with the oxidation and removal of heavy metals, such as arsenic, iron, and manganese, leading to additional costs in water treatment [2]. Moreover, the presence of these substances increases both the biological growth in the distribution network and the coagulant and disinfectant doses and, most importantly, causes the formation of disinfection by-products (DBPs) [4-6]. The major compounds are trihalomethanes and haloacetic acids, which are mostly toxic, carcinogenic, and mutagenic compounds [7]. The WHO has set a NOM concentration limit of <0.3 mg/L in drinking water in comparison to that of about 2.47 mg/L in surface waters [5].

NOM removal from water is possible through various treatment methods [4, 7]. Membrane technologies are among the promising methods that are widely being used in water treatment and the removal of pollutants [8]. To remove pollutants, including organic matter removal from water, the use of membrane systems has several advantages. These include no need for chemical addition, no waste production, easy to increase capacity, easy connection of membrane systems to other processing units, no high dependence on environmental conditions, removal of other soluble contaminants and microorganisms, and most importantly, no production of by-products and intermediates [9, 10].

Some disadvantages, such as the hydrophobicity of the membrane surface, clogging of the membrane, high water loss, and high energy consumption, limit the extensive use of membranes [9, 11, 12]. To reduce these limitations, recent years have witnessed the development of extensive studies on modifying polymer

membranes to achieve maximum efficiency and flux with less clogging [13]. Nanocomposite membranes are a new and modified class of polymeric membranes produced from combining mineral nanoparticles in a polymer matrix [14]. Inorganic nanoparticles (NPs) used in membrane modification include aluminum(III) oxide (Al_2O_3), titanium dioxide (TiO_2), silica (SiO_2), zirconium dioxide (ZrO_2), and magnesium oxide (MgO) NPs [15]. Zinc oxide (ZnO) is the other NPs used in membrane modification [16].

The ZnO NPs are one of the inexpensive metal oxides [14] and their antimicrobial and anticorrosive properties, as well as good mechanical and thermal stability, have been of interest to various industries including glassmaking, electronics, and, more recently, the modification of polymeric membranes [12]. The combination of the ZnO NPs with the structure of membrane-forming polymers, such as polysulfone (PSF), polyethersulfone, and polyvinyl fluoride, have been reported in various studies, mainly resulting in increased permeability, porosity, hydrophilicity, and the anti-clogging properties of membranes [12]. This study aimed to fabricate and characterize a nanohybrid ultrafiltration membrane (PSF/ ZnO) to evaluate its efficiency in NOM removal from water.

2. Materials and Methods

In the present experimental interventional study on a laboratory scale, the study consisted of solutions containing different amounts of humic acid (HA) as the representative of organic matter examined in different values of operating parameters (concentration, pH, pressure, and passage time) using a membrane system. In this study, all chemicals were prepared with laboratory purity to produce nanohybrid membranes with different percentages of the ZnO NPs (Table 1).

Synthesis of nanohybrid membranes (PSF/ ZnO)

In this study, crude PSF membranes and modified nanohybrid membranes (PSF/ ZnO) were synthesized by the phase inversion method in which the casting solution contained the PSF polymer as the main polymer of the membrane body, the ZnO NPs with specific weight percentages as the membrane modifying agent, and the PVP polymer as the pore former. Dimethylformamide (DMF) and deionized water were also used as the polymer solvents and non-solvents, respectively (Table 2). Accordingly, different percentages of the ZnO NPs with PVP were first dispersed in the DMF solvent and the resulting mixture was placed on a magnetic stirrer (500 rpm) at 40°C for 30 min to completely dissolve NPs. Then, the

Table 1. The characteristics of the materials used in this study

Name	Shape	Molecular Weight (gr/mol)	Chemical Structure	Co.
PSF	Solid; granule; yellow	35000~	$[C_6H_4-4-C(CH_3)_2-C_6H_4-4-OC_6H_4-4-SO_2C_6H_4-4-O]_n$	BASF
DMF	Liquid; colorless	73.09	$HCON(CH_3)_2$	Merck
ZnO	Solid; powder; white	81.408	ZnO	Sigma
PVP-K40	Solid; powder; white	40000	$(C_6H_9NO)_n$	Sigma Polyvinylpyrrolidone
Humic acid	Solid; powder	227.172	$C_9H_9NO_6$	Sigma

PSF: polysulfone; DMF: Dimethylformamide; ZnO: Zinc oxide; PVP-K40: Polyvinylpyrrolidone-K40

PSF polymer was added to the mixture and placed on the magnetic stirrer (500 rpm) at 40°C for another 8 h until the PSF polymer was completely dissolved in the solvent, yielding a uniform mixture. Next, the resulting mixture was kept staying for 24 h to ensure the absence of air bubbles. Before casting, the resulting solution was placed in an ultrasonic device at a frequency of 40 kHz for 10 min. Afterward, a film layer with a thickness of 20 microns was cast on a perfectly smooth glass surface using a 20 cm wide film applicator. After 20 sec, the polymer film was placed in a coagulation bath. Finally, the synthesized membranes were washed with deionized water to remove the likely residual solvent and then kept in deionized water until use [15].

Characterization of the synthesized membranes (PSF/ZnO)

In this study, changes in the hydrophilic properties of the synthesized membranes were measured using the contact angle between water and the upper surface of the membrane by a contact angle system (OCA 15 Plus, Dataphysics, Germany). The functional groups at the membrane surface were determined using Fourier-transform infrared spectroscopy (FTIR) device (Tensor 27, Bruker Inc., Germany). The structure, morphology, and surface

roughness of the synthesized membranes were analyzed by a scanning electron microscope (SEM) (MIRA3, TSCAN Co., Czech Republic) and an atomic force microscope (AFM) (Full No. 0101/A Nano Experts Advanced, Iran).

Membrane system, determination of flux, and HA removal efficiency

In this study, the passing flux and rejection of nano-composite membranes were evaluated using a laboratory-scale filtration system (Figure 1). According to the figure, the membrane system was patterned as a completely sealed, stainless steel plate with an effective surface of 63.62 cm² to provide the possibility of maneuvering and changing the operating situation depending on the intended goal. Also, the pilot was designed in such a way to facilitate having both cross flow and dead-end modes.

The initial concentration of HA (3, 11, and 20 mg/L), inlet pressure to the membrane (4, 6, and 8 bar), operating time (10, 35, and 60 min), and pH of the solution (5, 7, and 9) were considered to determine the optimal operating parameters. Pure water flux (PWF) passing through the membrane was calculated by measuring the

Table 2. Properties of the synthesized membranes using the phase inversion

Membrane Code	Casting Solution			
	PSF (%)	DMF (%)	PVP (%)	ZnO (wt%)
M-0	18	81	1	0
M-1	18	81	1	1
M-2	18	81	1	2
M-3	18	81	1	3
M-4	18	81	1	4

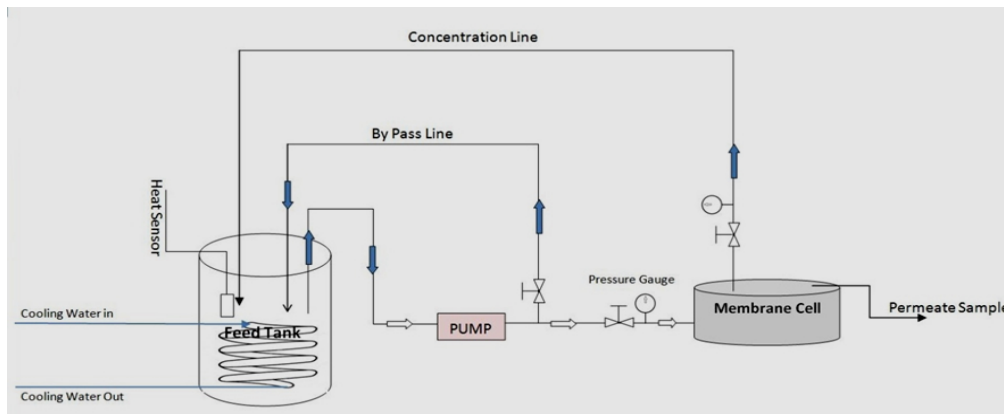


Figure 1. Schematic diagram of the membrane system used in this study

volume of infiltrating water from the membrane surface exposed to feed passage per unit time using Equation 1.

$$1. J_w = V / A \Delta t$$

In this equation, (J_w) is the infiltrated PWF ($L / (m^2 \cdot h)$), Δt is sampling time (h), V is the volume of water passing through the membrane (L), and A is the effective membrane surface area (m^2). Finally, HA removal efficiency was calculated using Equation 2.

$$2. R(\%) = 1 - C_p / C_f \times 100$$

In this equation, R is membrane removal efficiency (%), C_p is HA concentration (mg/L) as the feed in the tank, and C_f is HA concentration (mg/L) in water passing through the membrane. In this study, the NOM concentration was determined by spectrophotometry according to the following reference [17].

3. Results and Discussion

The results of the contact angle of the synthesized membranes with different percentages of NPs and SEM images of the upper surface and cross-sectional area of these membranes are presented in Figures 2-7 show the results of energy-dispersive X-ray (EDX), ATR-FTIR, and AFM spectroscopy analyses of the synthesized membranes.

As shown in Figure 2, adding the ZnO NPs to PSF membranes reduces the contact angle in PSF/ZnO nano-hybrid membranes, which may result from the presence of the hydrophilic ZnO NPs synthesized on the upper surface of the membrane. The hydrophilic nature of the ZnO NPs significantly increases the viscosity of the casting solution and accelerates the solvent and non-solvent exchange rate [18]. Evidently, more permeability is observed in all the synthesized nanohybrid membranes

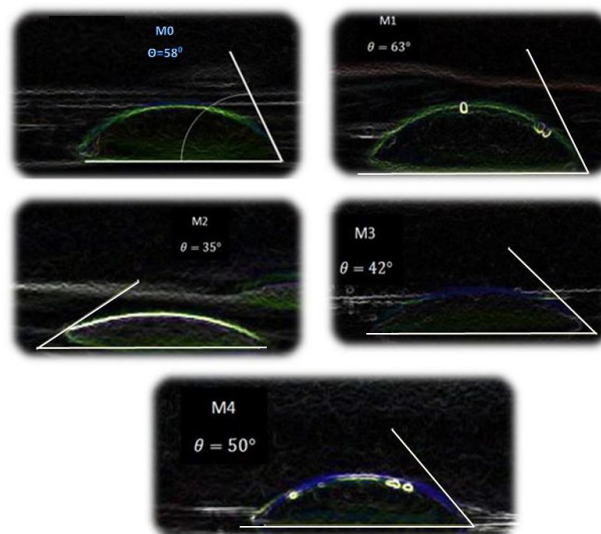


Figure 2. Contact time of the polysulfone membranes modified with different percentages of ZnO

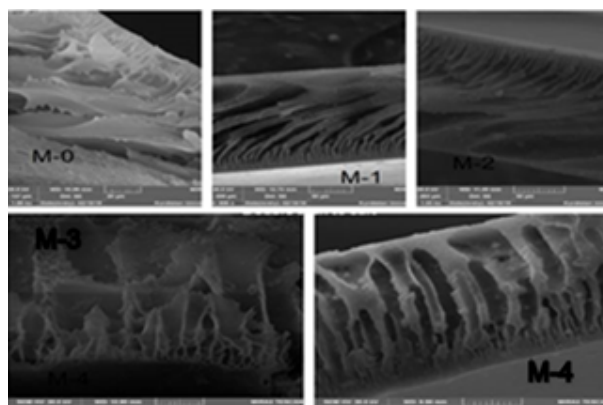


Figure 3. The SEM of upper surface of the synthesized membranes

than pure PSF membranes due to the synergistic effects between the polymer and the used NPs [19]. [Figure 3](#) depicts the SEM analysis images obtained from the upper surface of the synthesized membranes. Images of membranes without NPs reveal a denser and more compact upper surface of this membrane with fewer pores than that containing the ZnO NPs. Compared to modified membranes, the porosity increased by increasing the amount of NPs. To explain this phenomenon, the increase of NPs can increase the solvent-non-solvent displacement rate, thereby creating a porous structure [12]. In the M-0 membrane (without the ZnO NPs, [Figure 4](#)), the dense layer has a spongy structure and is thicker than that of the other synthesized membranes. However, the thickness of the dense membrane layer decreases in synthesized membranes containing the ZnO NPs, and the porous structure of the membranes changes from a spongy, teardrop shape to a finger-shaped channel structure with increasing the ZnO concentration. This morphological change arises from an increase in the solvent and non-solvent exchange rate due to the presence of hydrophilic the ZnO NPs. The increased viscosity of the casting solution due to the addition of higher amounts of

the ZnO NPs (weight percentages higher than 2 wt%) gradually reduces the solvent and non-solvent exchange rate, which results in the suppressed formation of macropores in the membrane, thereby increasing the spongy membrane in the system. Rosnan et al. obtained similar results in a study on the effects of loading ZnO NPs on the improved performance of a PSF/ZnO-GO mixed-matrix membrane [20].

The results of EDX spectroscopy for the synthesized membranes ([Figure 5](#)) show the presence of ZnO NPs and confirm the claim of using different percentages of ZnO NPs distributed uniformly in the synthesized membranes. The results of the FTIR spectra of the synthesized membranes ([Figure 6](#)) indicate that there is a dominant peak in the 500 cm^{-1} region, which belongs to the stretching frequency of ZnO [21]. Two absorption peaks observed in the range of 1230 and 1320 cm^{-1} correspond to the stretching frequency of O=S=O and the presence of PSF [22, 23]. In addition, an adsorption peak is present in the range of 3409 cm^{-1} in all synthesized membranes, except the membrane without the ZnO NPs, which is related to the stretching frequency of O-H. The

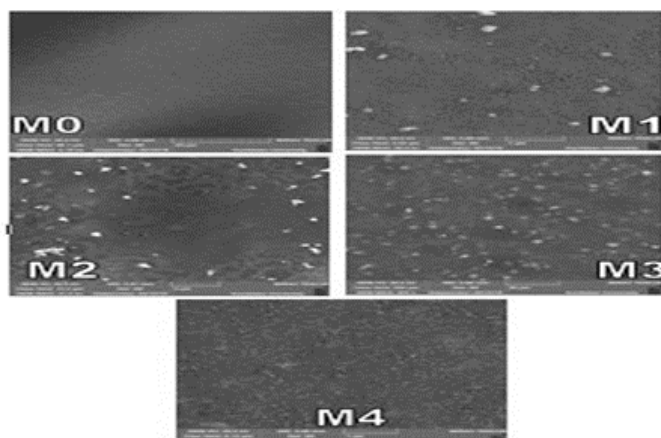


Figure 4. SEM of cross-section of the synthesized membranes

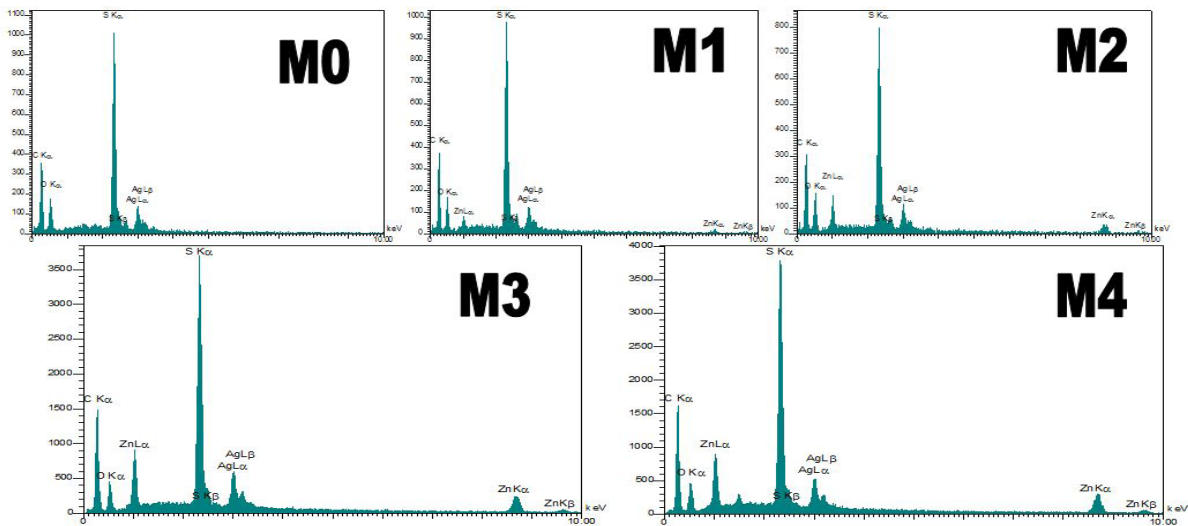


Figure 5. Energy-dispersive X-ray spectroscopy of the synthesized membranes

stretching strength of this peak differs in different percentages of NPs, indicating that ZnO NPs contain hydrophilic groups, and PSF/ZnO nanohybrid membranes possess better hydrophilic properties than PSF membranes without the ZnO NPs [23]. Other peaks observed in 690 and 2960 cm^{-1} belong to the amine stretching and the asymmetric stretching of the CH_2 ring [24]. Compared to nanoparticle-free membranes, the absence of new peaks indicates the absence of a covalent bond [23]. According to the AFM images (Figure 7), the higher the amount of NPs used in the casting solution, the greater the surface roughness of the membrane. This phenomenon occurs due to the hydrophilicity of the NPs and their

tendency to stay in the coagulation bath [25, 26]. The presence of mineral NPs causes surface roughness of the membranes, and the amount of roughness rises with the increasing amount of the NPs [12]. The performance results of the synthesized membranes under similar operating conditions are presented in Figures 8 and 9.

According to the results of PWF passing through the synthesized membranes (Figure 8), the PWF values passing through the synthesized membranes M-0, M-1, M-2, M-3, and M-4 are 18.87, 25.12, 55.1, 41.42, and 37.82 $\text{L}/\text{m}^2\cdot\text{h}$, respectively. As shown in the figure, the PWF values of all modified membranes are higher

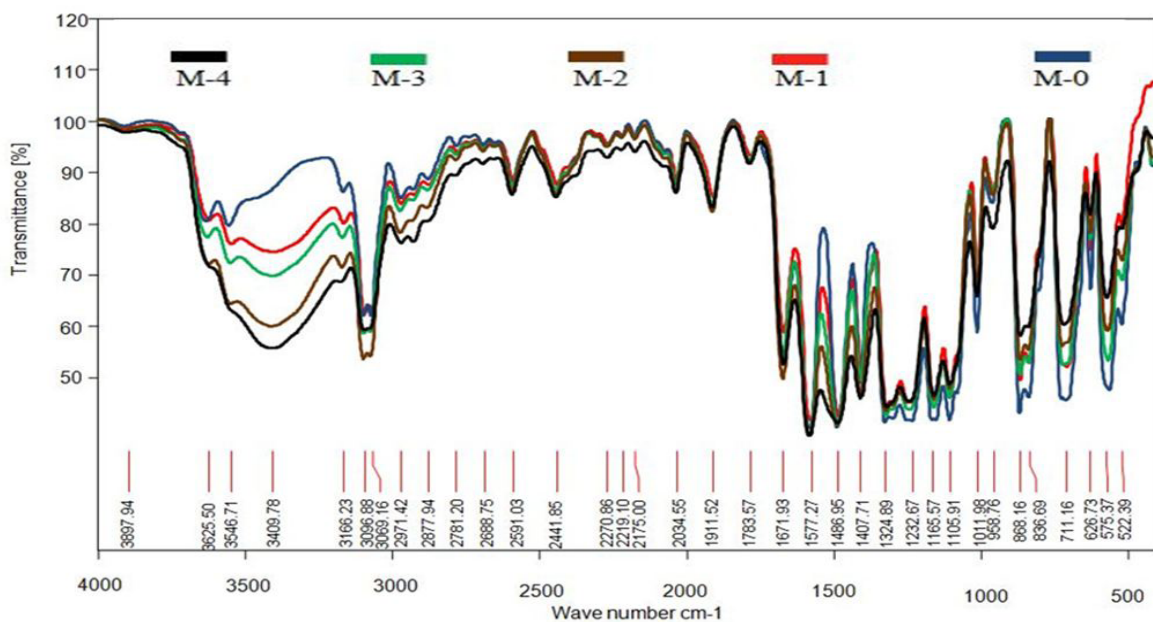


Figure 6. Fourier-transform spectroscopy of the synthesized membranes

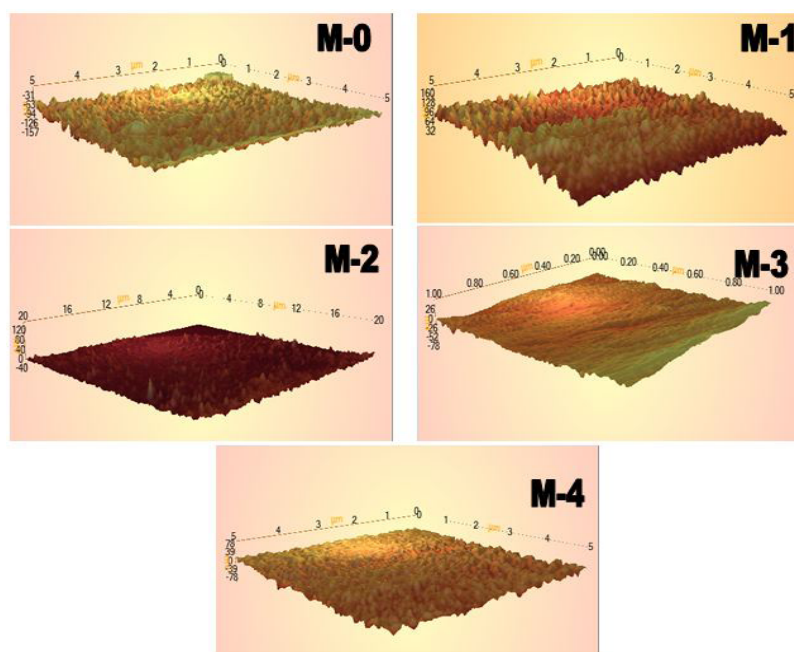


Figure 7. 3D AFM images of the upper nanocomposite membranes PSF and PSF/ZnO

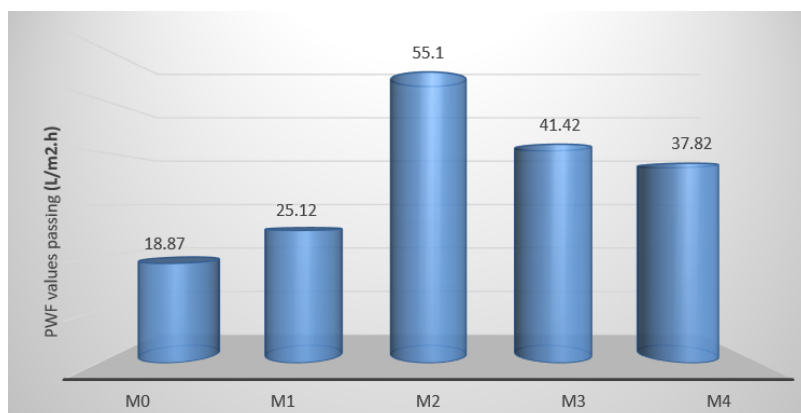


Figure 8. Pure water flux (PWF) of the synthesized membranes (pressure=5 bar and temperature=25±3 °C)

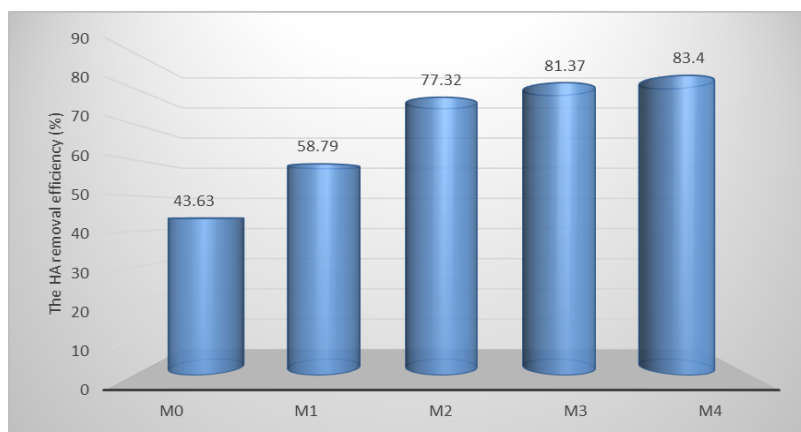


Figure 9. Percentage of HA removal using the synthesized membranes containing different amounts of the ZnO nanoparticle (initial HA concentration=2 mg/L, pressure=5 bar, temperature=25±3°C and time=30 min).

than that of the control membrane. The hydrophilicity of the ZnO NPs can influence the structure of the prepared membranes while affecting the phase separation process speed during the membrane synthesis process in the coagulation bath. This is because the high speed of the phase exchange process in the coagulation bath causes a more porous structure, and a reduction in this speed results in a denser membrane [12, 27]. The improved membrane hydrophilicity can be explained by the symmetry and polarity of the ZnO NPs and the presence of active surface groups that have a higher affinity for water molecules, which can absorb more water molecules to pass through the membrane [28]. Increasing the NPs up to 2%w to the polymer matrix of the membrane fabrication elevates PWF by creating hydrophilic conditions on the membrane surface and increasing the pores of the membrane. The hydrophilicity of the membrane relatively decreases with more than a 2%w increase in NPs, which can be attributed to the accumulation and irregular arrangement of NPs in the membrane structure and thus the reduction of functional groups on the surface. Additionally, adding more than 2%w the ZnO NPs increases its density in the casting solution, thereby increasing the viscosity of the casting solution. This delays the exchange of solvent with non-solvent and thus reduces the porosity and pore size. The HA removal efficiency was 43.63% for pure membranes, and its values for modified membranes with 1, 2, 3, and 4%w ZnO were 58.79, 77.32, 81.37, and 83.4%, respectively. Obviously, all synthesized nanoparticle-containing membranes are more efficient in HA removal from the feed injected into the system than pure PSF membranes, which can suggest the effect of the ZnO NPs on increasing HA removal from aqueous solutions [29, 30]. Furthermore, the membrane rejection efficiency increased by increasing the percentage of NPs used in the synthesized membranes [29]. Hydrophilic functional groups on the modified membrane surface can create more negative zeta potential on the membrane surface by inducing a negative charge and causing further rejection of humic substances by increasing the effects of electrostatic repulsion.

4. Conclusion

The results of contact angle measurement, SEM, ATR-FTIR, and AFM analyses of the synthesized membranes demonstrated that the addition of the ZnO NPs to the PSF polymer matrix could considerably improve the morphology and performance of the membrane. The use of the ZnO NPs in the synthesized membrane matrix could cause the hydrophilicity of the membrane and

the formation of porous nanostructures by accelerating the solvent-non-solvent exchange in the phase change step. The increased porosity and hydrophilicity mean an increase in the PWF. The ZnO NPs also increase the removal efficiency by changing the morphology and affecting the pore structure.

Ethical Considerations

Compliance with ethical guidelines

There was no ethical consideration to be considered in this research.

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Authors' contributions

Conceptualization, study design, and literature review, structuring, data analysis and interpretation, manuscript preparation, and edition: Reza Rezaee, Reza Rahimpour, Afshin Maleki and Ali Jafari; Study design, review, editing, and project administration: Mahdi Safari, Behzad Shahmoradi and Seyyed Abbas Mousavi.

Conflict of interest

The authors declared no conflict of interest.

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