Mansoura Engineering Journal

Volume 49 | Issue 1 Article 2

October 2023

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ORIGINAL STUDY

Novel Composite Membrane(Polyethersulfone/Clay/Zeolite) for Ammonium and Sulfide Ions Removal from Sour Water

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Abstract

The viability of human life depends heavily on the accessibility of clean water, not only for personal use but also to support various industrial, agricultural, and industrial-related activities. Composite membranes were prepared using polyethersulfone (PES) with zeolite and clay. The membranes were fully characterized. Membrane performances were tested using sodium sulfide with a concentration of 0.365 g/l and ammonium chloride with a concentration of 0.148 g/l to study the effect of prepared composite membranes on the treatment of sour water. The membrane's mechanical properties were tested and M3 (20% PES, 2% Zeolite, 78% Dimethylacetamide (DMAc)) has the greatest tensile strength of 64 MPa with an elongation of 31.7%. M3 provides good membrane performance where the removal percentage of sodium sulfide reached 80.6% and the permeate flux reached 231.2 LMH (l/m^2/h). Also, M3 exhibits good results in ammonium chloride removal where the percentage of removal attained 75.7% and permeate flux reached 92.5 LMH. The results indicate that the addition of Zeolite in the polymeric mixture enhances the dense top layer of the membrane, while the addition of clay enhances the membrane hydrophilicity.

Keywords: Ammonium ion, Composite membrane, Nano-zeolite, Polyethersulfone, Sour water, Sulfide ion

1. Introduction

Wastewater from atmospheric and vacuum crude columns at refineries is known as sour water. Before sour water can be used elsewhere in the plant, hydrogen sulfide and ammonia must be removed from the mixture. Sending the sour water from the process to a stripping tower where heat is delivered in the form of steam removes these components. The heat causes the water's ammonia and hydrogen sulfide to escape from the top of the tower (Mestre-Escudero et al., 2020).

Desalination and water treatment frequently employ membrane-based techniques. The most common membrane technologies are reverse osmosis (RO), microfiltration (MF), ultrafiltration (UF), and nanofiltration (NF). To increase the effectiveness of these procedures, improvements must be made to the rejection percentage, antifouling qualities, and permeate flux. Surface chemistry and membrane shape are two factors that can influence how well a membrane performs (Baghbanzadeh et al., 2016). The hydrophobic surface is more susceptible to fouling than the hydrophilic

Received 24 July 2023; revised 6 September 2023; accepted 11 September 2023.
Available online 25 October 2023

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https://doi.org/10.58491/2735-4202.3117

surface because of its fouling resistance that can be reversed (Chen et al., 2015). There are numerous methods for treating the membrane surface, including coating, grafting, and mixing (Kang and Cao, 2014; Liu et al., 2011; Ochoa et al., 2003; Boributh et al., 2009). The most straightforward and affordable strategy is to change the hydrophilic properties of the polymer during membrane synthesis (Lü et al., 2016). Additionally, blending two hydrophobic and hydrophilic polymers can vary the rate of solvent/nonsolvent exchange that occurs in the process of phase inversion during membrane development, which can have an impact on the mechanism of membrane formation, and the structure of the membrane (Baghbanzadeh et al., 2015, 2016). Numerous reactive groups, including sulfhydryl groups, hydroxyl groups, and amine groups can have an impact on the membrane structure during the production processes of various polymers. Under simple conditions, the reactive groups can be employed as an altering agent to produce functional groups (Kubota et al., 2015; Hermanson et al., 1992; Liu et al., 2020).

Zeolites are inorganic, microporous aluminosilicates with significant gas separation potential due to their well-defined pore apertures and molecular sieving properties. The size-selective property of zeolite allows for the selective separation of smaller gas molecules from larger gas molecules (Barsema et al., 2003; Al-Akwaa et al., 2021). Carbon nanotubes, clays, and metal nanoparticles are the most common inorganic materials used (Hou et al., 2016; Slater and Cooper, 2015). Incorporating clays would result in good interaction at the polymer and filler interfaces due to the organo-modification of the silicate layers. Clays are compatible with the majority of thermoplastic polymers that have hydrophobic properties (Rafiq et al., 2011; Herrera-Alonso et al., 2009; Villaluenga et al., 2007; Anadao et al., 2013).

Several methods can be used to remove hydrogen sulfide and ammonia from wastewater such as Modified active carbons (Lupascu et al., 2006). Combination system of trickling biofilter (BTF) and biofilter (BF) filled with ceramic packing materials can used to remove H_2S from sour water (Fasihi et al., 2020).

The novelty of this work is the preparation of polyethersulfone (PES) blended with Zeolites and clays to enhance the membrane performance for sour water separation. In this work, zeolite, and clay were added in the percentage to the polymeric solution of PES to perform membrane performance. The membranes were characterized and the performance was tested to get the optimum membrane for sour water treatment.

2. Experiments

2.1. Materials

BASF Germany Company supplied PES (Ultrason 6020). The used solvent was Dimethylacetamide (DMAc) which was obtained from Sigma Aldrich Company. Zeolite was purchased from Alfa Asser Company. Clay was obtained from ceramic companies. Sodium Sulfide and Ammonia were purchased from Fluka Company. The clay and zeolite X-ray fluorescence (XRF) analysis is illustrated in Table 1.

2.2. Preparation of membranes

An immersion precipitation process was used to create mixed matrix PES membranes with additives of clay and nano-zeolite to improve the performance. The polymer PES was dissolved in DMAc as a solvent, then the percentage of nano-zeolite and clay were added to prepare four different composite membrane (M1, M2, M3, and M4) as polymeric solutions that its composition described in Table 2. The stirring time was acted upon for 24 h. The membrane solution was cast on nonwoven support using a film applicator to apply a wet membrane thickness of 200 μm. Then it was immersed in a water coagulation bath as shown in Fig. 1.

Table 1. XRF analysis for nano-powder of Zeolite and clay.

Compounds	Clay	Zeolite
SiO_2	56.37	41.58
TiO_2	0.58	0.12
Al_2O_3	14.42	21.81
Fe_2O_3	3.99	0.94
MgO	1.81	1.50
CaO	2.46	0.32
Na_2O	0.41	11.10
K_2O	0.58	0.17
P_2O_5	0.21	0.06
LOI	17.13	22.17
MnO	0.051	0.034
NiO	0.005	0.005
ZnO	0.009	0.006
SrO	0.020	0.006
ZrO_2	0.032	0.011
CuO	0.006	–
V_2O_5	0.026	–
Co_3O_4	0.026	–
PbO	0.005	–

Table 2. Polymeric solution composition for prepared membranes.

Membrane symbol	Composition (Weight %)			
	PES %	Zeolite %	Clay %	DMAc %
M1	20	0	0	80
M2	20	0	2	78
M3	20	2	0	78
M4	20	2	2	76

2.3. Membrane characterization

2.3.1. Morphology

The morphology of produced membranes was displayed using scanning electron microscope (SEM). To increase electrical conductivity, gold was applied to the membrane sample surfaces. The pores of microfiltration membranes were visible due to scanning with a JEOL 5410 SEM of the top surface of the membranes.

2.3.2. Mechanical properties

To determine the impact of the blending percentage on the mechanical properties of the manufactured blend membranes, this study was conducted. H5KS Tinus Olsen apparatus was used to estimate the membranes' tensile strength and elongation.

2.3.3. Porosity measurements

Most materials' membrane porosity and air permeability were determined by a densimeter apparatus. By obtaining a sample of the membrane covering a 25 cm^2 area, the gadget was operated. The apparatus was run on compressed air. The following equations were utilized to determine the porosity and air permeability (Monticelli et al., 2007):

$$P = \frac{135.5}{t} \quad (1)$$

The porosity (Ø) can be calculated based on the following equation:

$$\varnothing = \frac{PC}{r^2} \quad (2)$$

That, the permeability of air ml/(cm^2.s.psi) expressed by P, the device circular ring radius expressed by r; where, r^2 = 6.25 cm^2, constant (equals 2 for the circular device rings) denotes by C, and Ø is the membrane porosity.

2.3.4. Membrane contact angle

The hydrophilic properties and the ability of the membrane surface to be moist are regulated by the contact angle. The contact angle was assessed using a compact video microscope (CVM). Membrane by average drop volume, the contact time was 10 s of 10 μl and each value was calculated as the average of ten repeating measurements. The testing procedure is based on ASTM D724-99 standard techniques for the surface wet ability of paper and for corona-

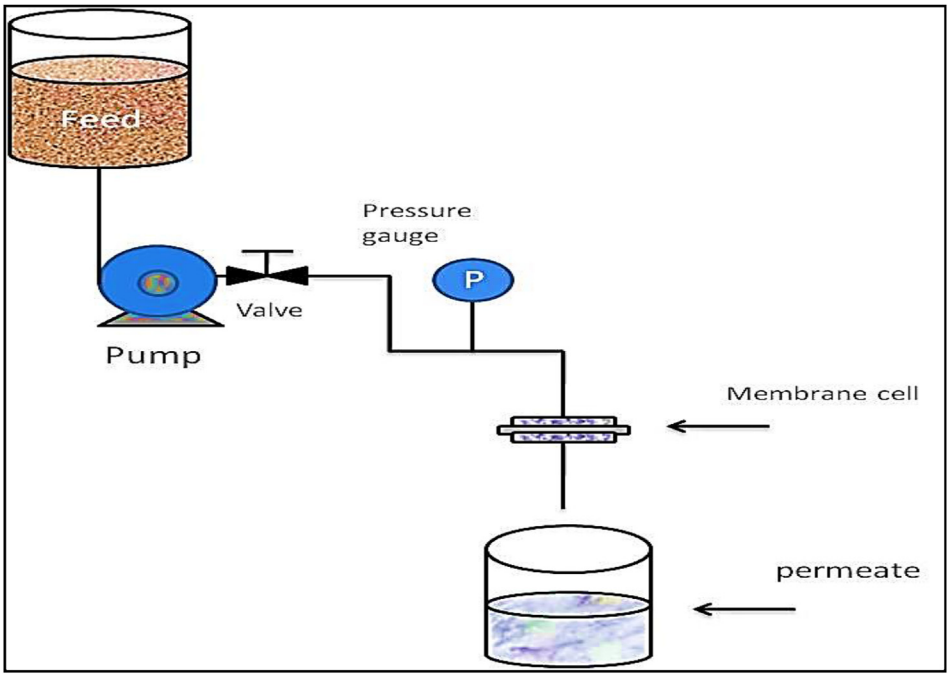

Fig. 1. A diagram of the experimental laboratory setup for membrane testing.

treated polymer films using measurements of the water contact angle process based on ASTM D5946-96 (Castejón et al., 2018).

2.4. Membrane performance

The effectiveness of the fabricated membranes was evaluated using a reservoir and its auxiliaries and a membrane unit comprising the dead-end mode of a laboratory membrane testing cell under a variable pressure (2–4 bar) with the concentration of 0.36 g/l of sodium sulfide and 0.148 g/l of ammonium chloride. The permeate flux and the percentage of separation were calculated based on equations (3) and (4) (El-Gendi et al., 2017; Abdallah et al., 2018):

$$J(W) = \frac{Q}{\Delta T * A} \tag{3}$$

where; the permeate volume (l) is denoted by Q, the effective area of a membrane (m^2) is expressed by A and ΔT is the permeation time (h).

$$S = \left(1 - \frac{Op}{Of}\right) * 100 \tag{4}$$

where O_P is the concentration of the oil in permeate and O_f is the concentration of the oil in feed.

3. Results and discussions

3.1. Characterization of membrane

3.1.1. Morphology

PES blend membranes with clay and zeolite were successfully fabricated through the wet phase inversion steps using DMAc as a solvent Fig. 2 indicates the SEM images of prepared membranes. Fig. 2a shows a cross-sectional image of M1 of a spongy structure and (Fig. 2b) indicates the surface of a blank membrane which provides pores in the surface. Fig. 2c indicates a cross-section of M2 which exhibits a spongy structure and the top surface indicates a reduction in the size of the pores after the addition of 2% clay as a filler. Fig. 2e and g indicate cross sections of M3 and M4 which exhibit a spongier structure of membranes. While the membrane surfaces indicate a dense top layer due to blending with nano-zeolite and clay (Bilad et al., 2015).

3.1.2. Mechanical properties

Mechanical testing of the membrane samples (M1–M4) was measured. Fig. 3 depicts the mechanical properties of the ready-to-use blend membranes, where the results investigate that the M3 has the highest tensile strength of 64 MPa with an elongation of 31.7%, and M4 gives the greatest elongation of 38.6% with the tensile strength of 57.2 MPa, while M1 has the low tensile strength compared with other membranes. The tensile strength is in the order listed M3 greater than M4 greater than M2 greater than M1, while the elongation is in the order listed M4 greater than M2 greater than M1 greater than M3. As stated by the results; the addition of clay with nano-zeolite enhances the elongation of the membrane by improving the membrane elasticity but the addition of zeolite alone improves the tensile strength of the membrane which was considered an indication of the ability of the membrane to carry pressure.

The mechanical properties of all manufactured membranes were enhanced by using nonwoven support, which also reduced membrane wrinkling and shrinking (Bilad et al., 2015).

3.1.3. Contact angle, air permeability, and membrane porosity

The manufactured blend membranes' overall porosity percentage is shown in Table 3. As more clay and zeolite were added, the porosity of the composite membranes decreased. This means that the use of them lowers the porosity of the membrane's internal structures (Yang et al., 2016). The reduction of porosity was related to delaying the coagulation time because the great viscosity of polymeric solutions causes a lag in demixing time that causes a reduction in the size of the pores when the membrane is formed (Yang et al., 2016; Shaban et al., 2015).

Measurements of water contact angles were used to determine the hydrophobicity of prepared blend membranes. Table 3 shows the blend membranes' water contact angles and membrane wetness with various nanoparticle additions. The lowest hydrophobicity of the membranes is reflected in the bare PES membrane M1, which has the maximum contact angle (81.6°). In the case of prepared blend membranes (with 2% clay), the hydrophilicity improved by a reduction in contact angle to 59.6°. The addition of zeolite increases the contact angle, which means it improves htdrophocoitcy compared with the addition of clay. The addition of clay with zeolite reduces the contact angle again from 69.7° to 60.4° and improves hydrophilicity again.

As demonstrated in Table 3, decreasing porosity relates to decreasing air permeability since air permeability, not hydrophobicity like water permeability, is an indicator of membrane porosity.

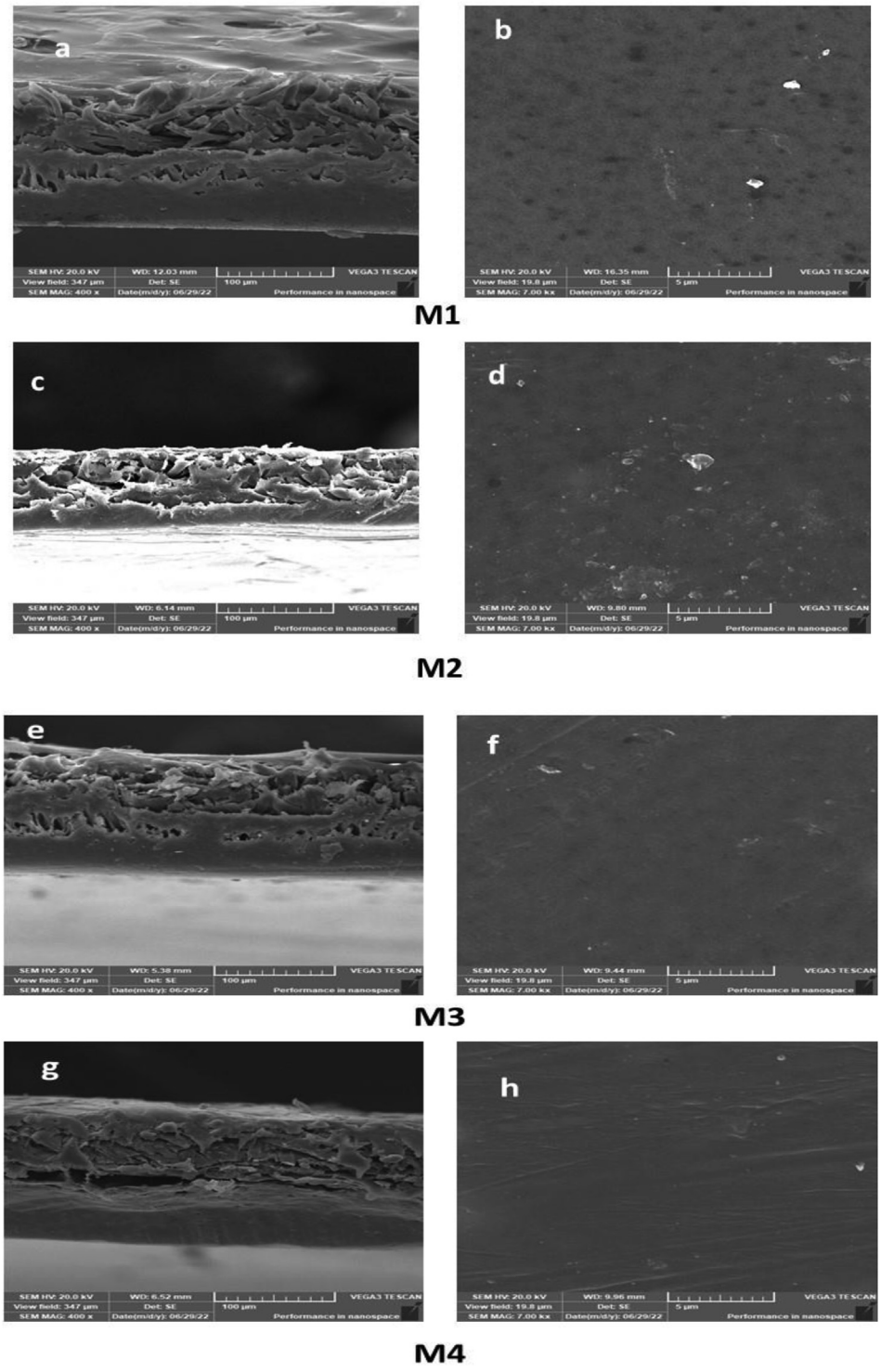

Fig. 2. Scanning electron microscope of the prepared membranes.

3.2. Membrane performance

3.2.1. Removal of sodium sulphide

The prepared membranes were tested on the separation of sodium sulphide at a concentration of 0.365 g/l. Fig. 4 indicates the results indicate that M3 provides the highest separation and high permeate flux because this membrane gains two merit-dense selective layers due to the addition of zeolite and hydrophilic surface nature. The separation

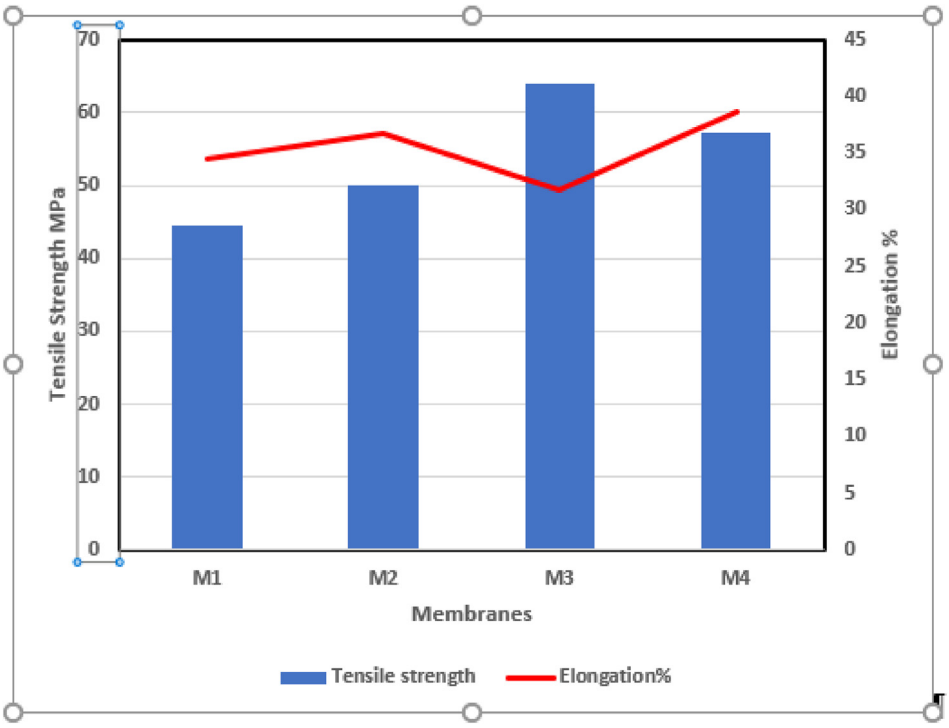

Fig. 3. Mechanical properties of the prepared membranes.

Table 3. Porosity and contact angle of prepared membranes.

Symbol	Nano%	Porosity %	Air permeability $cm^3/cm^2.s$	Contact angle	Membrane wettability
M1	0	58.4	2.7	81.6°	
M2	Clay 2%	38	1.23	59.6°	
M3	Zeolite 2%	14.7	0.543	69.7°	
M4	Clay + Zeolite 4%	16.4	0.764	60.4°	

percentage reached 80.6% with a permeate flux of 231.2 LMH (Lm^2/h). M2 indicates the highest permeate flux due to improvement of surface hydrophobicity after using 2 wt% clay in the polymeric mixture but the separation percentage was lower than M3. The addition of 2 wt.% clay and 2 wt.% Zeolite in M4 reduced the permeate flux and separation percentage due to a reduction in the thickness of the dense layer and an increase in porosity to 16.4% compared with 14.7% for M3. That means the addition of clay reduces the dense layer thickness and increases the porosity.

3.2.2. Removal of ammonium chloride

The prepared membranes were tested on the separation of Ammonium chloride at a concentration of 0.148 g/l. Fig. 5 indicates the results indicate that M3 provides the highest separation but the lowest permeate flux due to dense selective layers which can reduce the flux. The separation percentage reached 75.7% with permeate flux of 92.5 LMH. M2 indicates the highest permeate flux due to the hydrophilic surface after using 2 wt.% clay in the polymeric mixture while the separation percentage reached 38.8%. M4 provides permeate flux

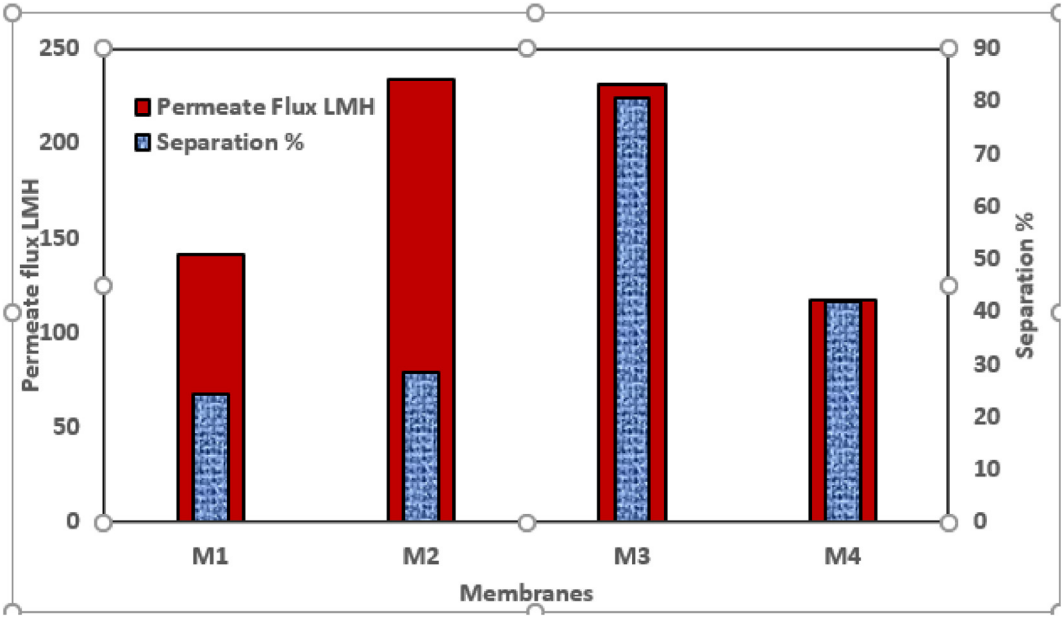

Fig. 4. Membranes performance in sodium sulfide removal.

of 115.3 LMH with a separation percentage of 69.6% due to improvement in the hydrophilic nature of the surface and the existence of a dense top layer although it is less in thickness compared with M3. According to the literature, Darcy's law established that the feed solution viscosity typically affected how much permeate porous membranes could pass through. In contrast, the pore size of the membrane or porosity of the membrane matrix can influence the permeate flux based on the narrow pore size, or other physical factors like molar volume and surface tension can reduce the membrane flux (Dekel et al., 2000; Shukla and Cheryan, 2002).

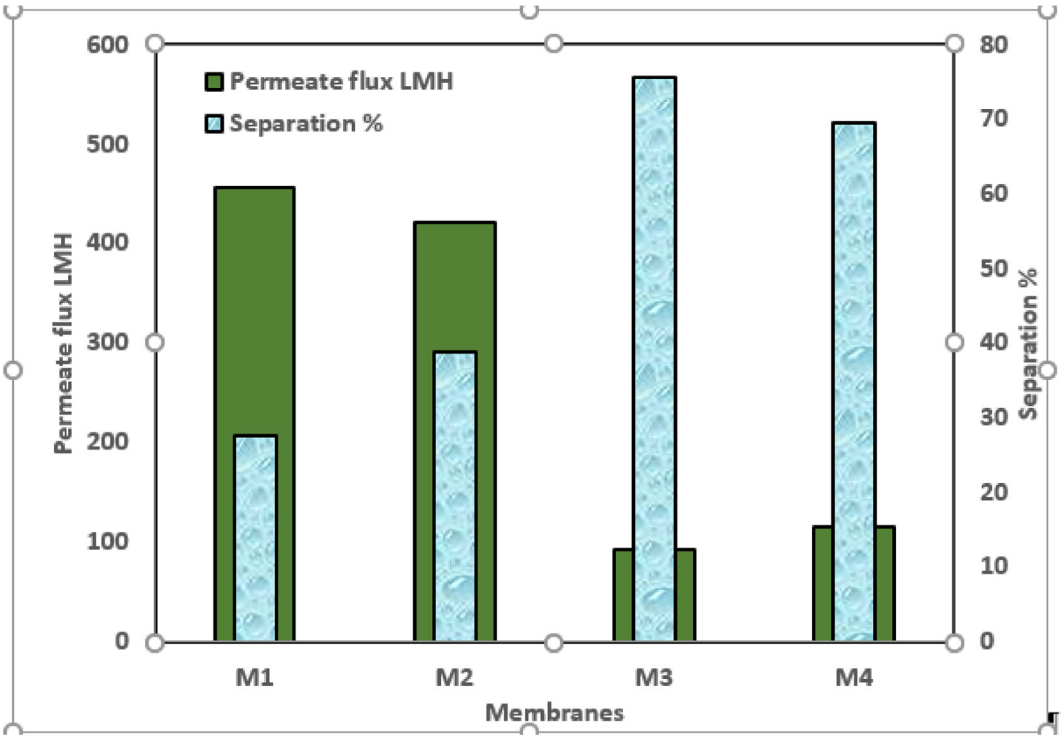

Fig. 5. Membranes performance in ammonium chloride removal.

Table 4. Comparison with other types of membranes.

Type of membrane	Separation %	The type of ions removed	Reference
Liquid membrane	98% in 15 s extraction time	Ammonium	Sïmsek and Altas, 2022
Membrane Biofilm Reactor	>97.0%	Ammonium, dissolved methane, and sulfide	Chen et al. (2016)
M3 (20% PES, 2% Zeolite)	75.7%	Ammonium	Current work
	80.6%	Sulfide	
M4 (20% PES, 2% Zeolite, 2% clay)	69.6%	Ammonium	Current work
	41.9%	Sulfide	

The different compositions of membranes used investigate good results for the removal of ammonium and sulfide ions compared with other works as shown in the following Table 4.

4. Conclusion

Composite membranes were prepared using PES, clay, and zeolite. The membranes were examined by SEM, Mechanical testing, contact angle determination, and membrane performance. The mechanical testing indicates M3 provides the best mechanical properties, where the tensile strength is in the subsequent order M3 greater than M4 greater than M2 greater than M1, while the elongation is in the order listed M4 greater than M2 greater than M1 greater than M3. So, the addition of clay with nano-zeolite improves the elongation of the membrane by improving the membrane elasticity but the addition of zeolite alone enhances the tensile strength of the membrane which was considered an indication of the ability of the membrane to carry the pressure. Also, the addition of zeolite increases the contact angle, which means it improves htdrophocoitcy compared with the addition of clay. So, the combination of clay with zeolite reduces the contact angle again from 69.7° to 60.4°.M3 gives the highest separation and great permeate flux due to dense selective layers and hydrophilic surface nature. The separation percentage reached 80.6% with a permeate flux of 231.2 LMH for sodium sulfide. M3 provides the highest separation but lowest permeate flux for ammonium chloride where the separation percentage reached 75.7% with a permeate flux of 92.5 LMH.

Author contributions

Conceptualization: M S, H A, M A F, A B E, M M. Methodology: M S, H A, M A F, A B E, M M. Formal Analysis: M S, H A, M A F, A B E, M M. Data Curation: M S, H A, M A F, A B E, M M. Original Draft Preparation: M S, H A, M A F, A B E, M M. Writing Review and Editing: M S, H A, M A F, A B E, M M. Supervision: M M, M A F, I I I.

All authors discussed the results and contributed to the final manuscript.

Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The authors declare no conflict of interest.

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