

Polysulfone/Silver-catecholamine (PSf/Ag-CA) mixed-matrix hybrid membrane for the removal of oil-in-water emulsions

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ABSTRACT

Membrane technology is recognized as a reliable and efficient method for the treatment of oily wastewater, particularly in the removal of oil-in-water emulsions with droplet sizes below 20 μm . However, the fouling problem remains one of the major challenges for polymeric membranes. This study investigates the impact of incorporating silver-catecholamine nanoparticles on the performance of Polysulfone (PSf)/Silver-Catecholamine (Ag-CA) mixed-matrix hybrid membrane for oil-in-water emulsions removal. The Ag-CA nanoparticles were synthesized by mixing tannic acid and tetraethylenepentamine into a tris-HCl solution, then incorporated into the membrane dope solution. The PSf/Ag-6C1A hybrid membranes were cast using an automatic membrane casting machine via a phase inversion process, and then the permeability and separation performance of the membranes were measured using a membrane test rig to observe the flux, removal efficiency, and flux recovery ratio of the membranes. Based on the experiment, the PSf/Ag-6C1A hybrid membrane has shown increased pure water flux and high removal efficiency, as well as a flux recovery ratio compared to the pristine PSf membrane. It is shown that the silver-catecholamine nanoparticles can improve the antifouling properties of the pristine PSf membranes. The PSf/Ag-6C1A hybrid membrane exhibits enhanced surface properties with a water contact angle of 76° and an underwater oil contact angle of 130.7°. These characteristics

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contributed to its higher water flux (14.01 LMH), excellent removal efficiency (99.79%), and flux recovery ratio of 95.71%.

1. INTRODUCTION

Oily wastewater is one of the pollutants that contributes to the water pollution problem. The removal of emulsions and surfactants is particularly challenging due to the presence of diverse cationic and anionic surfactants [1], the small size of oil droplets, which are typically less than 20 μm [1], and the enhanced stability caused by components such as asphaltenes, resins, mineral salts, and clay particles acting as natural emulsifiers [2]. Hence, advanced treatment technologies such as membrane filtration are recommended to meet the regulation of industrial effluent discharge standards. Membrane technology is one of the most efficient methods in treating oily wastewater, as it is effective, simple, and economically viable, and can get rid of the smallest oil droplets ($< 20 \mu\text{m}$) in the oily wastewater. Membrane technology also involves a relatively low energy cost [3], minimal membrane fouling [4], low operating pressure [5], and low operating temperature. Moreover, this method produces highly pure water flux that meets high industrial quality standards, compared to other conventional methods [6].

Recently, mixed-matrix hybrid membranes have gained significant attention due to their ability to remove pollutants and low fouling properties. A mixed-matrix hybrid membrane is prepared by mixing an inorganic filler into a polymer matrix using the blending method [7]. Inorganic materials such as silver [8], zinc oxide [9], titanium dioxide [10], and iron oxide [11] have been used in polymeric membranes to enhance the membrane hydrophilicity. Metal-based antimicrobial particles are commonly used as fillers in polymeric membranes to prevent fouling in membrane separation. These metal-based nanoparticles can reduce foulant adsorption and adhesion to pores and surfaces on the membrane or actively destroy cell structures to prevent fouling [12]. Silver (Ag) ions have been proven beneficial in decreasing membrane biofouling by disrupting cell membranes. Because of its strong affinity for water molecules, Ag tends to improve membrane hydrophilicity, which causes modified membranes to have significantly higher permeate fluxes than unmodified or bare membranes [13].

Green synthesis of silver nanoparticles (Ag NPs) is an environmentally friendly method of producing silver nanoparticles using biological agents such as plant extracts [14], which follows green chemistry principles of sustainability, low energy consumption, and minimal toxicity, compared to using chemicals such as sodium borohydride [15]. The green synthesis of Ag NPs, which uses various parts of plants such as leaves, peels, and seeds to produce plant extracts, which are both reducing and stabilizing agents, has various advantages over chemical methods. The advantages include environmental safety, which uses renewable biological ingredients, while minimizing impact on the environment [16], sustainability that is cost-effective because of the use of readily accessible natural resources [14], and biocompatibility, which has strong antibacterial characteristics that are good for both medical and environmental usage [17].

Several compatibilizers have been used in mixed-matrix hybrid membranes, such as amphiphilic polymers [18], miscible polymers such as poly(ethylene glycol) [19] and poly(ethylene-co-vinyl alcohol) [20], and polydopamine (PDA) [21]. Despite many studies showing improved polymer-filler compatibility, copolymers have been limited to low concentrations and low molecular weight additives due to pore blockage that reduces membrane performance. Thus, this study investigates the substitution of conventional compatibilizer with catecholamine synthesized from natural polyphenol and amine as a novel, low-cost alternative compatibilizer exhibiting strong interfacial bonding properties. Catecholamine exhibits great structural similarity, adherent effectiveness, and cost-effectiveness. Catecholamine (CA) is a mixture consisting of catechol, which is synthesized from a mixture of tannic acid (TA) and tetraethylenepentamine (TEPA). Tannic acid, a plant polyphenol with a high concentration of phenolic hydroxyl groups, tends to

oxidize in an alkaline environment and react with amine groups [22], due to its capability to complete or crosslink macromolecules at multi-binding sites [23]. Owing to these properties, catecholamine has been chosen in this research for the development of mixed-matrix hybrid membranes. This modification has been made to improve the permeability, separation efficiency, and antifouling performance of mixed-matrix hybrid membranes for oil-in-water emulsions separation.

2. MATERIALS AND METHODS

2.1 Materials

The chemicals used in this study were analytical grade and used as received. Tannic acid (TA), tetraethylenepentamine (TEPA), hydrochloric acid (37%), and polysulfone (PSf) were purchased from Sigma-Aldrich. Tris(hydroxymethyl)aminomethane (Tris-HCl) was purchased from Merck. Silver nitrate ($\text{Ag}(\text{NH}_3)_2$) was purchased from R&M. Polyvinylpyrrolidone (PVP), N-methyl-pyrrolidone (NMP), and tween 80 (T80) were purchased from Chemiz, Malaysia.

2.2 Preparation of polymeric membrane

Catecholamine (CA) formulation was prepared by dissolving 0.6% w/v tannic acid (TA) and 0.1% w/v tetraethylenepentamine (TEPA) into a 10 mM Tris-HCl buffer solution [23]. The solution was stirred for 24 hours and subsequently centrifuged to collect the CA suspension, which was then dried at 50°C for 24 hours. A total of 0.1g of dried CA was added into 10 mL of deionized water before being added dropwise into 0.02 M silver nitrate (AgNO_3) solution, and the solution was stirred for 0.5 hour [24]. After that, the Ag-CA nanoparticles suspension is collected by centrifugation, which is dried for 24 hours at 50°C.

The dope mixture, consisting of 14.4 wt% of polysulfone (PSf), 83.5 wt% of N-methyl-pyrrolidone (NMP), and 1.6 wt% of polyvinyl pyrrolidone (PVP), was prepared by stirring at 500 rpm and 60°C for 24 hours [25,26]. Afterwards, 0.5 wt% of Ag-6C1A nanoparticles [24] were added to the mixture solution. The mixture was then sonicated until completely dissolved at room temperature to homogenize the solution before casting at room temperature using an automatic membrane casting machine. Next, the membrane film was left to air-dry for 30 seconds before fully immersed in a water coagulation bath to initiate the phase inversion process. The produced hybrid membrane was subsequently dried at room temperature and stored in a desiccator for further use. The preparation of the membrane was carried out, as shown in Fig. 1.

2.3 Characterization of polymeric membrane

The surface morphology and elemental distributions across the cross-section of the hybrid membranes of the pristine PSf and PSf/Ag-6C1A mixed-matrix hybrid membrane were evaluated using scanning electron microscopy (SEM, Quanta FEG 650). Surface hydrophilicity of pristine PSf membrane and PSf/Ag-6C1A mixed-matrix hybrid membrane was measured using a dropmeter contact angle system (Goniometer, AST/VCA-3000S) at a constant water droplet of 2 μL for the aqueous phase. At the same time, the underwater oleophobility was evaluated using n-octane as the model for the oily phase. Identification of functional groups within the hybrid membrane structure was analyzed using Fourier transform infrared spectroscopy (FTIR).

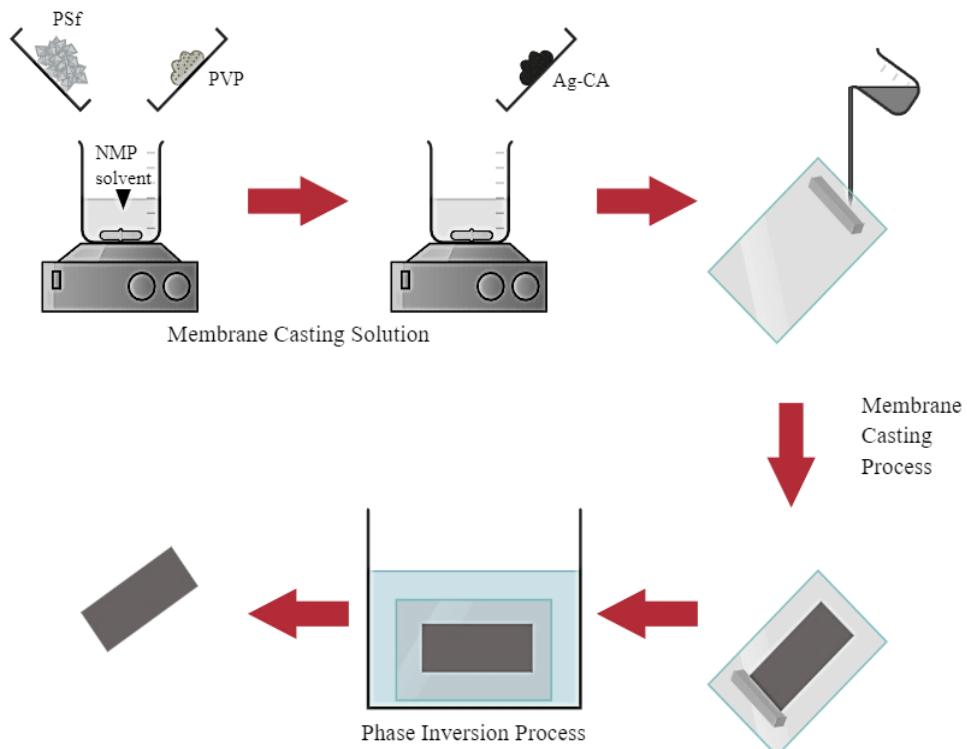


Fig. 1. Preparation of PSf/Ag-6C1A membrane

2.4 Oil-in-water emulsion separation

A 1500 ppm surfactant-stabilized oil-in-water emulsion was prepared by mixing between 80 and diesel at a 1:9 ratio in 5 L of deionized water, followed by emulsification using an electrical mixer at 400 rpm for 45 min [20]. The permeability and separation performance were evaluated using an experimental rig of a cross-flow filtration system, as illustrated in Fig. 2. The water flux (J_w) was determined by pure water filtration after 60 minutes. The feed solution was then replaced with an oil-in-water emulsion for 60 minutes until a stable flux of permeate (J_p) was obtained. The membrane was subsequently cleaned using the flushing method, after which pure water filtration was performed again for 60 minutes to record the pure water flux (J_c) after the cleaning process.

2.5 Membrane performance analysis

The flux is calculated using Eq. (1), where m is the mass of water, ρ_w is water density, A is the area of the membrane, and t is the time interval. The flux recovery ratio (FRR) has been applied to examine the antifouling properties using the following equation, in which J_c is the water flux of the cleaned membrane after each cycle. J_w is the initial pure water flux as shown in Eq. (2). The removal efficiency (RE) of the oil-in-water emulsion was calculated using Eq. (3), where C_p is the concentration of emulsion in permeate. C_f is the concentration of emulsion in feed.

$$J_w (L/m^2 \cdot h) = \frac{\Delta m}{\rho_w A \Delta t} \quad (1)$$

$$FRR (\%) = \frac{J_c}{J_w} \times 100\% \quad (2)$$

$$RE (\%) = \left(1 - \frac{C_p}{C_f} \right) \times 100\% \quad (3)$$

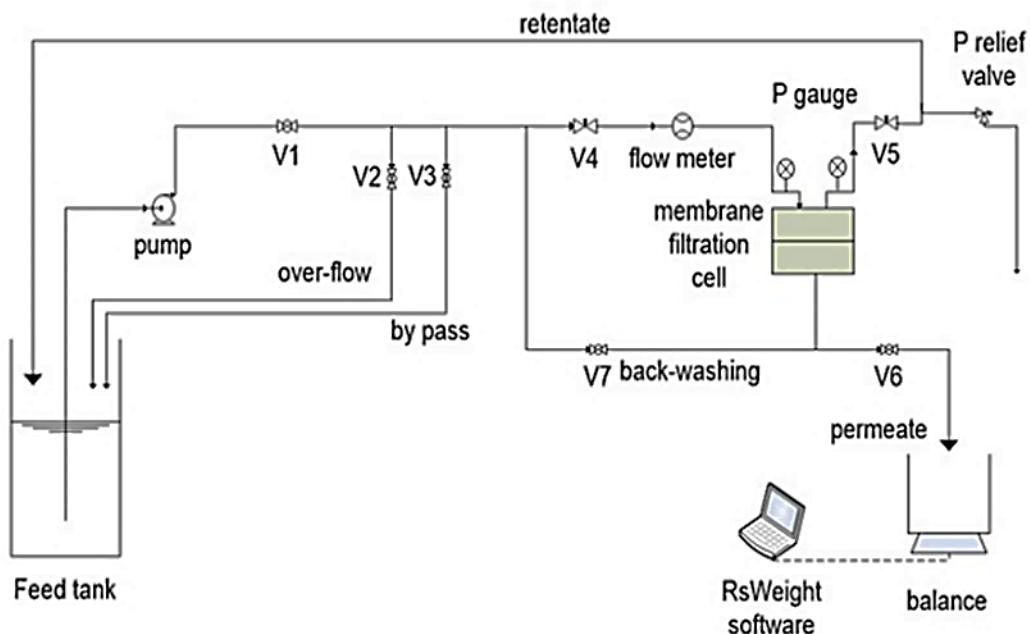


Fig. 2. Cross-flow filtration system [23]

3. RESULTS AND DISCUSSION

3.1 Characterization of mixed-matrix hybrid membrane

Fig. 3 (a-d) shows SEM images of pristine PSf and PSf/Ag-6C1A mixed-matrix hybrid membranes at different magnifications. Both membranes exhibit an asymmetric structure, characterized by a dense top layer and a finger-like porous sublayer. The pore size in the finger-like porous sublayer gradually increased with the addition of Ag-6C1A nanoparticles to the membrane matrix [24]. This increase indicates enhanced hydrophilicity in the PSf/Ag-6C1A mixed-matrix hybrid membrane, attributed to the phenolic hydroxyl groups in natural polyphenols, which promote the hydrophilic properties [27]. During phase inversion, the NMP solvent quickly diffused through the non-solvent coagulation solution, resulting in immediate demixing and forming a porous membrane [8]. In contrast to pristine PSf, which exhibited only C, O, and S

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elements (Fig. 4 (a)), EDX analysis of the PSf/Ag-6C1A mixed-matrix hybrid membrane revealed the presence of additional N and Ag elements, confirming the incorporation of Ag-6C1A (Fig. 4 (b)).

The surface wettability applied for water contact angle and underwater oil contact angle of PSf and PSf/Ag-6C1A mixed-matrix hybrid membrane were measured, as shown in Fig. 5 and Table 1. Water contact angle of the PSf membrane was 90.90° , which shows the nature of its hydrophobicity, while the PSf/Ag-6C1A mixed matrix hybrid membrane showed a lower value of water contact angle, which was 76° , compared to the pristine PSf membrane. The decreased water contact angle value proved that the membrane surface is more hydrophilic than the pristine PSf membrane [26,28,29]. The addition of Ag-6C1A nanohybrids improved the surface hydrophilicity of polysulfone membranes, decreasing water contact angles, indicating improved wettability, which is important in underwater applications [30]. The underwater contact angle for PSf/Ag-6C1A mixed-matrix hybrid membrane was 130.7° , compared to the pristine PSf membrane, which was 109.6° . The larger underwater oil-water contact increased the oleophobility of the membrane; hence, the fouling during performance can be reduced [31].

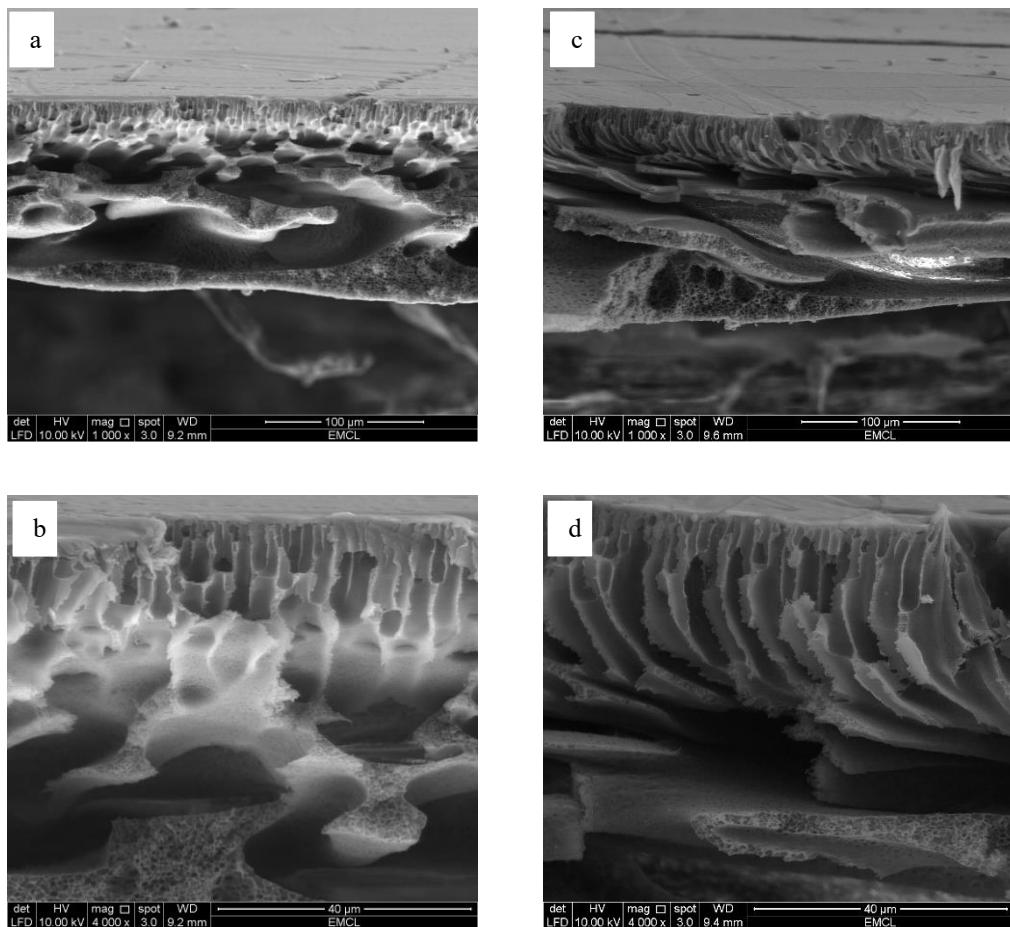


Fig. 3. SEM images of (a, b) pristine PSf and (c, d) PSf/Ag-6C1A mixed-matrix hybrid membranes at (a, c) 1000 \times and (b, d) 4000 \times magnifications

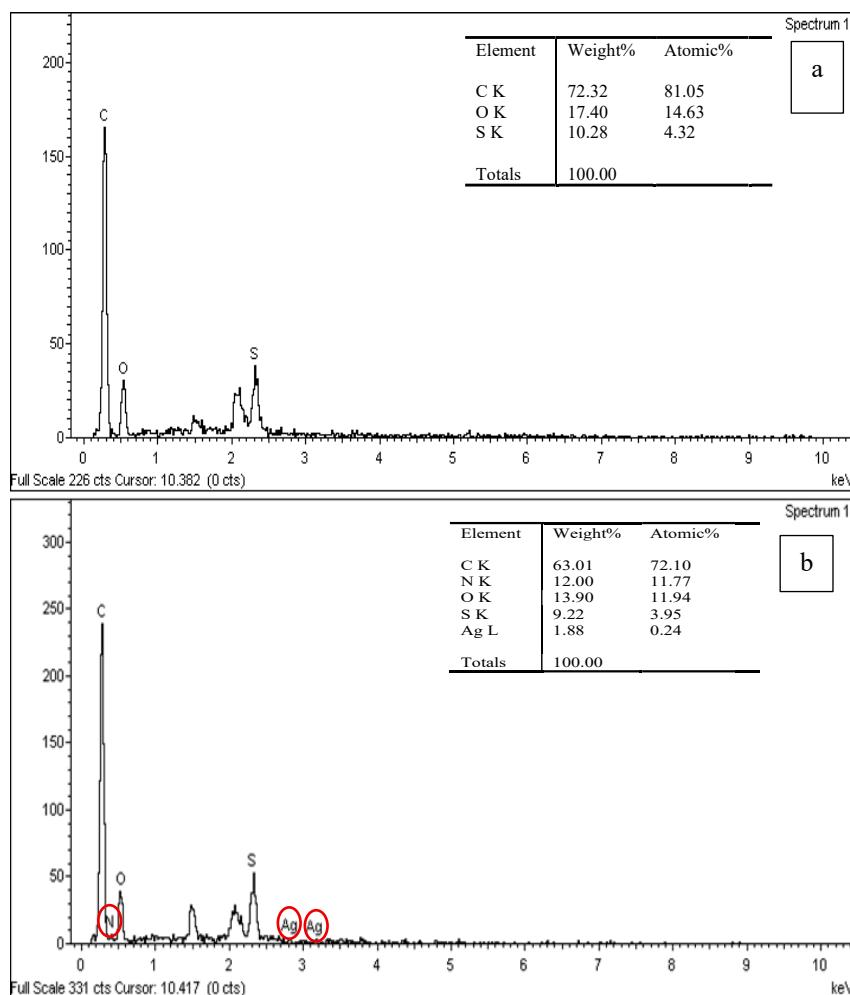


Fig. 4. EDX of pristine (a) PSf and (b) PSf/Ag-6C1A mixed-matrix hybrid membranes

Table 1. Water contact angle and underwater oil contact angle of membranes

Membrane	Water Contact Angle (°)	Underwater Oil Contact Angle (°)
PSf	90.90	109.60
PSf/Ag-6C1A	76.00	130.70

The surface functional group of the membranes was analyzed using FTIR, as shown in Fig. 6. The functional groups found in both membranes are O-H stretch, C-C stretch (in-ring) (aromatic), O=S=O stretch (sulfone), and C-O stretch (alcohols) [29] at the wavenumber of 3410.13, 1293.65, 1488.27, and 1240.70 cm^{-1} , respectively. The stronger vibration can be seen around 3400-3600 and 1500-1600 cm^{-1} for PSf/Ag-6C1A mixed-matrix hybrid membrane, showing the presence of O-H stretch (hydroxyl) of polyphenolic tannic acid and N-H bend (amine) of TEPA, respectively [24]. Thus, it proved the coordination of Ag-CA nanoparticles.

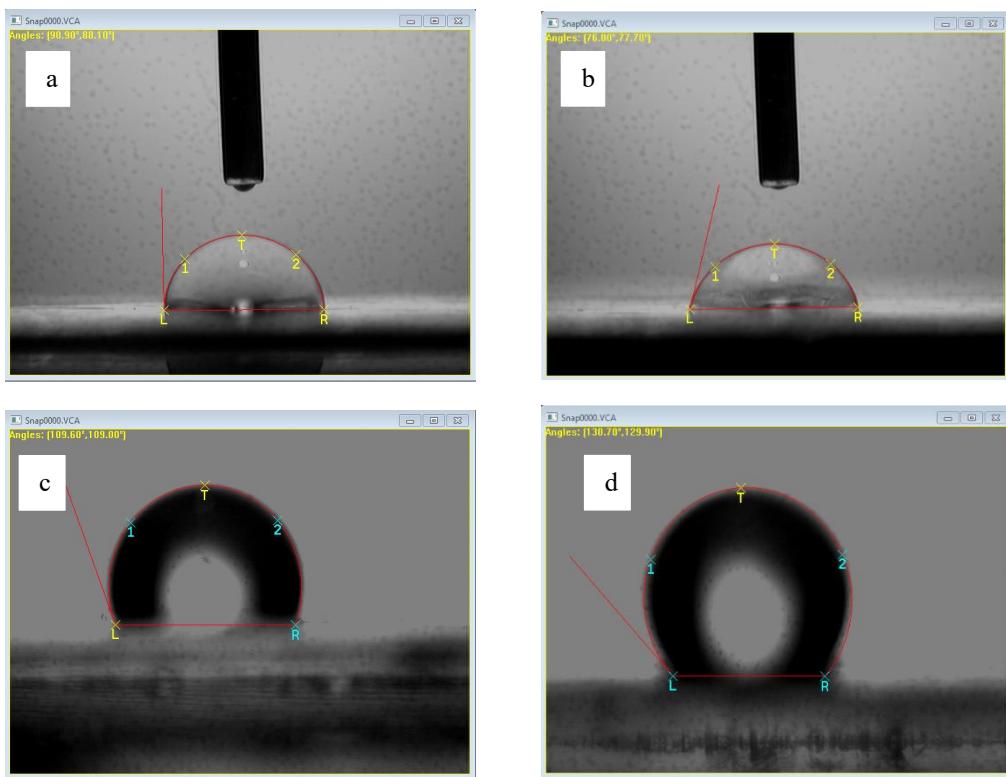


Fig. 5. Water contact angle of (a) PSf, (b) PSf/Ag-6C1A, and underwater oil contact angle of (c) PSf, (d) PSf/Ag-6C1A

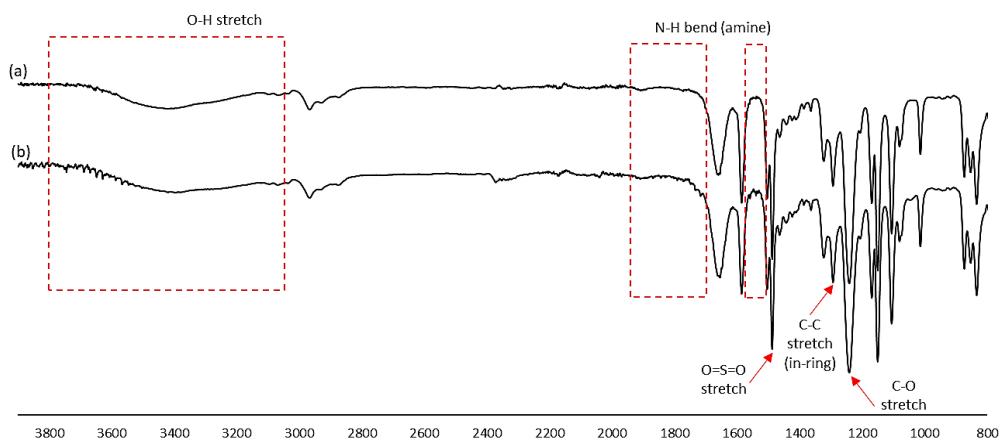


Fig. 6. FTIR spectra of (a) PSf and (b) PSf/Ag-6C1A mixed-matrix hybrid membrane

3.2 Mixed-matrix hybrid membrane performance analysis

The performance of oil-in-water emulsion removal was evaluated for pristine PSf and PSf/Ag-6C1A mixed-matrix hybrid membrane, as shown in Fig. 7 and Fig. 8, respectively. Fig. 7 shows the flux of pristine PSf and PSf/Ag-6C1A mixed-matrix hybrid membrane. Region 1 represents the initial water flux (J_w), Region 2 corresponds to the stable flux of permeate (J_p), and Region 3 indicates the water flux after the cleaning process (J_c). The pristine PSf membrane exhibited an initial water flux of 12.86 LMH, permeate flux of 8.87 LMH, and water flux after the cleaning process of 11.10 LMH. The PSf/Ag-6C1A mixed-matrix hybrid membrane demonstrated improved performance, with an initial water flux of 14.01 LMH, a permeate flux of 9.52 LMH, and a post-cleaning water flux of 13.41 LMH.

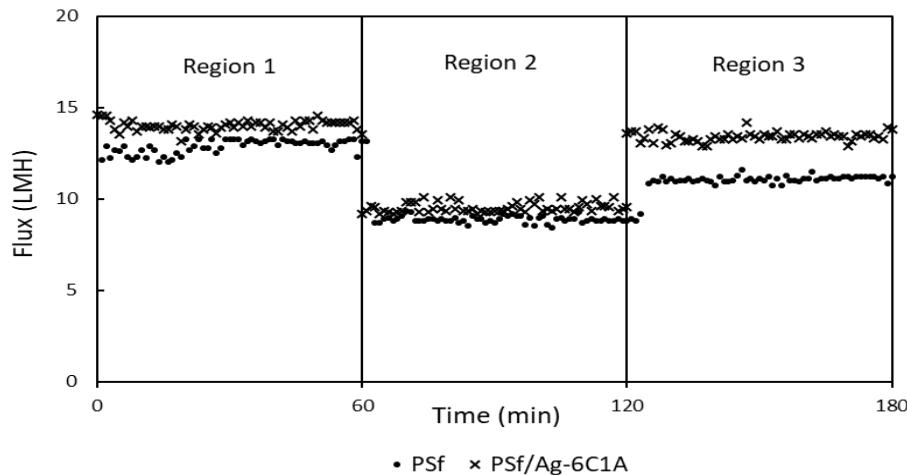


Fig. 7. Flux of PSf and PSf/Ag-6C1A mixed-matrix hybrid membrane

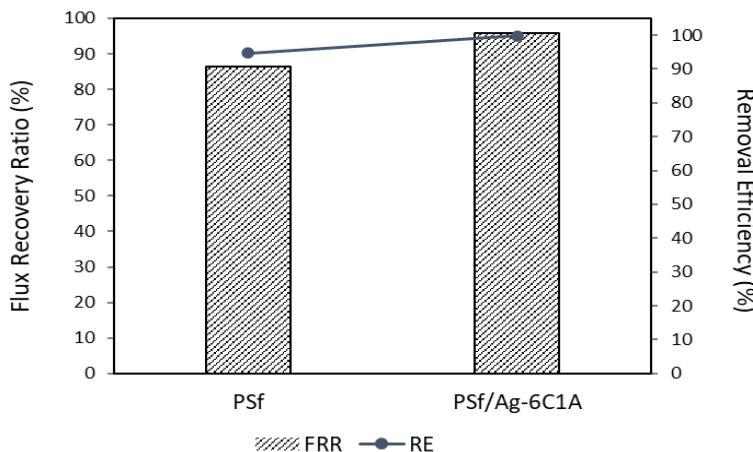


Fig. 8. Removal efficiency and flux recovery ratio of PSf and PSf/Ag-6C1A mixed-matrix hybrid membrane

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Interestingly, PSf/Ag-6C1A mixed-matrix hybrid membrane showed a higher flux recovery ratio (95.71%) for oil-in-water emulsion as compared to the pristine PSf membrane (86.32%). The removal efficiency of oil-in-water emulsion was evaluated using a UV-visible spectrophotometer. The PSf/Ag-6C1A mixed matrix hybrid membrane achieved a high efficiency of 99.79% (permeate concentration: 3 ppm), whereas the pristine PSf membrane showed 94.72% (79 ppm), compared to the initial feed oil-in-water emulsion's concentration of 1500 ppm as shown in Fig. 8. Silver nanoparticles were well bonded and dispersed inside the mixed polymer matrix, giving the hybrid membrane hydrophilicity and underwater oleophobility features [24]. Catecholamine improved chemical functionality with adhesive properties that bind these fillers into a polymeric material, effectively allowing for high-efficiency oil-in-water emulsion separation [22]. Hence, Ag-6C1A nanohybrid blending in membranes significantly improved the antifouling properties of mixed-matrix hybrid membranes [29-30].

4. CONCLUSION

This study successfully developed Polysulfone (PSf)/Silver-Catecholamine (Ag-CA) mixed-matrix hybrid membranes for the treatment of oil-in-water emulsions. The incorporation of Ag-CA nanoparticles, synthesized from natural polyphenols and amines, enhanced membrane hydrophilicity, surface wettability, and pore structure, thereby improving separation performance. Compared with pristine PSf, the modified membranes exhibited higher initial and stable water fluxes, superior flux recovery after cleaning, and markedly improved oil removal efficiency. Specifically, the PSf/Ag-CA membrane achieved a removal efficiency of 99.79% with a permeate concentration of 3 ppm, outperforming the pristine PSf membrane, which recorded 94.72% (79 ppm) under the same conditions. These results demonstrate that catecholamine serves as a low-cost, sustainable, and effective compatibilizer, providing strong interfacial bonding and improved antifouling resistance. Overall, this work highlights the potential of PSf/Ag-CA mixed-matrix membranes as a promising alternative for efficient and sustainable oily wastewater treatment applications.

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6. CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefit, commercial or financial conflicts, and declare the absence of conflicting interests with the funders.

7. AUTHORS' CONTRIBUTIONS

Anis Nazira Alizan: Investigation, lab work, and formal analysis, writing-original draft; **Faraziehan Senusi:** Conceptualization, formal analysis, supervision, writing and reviewing, **Nor Aida Zubir:** Conceptualization, supervision, and reviewing, **Nurulhuda Amri:** Supervision, and validation.

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