

POLYSULFONE-SWCNT COMPOSITE MEMBRANES FOR IRON REMOVAL FROM WATER

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The present study focuses on the evaluation of the ability to retain iron ions from aqueous solution of composite membranes based on polysulfone (PSf) and single-walled carbon nanotube (SWCNT). Different concentrations were employed, and their influence on the structural, thermal, morphological, and filtration features was systematically investigated. By using techniques such as XPS, SEM, and physicochemical analyses, the relationship between membrane composition and metal retention performance was determined. XPS analysis confirmed the successful retention of Fe species on all synthesized membranes. Optimal adsorption performance was determined for the PSf-SWCNT-0.25 sample, which showed the highest Fe content (0.48 at %) after filtration. SEM micrographs revealed that low nanotubes loading led to a denser porous network and interconnected morphology, while higher loading led to aggregation, compact structures, and reduced surface accessibility. The enhanced retention at 0.25 wt.% SWCNT is attributed to enhanced dispersion, increased hydrophilicity, and greater exposure of sulfone and ether groups that function as metal-binding sites.

Keywords: water purification, composite membranes, iron ions retention, carbon nanotubes composites

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1. Introduction

One of the most concerning environmental challenges of modern society consists of ensuring access to uncontaminated water. In recent years, the fast rate

of industrialization, urban growth, and intensive agriculture has caused a significant decrease in water quality, particularly due to the presence of heavy metals, organic pollutants, and microbial contaminants [1-4].

Iron ions (Fe^{2+} and Fe^{3+}) are pollutants of particular concern in water systems that are mainly generated by mining, metallurgical, and electroplating industries. Despite the fact that, normally, iron is an essential trace element with a significant role in the proper functions of organisms, its accumulation in higher concentrations in water can lead to the deposition of oxides in the pipeline streams and filtration systems that can compromise their performance and alter water quality [5-8].

Conventional water treatment techniques such as ion exchange, precipitation, and adsorption are usually the most used to remove contaminants from water; however, these methods are often effective due to several limitations, namely high operational costs, chemical consumption, and the generation of secondary waste [9]. In this context, membrane purification technologies represent an important alternative due to their high efficiency, low energy consumption, and scalability [10].

Polysulfone (PSf) is a thermoplastic polymer that is widely used in membrane fabrication due to its superior properties such as thermal stability, mechanical strength, and chemical resistance. It represents an ideal candidate for membrane synthesis for water treatment due to its film-forming capability and inherent structural rigidity [11, 12], meaning that it can be employed in continuous systems exposed to high temperatures.

However, the hydrophobic nature of this polymer, coming from its aromatic backbone and ether linkages, as well as its limited ability to retain metal ions, imposes the necessity of subsequent surface modification or functionalization to enhance its separation performance. Recent studies have demonstrated that incorporating hydrophilic additives, nanoparticles, or chelating functional groups permeability, selectivity, and fouling resistance of PSf membranes can significantly be improved [13-16]. Tupe et al. [17] have recently used banana leaf and date palm powders as additives for polysulfone membranes and enhanced permeability, porosity, antifouling, and mechanical strength even at a low concentration of 0.15%.

One of the most used reinforcing agents that can also enhance the performance of PSf membranes is carbon nanotubes (CNT) [18, 19]. CNTs offer a remarkably high surface area, high mechanical strength, and versatility, as their chemical structure endows them with the possibility of being functionalized with various chemical groups. Through this approach, their affinity for heavy metal ions can be tailored and significantly improved [20]. Their hollow, tubular structure also contributes to increased porosity and water permeability in composite membranes [21]. When embedded into PSf, carbon nanotubes not only enhance the structural

integrity but also introduce active sites for metal ion adsorption through complex mechanisms such as electrostatic interactions, π - π stacking, and surface complexation [22, 23]. Up to now, PSf has been used alongside carbon nanotubes for the removal of various heavy metal ions such as Pb^{2+} , Cr^{6+} , and Cd^{2+} , reaching a maximum removal efficiency of 96% [24].

The present study focuses on the development and characterization of polysulfone-based membranes for the retention of iron ions from aqueous solutions. The work aims to establish the relationship between membrane composition, structure, and separation efficiency, contributing to the advancement of sustainable membrane technologies for the effective removal of metallic ions from contaminated water.

2. Materials and Methods

2.1 Materials

Polysulfone (PSf) transparent pellets with an average Mw of $\sim 35,000$ determined by LS, average Mn $\sim 16,000$ determined by MO, Carbon nanotube, single-walled (SWCNT), N, N-Dimethylformamide (DMF) ACS reagent, $\geq 99.8\%$, and Iron (II) sulfate heptahydrate, ReagentPlus®, $\geq 99\%$ from Sigma-Aldrich were used as received.

2.2 Methods

Prior to membrane synthesis, 7.5 g of PSf was solubilized in DMF at 60°C for 16 h in order to obtain a solution of 15 wt.%. After complete dissolution, three nanocomposite formulations were prepared by dispersing SWCNT at loadings of 0.25, 0.5, and 1 wt.% (relative to polymer weight) into the PSf solution. Dispersion was achieved by ultrasonication for 15 min in an ice bath in order to prevent thermal degradation.

The membranes were synthesized by using the phase inversion casting method. 5 mL of PSf solution in DMF was poured onto a dry glass plate and evenly distributed on the surface to a standard thickness of $250\ \mu\text{m}$ with the aid of an Elcomer casting machine. The cast film was immediately immersed in a coagulation bath consisting of deionized water at ambient temperature. After complete phase inversion and solidification, the membranes were removed and thoroughly washed with water in order to eliminate residual solvent. Finally, the membranes were dried under vacuum at room temperature. The composition and name of each synthesized membrane are presented in Table 1.

Table 1. Compositions of the membrane casting solutions and sample abbreviations

Nr. Crt.	PSf solution in DMF (mL)	SWCNT (wt.%)	Sample name
1	5	-	PSf

2	5	0.25	PSf-SWCNT-0.25
3	5	0.5	PSf-SWCNT-0.5
4	5	1	PSf-SWCNT-1

2.3 Characterization

X-ray photoelectron spectrometry (XPS) analysis was conducted on a K-Alpha spectrometer with a monochromatic Al K α source (1486.6 eV) working in a vacuum base pressure of 2×10^{-9} mbar. Charging effects were compensated using a flood gun, and binding energy was calibrated by placing the C1s peak at 284.8 eV as an internal standard. Deconvolution of C 1 s peaks was performed by using a smart background algorithm with a convolved Gaussian - Lorentzian ratio. The pass energy for the survey spectra was set at 200 eV, while for the high-resolution C 1 s spectra registration it was 20 eV.

Thermogravimetric analysis (TGA) was performed using a Netzsch TG 209 F1 Libra equipment, from RT to 700 °C under a nitrogen atmosphere with a heating rate of 10 °C/ min.

Differential scanning calorimetry (DSC) curves were recorded on a Netzsch DSC 204 F1 Phoenix instrument. The measurements were conducted at a heating rate of 10 °C/min under a nitrogen atmosphere (20 mL/min flow rate) from RT to 300 °C.

UV-Vis analysis was conducted on a Shimadzu UV-3600 UV-VIS spectrometer equipped with a quartz cell having a light path of 10 mm. Analysis were performed on feed solutions before and after circulation through the membrane at a wavelength of $\lambda \sim 298$ nm.

Contact angle (CA) measurements were performed with the aid of a Drop Shape Analyzer-DSA100 from Krüss Scientific GmbH using water and ethylene glycol as probing liquids at room temperature using the sessile drop method. Measurements were performed by placing a 2 μ L solvent droplet on the sample surface, and the contact angle values were determined using the Young - Laplace equation in Advance software. The surface free energy for each system was calculated in the same software using the Young - Dupré and Fowkes equations, which considered the polar work of adhesion and corresponding surface tensions of the liquids used for analysis.

The equilibrium water content (EWC) of the membranes was evaluated through the gravimetric method. First, the membranes were dried at 60 °C for 24 h and weighed (W_d), then immersed in distilled water for 24 h. The wet weights of the membranes were determined after the excess water on the membranes was removed (W_w). The equilibrium water content of the membranes was calculated with the following equation:

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

where W_w is the wet weight of the membrane (g); W_d is the dry weight of the membrane (g);

Three measurements were performed for each sample, and EWC is reported as the average value.

The overall porosity of the membranes ($P\%$) was determined by employing the gravimetric method [25]. The membranes were weighed in the dry state (w_d) and then were immersed in DI water at room temperature for 24h. Further on, the excess water was removed from the membrane surface, and the wet weight of the samples was measured (w_w). The following equation was used to calculate the membrane porosity:

$$P (\%) = \frac{W_w - W_d}{\rho_{Al} l} \times 100 \quad (2)$$

where W_w is the wet weight of the membrane (g); W_d is the dry weight of the membrane (g); A is the membrane area (cm^2); l is the membrane thickness (cm); and ρ is the density of the water (0.998 g/cm^3).

Three measurements were performed for each sample, and porosity is reported as the average value.

The membrane performance in terms of heavy metal retention was assessed at RT using a dead-end filtration setup. Before performing the experiments, each membrane was washed with 10 mL ethanol and twice with 20 mL of distilled water. The metal ion retention was evaluated using 50 mL of $0.01 \text{ g}\cdot\text{L}^{-1}$ FeSO_4 aqueous solutions as feed solutions, at a transmembrane pressure of 2.0 bar. Each test was performed on three different membranes having the same composition; the permeate was recirculated five times, and the water flux and heavy metal retention values were averaged.

The water flux (Wf) expressed in $\text{L}/\text{m}^2\cdot\text{h}$ and the percentage drug retention (R) were calculated by the following formulas (Eqs. (3) and (4)) where Wf is the water flux ($\text{L}/\text{m}^2\cdot\text{h}$), V is the volume (L) of the feed solution, A is the area (m^2) of the membrane, t is the time (h), R is the drug retention (%), and C_F and C_P are the concentration of drugs in the feed and the permeate, respectively.

$$Wf = \frac{V}{A \cdot t} \quad (3)$$

$$R = \frac{C_F - C_P}{C_F} \times 100 \quad (4)$$

Scanning electron microscopy (SEM) analysis was performed on a Quanta Inspect F microscope (Hillsboro, OR, USA), equipped with an energy-dispersive X-ray spectrophotometer (EDX), with the accelerating voltage set at 30 kV.

3. Results and Discussion

3.1. XPS

The chemical compositions of pure PSf and SWCNT nanocomposite membrane were characterized by XPS analysis, and the corresponding results are presented in Figure 1. Elemental composition results of the pristine PSf and composite membranes showed only minor variations in the relative atomic percentages of C, O, and S (Figure 1). The deconvolution of C1s high-resolution spectra (Figure 1) reveals three secondary peaks associated with C sp² species (at 284.8 eV), C sp³ (at ~285 eV), and C-O species (at ~286.2 eV) [26]. Analyzing the data resulting from the deconvolution of C1s, one can observe that the incorporation of SWCNT slightly increases the contribution of sp³-hybridized carbon (~285 eV) to the overall C1s area, which is characteristic of the end groups located at the edges of the tubular structure of the carbon nanotubes. The O and S ratio from the surface elemental composition is constant for all samples. These results show that while the overall surface chemistry of the PSf membranes remains largely unaltered by SWCNT incorporation, there is a preference of these tubular nanostructures to orient themselves with the terminal groups of the ends towards the extreme outer surface.

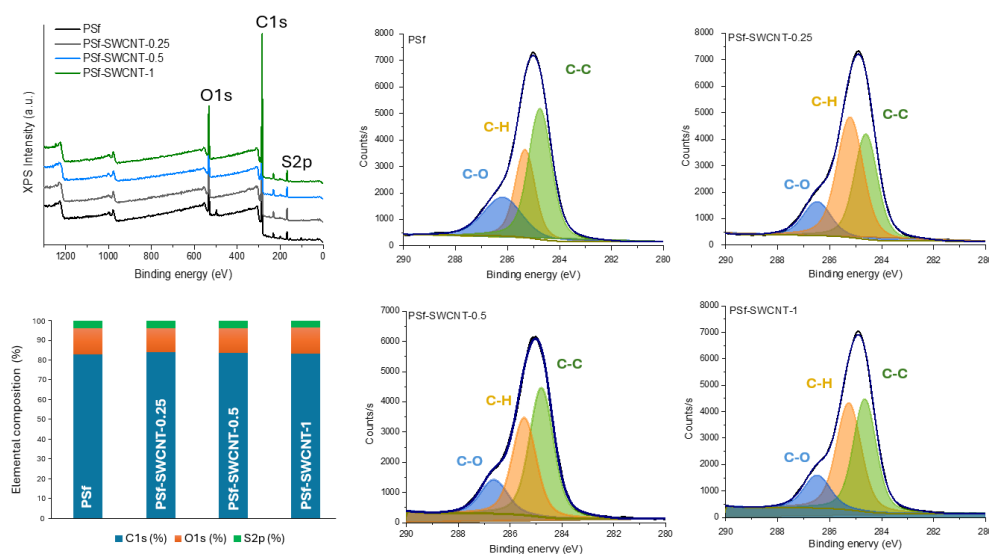


Figure 1. Elemental composition of PSf membrane and its SWCNT nanocomposites before heavy metal retention.

3.2 Thermal properties

The thermal behavior of the samples was investigated using Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA). The results are presented in Figure 2 and Table 2 and provide insights into the thermal stability, phase transitions, and decomposition characteristics of the material.

DSC results show that all samples exhibit a single glass transition temperature (T_g). T_g slightly increases with SWCNT content, indicating restricted mobility of the polymeric chains. This may be a consequence of additional interactions that may take place between the nanotubes and the polymeric matrix [27]. At the same time, the increase in T_g value suggests that SWCNT can also act as nano-reinforcing agents.

Thermal stability of the membranes was evaluated through thermogravimetric analysis, and the corresponding thermograms are presented in Figure 2. All membranes demonstrate high thermal stability with a single degradation step between 450–550 °C, which corresponds to the thermal decomposition of the polysulfone backbone. The overall thermal properties of the membranes were not significantly altered or influenced by the incorporation of SWCNTs. This may suggest that a good dispersion of the nanotubes within the polymer matrix was achieved [28, 29]. However, a slight shift in the onset and maximum degradation temperatures ($T_{d3\%}$ and T_{max}) was observed for the PSf-SWCNT composites compared with neat PSf. This may suggest that the functional groups present on the surface of the nanotube exert a minor catalytic effect on the polymer chain scission process.

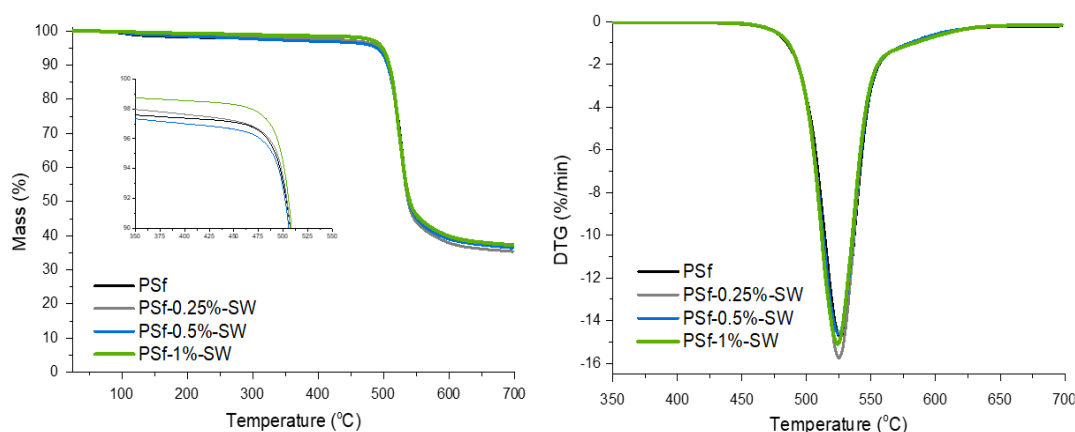


Figure 2. TGA and DTG thermograms for PSf membrane and its SWCNT-based nanocomposites

As expected, the residual mass at 700 °C increased with SWCNT loading, as a consequence of the high thermal stability of the carbonaceous nanostructures and their ability to promote char formation during thermal decomposition. These

results highlight that while the incorporation of SWCNTs slightly modifies the degradation behavior of PSf, it enhances the overall thermal resistance and residual mass of the composite membranes [30, 31].

Table 2. TGA and DSC data for PSf membrane and its SWCNT-based nanocomposites

Sample	T _g (°C) from DSC data	T _{d3%} (°C)	T _{max} (°C) from DTG	Residual mass (%)
PSf	183.6	500.4	526.0	36.53
PSf-SWCNT -0.25	183.9	501.4	525.1	35.35
PSf-SWCNT -0.5	184.8	499.3	524.9	36.38
PSf-SWCNT -1	187.9	503.8	528.3	37.22

3.3 Contact angle measurement

Surface properties were assessed in terms of contact angle measurement (CA) and surface free energy (SFE) and the corresponding results are presented in Figure 3 (a). The neat PSf membrane exhibited a water contact angle of $\sim 95^\circ$ and a relatively high SFE (~ 70 mN/m) [32]. In contrast, with the gradual inclusion of SWCNT, the contact angle shifts to hydrophobic values while SFE sharply decreases to ~ 20 mN/m. This may indicate that the carbonaceous structures tend to orient at the membrane surface during the phase inversion synthesis process, governing the surface properties [33, 34]. These findings correlate with XPS elemental analysis results, which demonstrate that the proportion of polar functional groups in all synthesized membranes is quite similar, and the effect of graphitic carbon at the outer layer is the one that can influence the surface properties.

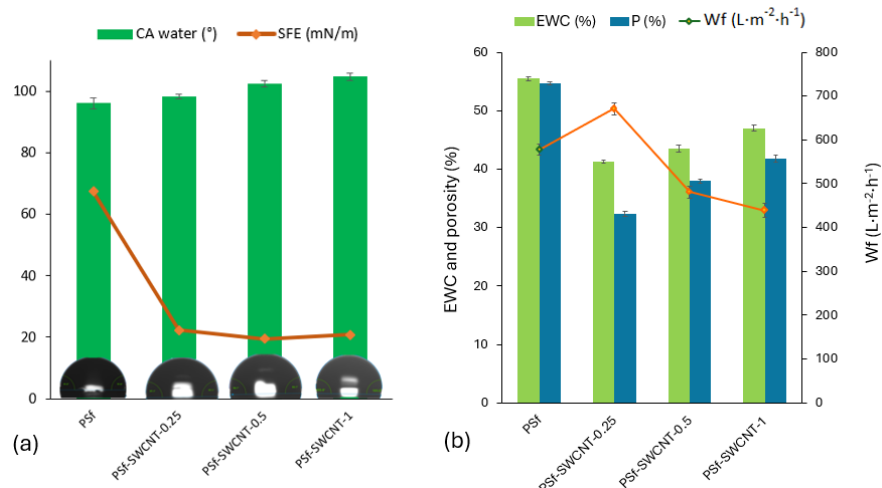


Figure 3. Contact angle (a) and equilibrium water content and porosity (b) of PSf and its SWCNT-based nanocomposite membranes.

The observed decrease in equilibrium water content (EWC) and porosity (P) from Figure 3 (b) with increasing SWCNT concentration may be attributed to a denser membrane structure caused by SWCNT nanofiller and its possible agglomeration. Additionally, SWCNT can alter the phase inversion process, reducing pore formation and water uptake capacity.

In the case of the PSf-SWCNT-0.25 sample, the lowest value for porosity suggests a densification of the membrane matrix caused by interactions between the polymer and carbon nanotubes that restrict solvent-nonsolvent exchange during phase inversion [35]. However, at this SWCNT loading, the water flux reached the highest value, indicating a good dispersion of the carbonaceous structures that may enhance pore interconnectivity and generate preferential pathways for water transport [34, 36].

Higher loadings of SWCNT (0.5-1 wt.%) led to higher values for EWC and porosity but, at the same time, to a decline in water flux, which can be attributed to nanotubes aggregation and increased transport resistance within the membrane structure [28].

3.4 Fe^{2+} and Fe^{3+} ions removal efficiency

The metal ion retention tests revealed a non-linear dependence on SWCNT content (Figure 4). While the neat PSf membrane removed ~38% of Fe^{2+} ions, incorporation of 0.25 wt.% SWCNTs significantly increased retention up to ~70%. This behavior can be assigned to the fact that at lower concentration, SWCNTs achieve a good dispersion within the polymeric matrix. Along with that, more adsorption sites, such as graphitic domains or oxidized functionalities, are exposed, favoring the interaction with the metallic species.

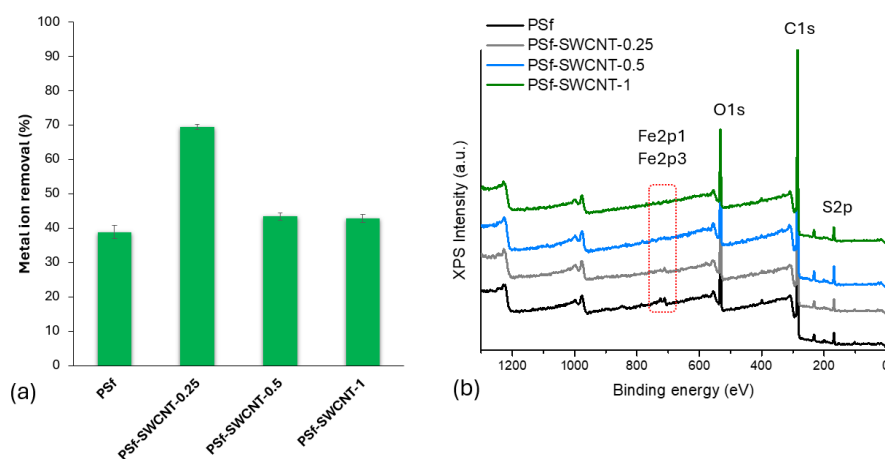


Figure 4. (a) Iron ion removal capacity of PSf and SWCNT nanocomposite membranes and (b) XPS Survey after metal retention.

The increase in SWCNT loading (0.5-1 wt.%) leads to a decrease in retention efficiency, possibly due to the inherent tendency of nanotubes to agglomerate and to the formation of a denser, more hydrophobic surface layer that hinders ion transport and adsorption. These results demonstrate that there is an optimal nanofiller concentration at which adsorption capacity and membrane structure are balanced, while excessive loading decreases performance.

By analyzing the C1S high-resolution spectra (Figure 5) and corresponding data (Table 3) for the membrane samples after Fe2p retention, one can observe a decreased contribution of C sp³ through higher values calculated for the C sp²/C sp³ ratio. Also, the lower contribution of C-O was extracted from the values obtained for the Csp²/C-O ratio. These results provide valuable insights on the mechanism of retention, suggesting that the Fe²⁺/Fe³⁺ species preferentially attach to the SWCNT ends.

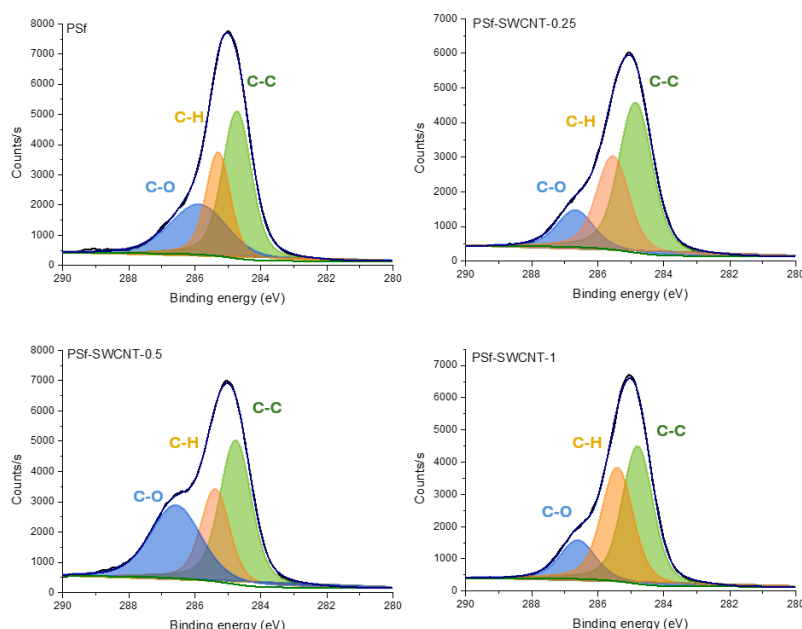


Figure 5. C1s high-resolution spectra for the membrane samples after iron retention

The surface elemental composition of the PSf membrane and its SWCNT nanocomposites after iron ion retention was analyzed by XPS, and the results are summarized in Table 3. The characteristic peaks of C1s, O1s, and S1s correspond to the polysulfone matrix, while additional Fe2p signals confirm the adsorption of iron species on the membrane surface. Among the samples, the PSf-SWCNT-0.25 membrane exhibited the highest Fe atomic content (0.48%), indicating the greatest metal retention. These results correlate with the retention tests that revealed that this sample exhibited the highest efficiency.

At low loadings, the well-dispersed SWCNTs increase the membrane's effective surface area and exposure of sulfone and ether groups, which can behave

as coordination sites for Fe ions. As the nanotubes loading increased to 0.5 and 1 wt.%, the retained Fe content decreased.

This decline is likely due to the aggregation of nanotubes at higher concentrations, which reduces the effective surface area and blocks membrane pores, thereby limiting ion diffusion and adsorption sites. A slight increase in O1s and S1s intensities across the composites indicates that Fe ions may have also interacted with sulfone and oxygenated groups on the membrane surface, forming Fe-O or Fe-S bonds.

Table 3. Elemental composition of PSf membrane and its SWCNT nanocomposites after heavy metal retention

Sample	C1s (%)	O1s (%)	S1s (%)	Cl2p (%)	Si2p (%)	Fe2p1 (%)	Fe2p3 (%)	N1s (%)
PSf	82.09	13.91	2.17	0.36	1.18	0.09	0.21	-
PSf-SWCNT-0.25	78.60	14.27	2.96	0.80	1.11	0.26	0.22	1.79
PSf-SWCNT-0.5	80.37	15.03	3.19	0.55	-	0.21	0.07	0.58
PSf-SWCNT-1	81.11	14.32	3.61	-	-	0.07	0.06	0.84

Based on heavy metal retention tests and XPS results, the retention of $\text{Fe}^{2+}/\text{Fe}^{3+}$ ions by PSf-SWCNT membranes can be ascribed to a combination of electrostatic and coordination interactions with sulfone and oxygenated sites, adsorption onto SWCNT defect and graphitic domains, and partial deposition of iron species [37-39]. At low nanotubes loadings, these mechanisms act synergistically to enhance ion removal, whereas at higher concentrations, the reduced porosity and increased hydrophobicity of the dense carbonaceous-rich layer hinder ion transport and suppress adsorption, leading to diminished retention efficiency.

3.5 SEM

The surface morphology of the pristine PSf and PSf-SWCNT composite membranes is presented in Figure 6.

Analyzing the micrographs, it can be observed that all samples display an asymmetric structure with large and well-defined pores, a structural characteristic that is typical for membranes obtained through the phase inversion process. Along with the inclusion of SWCNT, notable changes can be observed [40]. Therefore, low SWCNT loadings (0.25 wt.%) lead to uniform and interconnected pores. This may also be a consequence of the good and homogeneous dispersion of the nanotubes within the polymeric matrix, which favors the solvent-non-solvent exchange, thus increasing porosity.

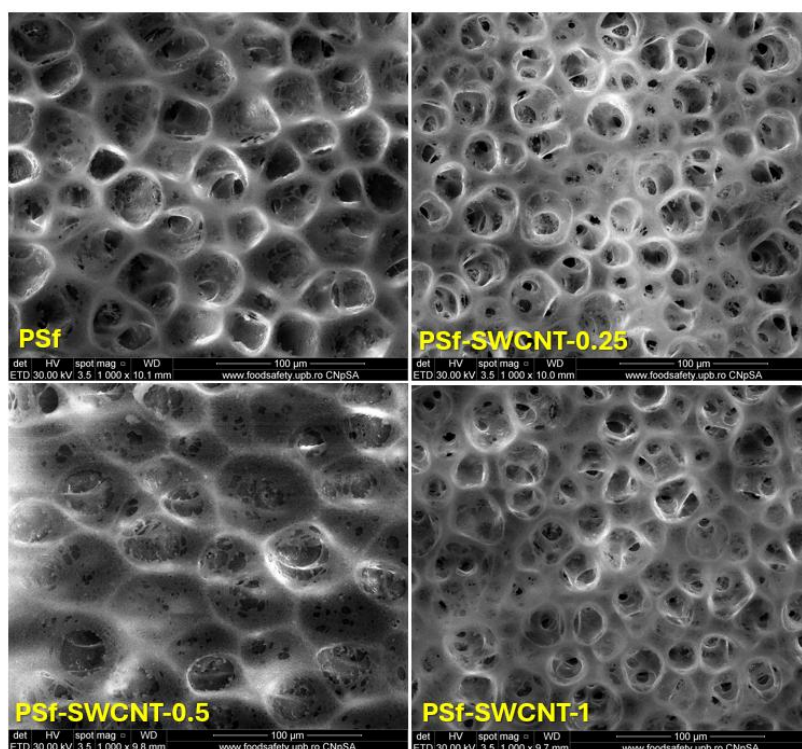


Figure 6. SEM micrographs of PSf and its SWCNT nanocomposites.

Along with the increase in SWCNT concentration, membrane density and pore size progressively increase [41]. This is also a consequence of nanotubes agglomeration, which in this case increases the viscosity of the polymeric solution and thus hinders solvent diffusion during the casting of the membranes. The resulting compact morphology leads to limited mass transport and decreased site accessibility.

These findings are in accordance with the low Fe content determined through XPS analysis after filtration. Therefore, the optimal SWCNT concentration, which provides an adequate morphology that enhances permeability and adsorption performance without altering membrane structure and function, is 0.25 wt.%.

4. Conclusions

In this work, composite membranes based on polysulfone and SWCNT were synthesized and characterized in terms of iron ion retention. Results highlighted the complex role of the reinforcing agent, which significantly impacted not only membrane performance but also thermal stability and morphology. Low SWCNT concentration (0.25 wt.%) favors adsorption and ion removal (~70%),

while at higher concentrations, carbonaceous nanostructures tend to agglomerate and decrease the properties. Along with that, porosity, water uptake, and surface properties decrease.

XPS results after filtration, as well as metal ion retention tests, sustain the increased affinity of iron ions towards the composite membrane with 0.25 wt.% SWCNT. In this case, the retention of $\text{Fe}^{2+}/\text{Fe}^{3+}$ species takes place through multiple electrostatic and coordination interactions with the exposed graphitic domains, oxygenated species, and sulfone moieties.

The overall results demonstrate that carbon nanotubes exert a significant effect at 0.25 wt.% concentration, ensuring optimal water permeability and iron ion removal efficiency.

Author Contributions

Conceptualization, S.I.V. and E.I.P.; methodology, E.I.P., C.M.D., M.O., R.M.O., A.M.P.; validation, E.I.P., C.M.D., M.O., R.M.O., A.M.P.; formal analysis, E.I.P., C.M.D., M.O., R.M.O., A.M.P.; investigation, E.I.P., C.M.D., M.O., R.M.O., A.M.P.; writing-original draft preparation, E.I.P.; writing-review and editing, S.I.V.; supervision, S.I.V. All authors have read and agreed to the published version of the manuscript.

Data Availability Statement

Data supporting the findings of this study are available upon reasonable request from the corresponding author.

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Conflicts of Interest

The authors declare no conflict of interest.

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