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Journal of Applied Engineering Science & Technology

Journal home page: <https://journals.univ-biskra.dz/index.php/jaest><https://doi.org/10.69717/jaest.v5.i2.136>

Study of the Hydrodynamic performance Membranes based on the Polysulfone and doped Polyaniline: Conception and application

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ABSTRACT

Polysulfone/polyaniline ultrafiltration (PSF/PANI-UF) membranes were prepared via the phase inversion method using six examples of the polyaniline (PANI) synthesized in the presence of doped by functionalized agent dopants by direct process. To enhance the hydrodynamic performances of these prepared membranes, the conducting polyaniline (PANIs) has been added at different mass compositions (0.0, 0.2, 0.5 and 1.0%). The performance surface hydrophobicity was estimated by the contact angle. The PSF/PANI Membranes were characterized by FTIR spectroscopy in order to identify the interaction between PSF, PEG and PANI in presence of NMP solvent. The UV-Visible results of the PANIs pellets indicate that PANIs were obtained as emerald salt, which confirmed by calculating the oxidation state of PANIs from the FTR spectra. The separation properties of the composite membranes were studied in terms of water permeability and methylene blue removal. The porosity and the permeability of the membranes PFS/PANI were improved in comparison with that of the pristine PSF membrane as well as rejection capacities (>97%) for methylene blue. In addition to this, the pure water flux of the membrane (PANI (B and D)) increased reaching up to 0.0444 and 0.05824-ml. min⁻¹ respectively at a pressure of 3.0 bars.

KEYWORDS

Membrane
Polysulfone
Polyaniline
Water Treatment

ARTICLE HISTORY

Received	Revised	Accepted	Published
12 Sept 2025	24 Oct 2025	27 Oct 2025	05 Nov 2025

1 Introduction

Water is not only an important basic resource for human existence, but also a strategic resource for sustainable development of economy and ecology. Water resources are overused and inappropriately used, which hinders the sustainable development of the economy, the society and the environment, so that the water resource shortage has evolved from a regional problem in certain water-deficient nations to a challenge across [1]. With the rapid development of contemporary society, the contradiction between the serious shortage of water resources and the increasing demand for water resources is becoming more and more prominent [2]. The sustainable use of water resources has become an urgent problem to be solved [3]. In order to guarantee a continuous clean water resource for humans, the treatment of the effluents produced by the industry is one of the most important challenges today. Until now, plentiful functional materials and procedures have been developed to treat wastewater.

Traditional methods used to remove molecular contaminants from wastewater include absorption, chemical degradation and flocculation, usually carries the risk of secondary pollution because of the addition of chemical reagents during these processes [4-6]. In contrast, nanofiltration (NF) technology has shown flourishing prospects in numerous applications such as drinking water treatment, seawater desalination, wastewater reclamation, due to its small footprint, environmental sustainability, low energy consumption [7-9].

Ultrafiltration (UF) is a well-developed separation process used in water/waste water treatment, reverse osmosis pretreatment, separations in food, chemical and biochemical industries which is applied in pre-treatment of desalination by reverse osmosis membranes, and it is able to produce water with constant quality regardless of the turbidity of the feed water [10-12].

The Polysulfone Membranes (PSf) are widely used in industrial water disinfection. Membrane material sowing to its outstanding acidic and basic resistance, good thermal stability and film forming ability [13]. However, PSF-UF membrane has low permeability and suffers serious membrane fouling due to their hydrophobic properties, which limits its application and shortens membrane life [13-15].

Extensive research has focused on the improvement of the mechanical and hydraulic properties of PS membranes. The membrane material can be elaborated by adding different additives, solvents, porous agents to the base polymer, and by varying the experimental conditions and chemical parameters during its synthesis. Polysulfone membranes are used in water treatment for the removal of contaminants through processes such as reverse osmosis or ultrafiltration [16]. However, to improve the functionality of PS membranes, such as separation capacity or adsorption, hydrophilicity, and fouling resistance, PS-based membranes can be modified. PANI, a conductive conjugated polymer formed from amine and imine groups, generally refers to modifications by incorporating polyaniline into the polysulfone membrane. This combination combines the thermal and mechanical stability of polysulfone with the conductive properties and

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hydrophilicity of polyaniline for more efficient wastewater treatment [17, 18]. It has been reported that blending of PS with hydrophilic additive in the casting solution can obtain excellent membrane performance with high permeability and antifouling property [19, 20]. In this context, as a desirable membrane material, polyaniline (PANI) have attracted a great deal of interest because of its appealing potential applications in nano/microelectronics, sensors, etc., stability, simple acid-base doping chemistry, and low cost [21]. PANI has a good ionic conductivity which can only be obtained with the help of protonic acids due to its selective conductive mechanism. So, after its doping with a protonic acid, hydrogen ions (H^+) of the latter enter into the PANI main chain, and thus make it possible to convert PANI from an insulator into a conductor [22, 23].

Concerning solvents, N-methyl pyrrolidone (NMP) has proven to be a good solvent for both sulfonated polymers and PANI [24]. To enhance PSF membrane surface properties like hydrophilicity and charge on a nanoscale and hydrodynamic properties such as permeability and rejection rate, polyaniline (PANIs) (A, B, C, D, E and F) at different compositions (from 0.2 % to 1.0 %) and Polyethylene glycol (PEG) was introduced as an additive during preparation of the membrane with the inversion process. This technique is

based on controlled interaction of solvent (NMP) and non-solvent (distilled water) solutions to cause a transition of the polymer separations.

2 Material and Methods

2.1 Reagents

Polysulfone (PSf, Udel P-3500, Mw: 35000 Da) used as membrane material was supplied by Solvay Advanced Polymer (Belgium), polyethylene glycol 1500 (PEG1500) from Merck, N-methyl-2-pyrrolidone (NMP, 99%) from Fluka was used as solvent directly without further purification. Six different types of polyaniline (PANIs) are synthesized with the same procedure (Aniline/dopant = 1.2 and Aniline/Oxydant = 1.0), but different solvents were used in the composition of the membranes. For the rejection and permeability performance of the membranes, methylene blue and pure water were used. The dopants as well as the solvents used for the synthesis of the different PANIs and the abbreviations used to represent them are listed in the Table 1.

Table 1. The different dopants and solvents used for the synthesis of PANI.

PANI	A	B	C	D	E	F
Solvents	DMSO + Ethanol	Toluene	Ethanol	Ethanol	Ethanol	insoluble in DMSO + Ethanol
Dopants	Naphtalene Disulfonic Acid (NDSA)	Cinnamic acid	Cinnamic acid (CA)	Tartaric acid (TA)	Naphtalene Disulfonic Acid (NDSA)	Naphtalene Disulfonic Acid (NDSA)

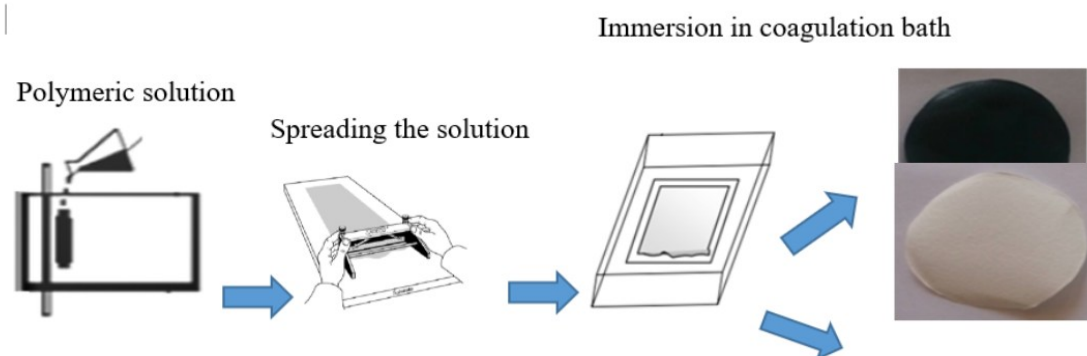


Fig.1. PSF/PANI Membrane preparation by phase inversion method.

Table 2. Compositions of PS/PANI membranes.

Membranes \ (%)	PSF	PANI (C, D, E)	PEG	NMP
M0	16.0	0.0	2.0	82.0
M0.2	16.0	0.2	2.0	81.8
M0.5	16.0	0.5	2.0	81.5
M1	16.0	1.0	2.0	81.0

2.2 Preparation of PSf/PANI -UF membrane

PSf/PANI nanocomposite membranes were prepared via phase inversion process. The mass ratio of PSF to total blended solution was 16 wt%, PEG was 2 wt %, and PSF/PANI mass ratio was varied from 0.0, 0.2, 0.5, and 1 wt (the compositions membranes are collected in table 2). Firstly, PSf was dried for 24 h in an oven at 50°C. Afterwards, PSf, PEG and PANI were dispersed in NMP solvent for 24 h at ambient temperature to form membrane casting solution. The casting solutions were left to degas for 45 min to allow complete release of bubbles, then casted on a glass plate with a steel knife to get membranes with homogeneous thickness. Finally, the glass plate was immersed in coagulation bath (Figure 1).

2.3 Characterization of PSf/PANI membranes

2.3.1 FTIR and UV-visible analysis

The chemical compositions of polymers in the elaborated membranes were identified using a Fourier transform infrared spectrometer (FTIR-ATR) in the range from 4000 cm^{-1} and 500 cm^{-1} . Samples were scanned 32 times with 4 cm^{-1} (model: Spectrum Two, Perkin Elmer). A UV-visible study of PANI was performed with OPTIZEN spectrophotometry in the wave number range of 200-800 nm.

2.3.2 Membrane hydrophilicity measurement

The hydrophilicity of PSF/PANI membranes was characterized by measuring the contact angle and the water uptake. The static contact angle was measured by the sessile drop method at 20 °C using a contact angle measurement instrument (model: GBX DIGIDROP[®] Digitizer of droplets). The PSF/PANI membrane at 1 % mass ratio was cut into a 2 cm x 2 cm shape. 2 µL of water was injected onto the membrane surface to measure the degree of contact. A minimum of five water contact angles in different locations was measured for each membrane sample.

Water uptake is an important parameter because it is related to the porosity of the membrane. In order to determine the percentage or water content of polymer membranes, the samples were cut into squares with size 2 cm x 2 cm. Then, the membrane samples were dried in an oven at 40°C for 24 h. After being wetted thoroughly by pure water, the samples were weighed under wet status and were dried until a constant mass was obtained (while the mass of the membrane sample itself is constant, the final result is influenced by the membrane's composition and physical structure. Water uptake of PSF/PANI membranes was measured using the following equation [25]:

$$\text{water uptake}_{(\%) } = \frac{(m_w - m_d)}{m_w} \times 100 \quad (1)$$

where, m_w and m_d are representing the mass of wet membrane sample (g), the mass of dry state membrane sample (g), respectively.

2.3.3 Pore size and porosity measurements

The porosity of the membrane was determined from the mass loss of the wet membrane before and after it dries. The porosity (ϵ) is the ratio of the pore volume to the geometric volume of the membrane it is calculated according the following equation (2):

$$\epsilon_{\%} = \frac{m_w - m_d}{A \times l \times \rho} \times 100 \quad (2)$$

The mean pore radius R_m (m) is calculated according to the Guerout-Elford-Ferry equation [24]:

$$R_m = \sqrt{\frac{(2.9 - 1.75\epsilon) \times 8\eta l Q}{\epsilon \times A \times \Delta P}} \quad (3)$$

where, η is the water viscosity (8.94×10^{-4} Pa.s); Q is permeate volume of pure water per unit time (m^3/s); P is Applied transmembrane pressure (Pa).

2.3.4 Filtration tests

The filtration tests were examined using a cross-flow UF experimental apparatus (effective membrane area of 13.4 cm^2) in a polyethylene cylindrical cell that could hold 50 ml of solution. Initially, the membrane compaction was carried out at 1 Bar transmembrane pressure (TMP) until a constant water flux was obtained. The tests were executed to examine permeation flow and rejection. The test of rejection was performed with methylene blue dye (MB).

Permeability depends on the pore size and the thickness of the membrane for porous membranes. It also depends on the chemical properties for dense membranes [26, 27]. The pure water flux was measured under the condition of 1, 2, and 3 Bar respectively, and it was calculated using the following equation (4):

$$J_w = \frac{V}{A \times \Delta t} \quad (4)$$

where, J_w is the pure water flux ($\text{ml.cm}^2.\text{min}^{-1}$); V is the volume of the permeate (ml); Δt is the permeation time (min).

The rejection property of the membrane was tested at pH = 7, $P = 3$ Bar, and $T = 26 \pm 1$ °C using MB aqueous solution at 20 mg.L^{-1} initial concentration. The residual concentrations of MB were measured using the UV-vis spectrophotometer, at a wavelength corresponding to the maximum absorbance of MB ($\lambda_{\text{max}} = 664$ nm). The MB rejection was calculated by the following equation (5):

$$R_{(\%)} = \left(1 - \frac{[MB]_p}{[MB]_f} \right) \times 100 \quad (5)$$

where, $[MB]_p$ and $[MB]_f$ are the concentrations of MB in permeate and in feed (mg.L^{-1}), respectively.

3 Results and discussion

3.1 Structural characterization of membranes

The UV-vis spectra of different PANIs synthesized (A, B, C, D, E and F) are given in Fig. 2 All spectra show three maximum absorption bands, which justify at first blush, obtaining PANI in the form of an emerald salt. The bands observed around 350 nm correspond to the $\pi-\pi^*$ transition of the benzoid system. The shoulders situated in 450 nm

correspond to the $n-\pi^*$ transition of the radical cation localization. The bands observed around 850 nm are attributed to the presence of localized polarons on the PANI structure [28, 29]. Concerning the doping effect, the latter results in the appearance of a band of low energy dispersion in the gap corresponding to the formation of polarons located along the polyaniline chain, and in the intensity of the band located around 350 nm, which decreases considerably. All these bands are characteristic of protonated polyaniline [30, 31].

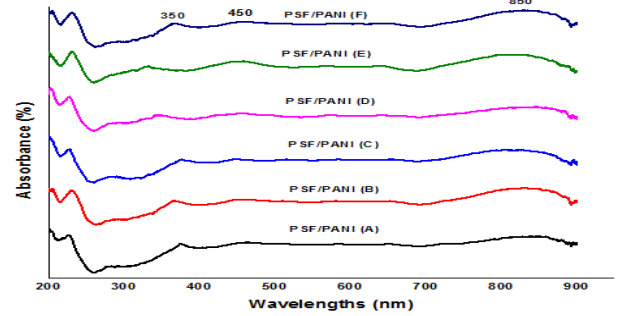


Fig.2. UV-visible spectra of PANI synthesized with the same procedure in the presence of different proton dopants and solvents.

Fig.3 is representative of the FTIR spectra of the different PANIs synthesized (A, B, C, D, E and F). As seen in Fig. 3, the peaks located at 1582, 1489, and 915 cm^{-1} are attributed to the stretching vibrations of various groups such as $-\text{N}(\text{C}_6\text{H}_4)-$, $\text{C}-\text{C}$ and $\text{C}-\text{C}/\text{C}-\text{H}$ in the benzene ring. Two bands recorded at 1300 and 1146 cm^{-1} are related to the vibrations of the $\text{C}-\text{N}$ bond in $(\text{N}-\text{B}-\text{N})$ system and $\text{C}=\text{N}$ bonds in the $(\text{N}=\text{Q}=\text{N})$ system, respectively [32]. The band at 1040 cm^{-1} presents in the PSF/PANIs (A, E, and F) membranes spectra, is attributed to so 3 bonds presented in the PANI dopant (NDSA) group.

The following formula (6) was applied from the FTIR spectrum to calculate the oxidation degree (DO) of the different PANIs [33]:

$$DO = \frac{A_{(\text{N}=\text{Q}=\text{N})}}{A_{(\text{N}=\text{Q}=\text{N})} + A_{(\text{N}-\text{B}-\text{N})}} \times 100 \quad (6)$$

where $A_{(\text{N}=\text{Q}=\text{N})}$ the band absorption of the quinoid rings in $(\text{N}=\text{Q}=\text{N})$ system and $A_{(\text{N}-\text{B}-\text{N})}$ the band absorption of the benzene rings in $(\text{N}-\text{B}-\text{N})$ system. The values found confirmed the emeraldine base structure ($y = 0.5$). The results of the oxidation degree calculation of the PANIs (A, B, C, D, E and F) are collected in table 3.

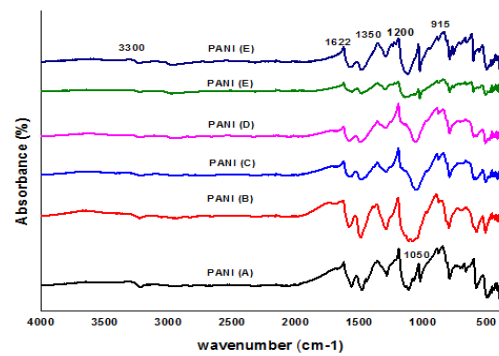


Fig.3. FTIR spectra of PANI synthesized with the same procedure in the presence of different proton dopants and solvents.

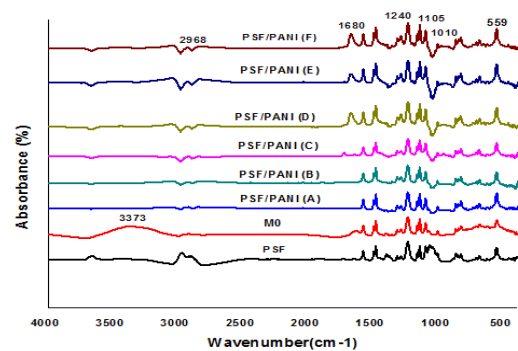


Fig.4. FTIR spectra of the pure PSF membrane and the PSF/PANI membranes at (0%, 1%) of PANIs.

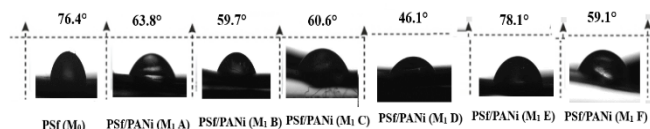
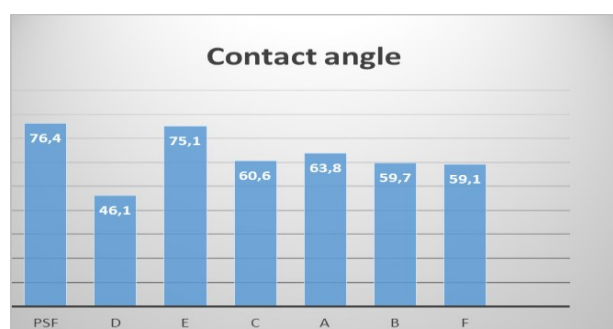
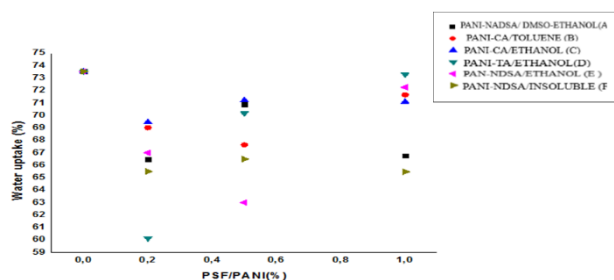
Table 3. Result of the oxidation degree determination of PANI from the IFTR spectrum.

PANI	A	B	C	D	E	F
DO(%)	51.2	50.8	51.02	51.2	50.3	49.9

The FTIR spectra of different membranes composite PSf/PANI: A, B, C, D, E, and F are shown in Fig. 4. The FTIR spectrum of pristine PS membrane reveals absorption bands at 1012 cm^{-1} (SO_3H), 1105 cm^{-1} (C-O), and 1240 cm^{-1} (asymmetric stretching vibration of the C-O-C (aryl ether group), which are characteristic of PSf. The peak at 2968 cm^{-1} is related to asymmetrical and symmetrical CH stretch of aliphatic CH_2 . The band at 1322 cm^{-1} is characteristic of the C-SO₂-C asymmetric vibrations [34, 35]. The band at 559 cm^{-1} is attributed to elongation vibrations of the C-S bond [36]. Otherwise, all the spectra of the composite membranes show a slight shift in peaks position compared to the spectrum of the pristine PSf membrane, indicating that the PANI molecules entered into the the interstitial space of PSf. Additionally, two new bands are located, one is broad at 3371 cm^{-1} in the spectrum of M0 and the other is medium at 1680 cm^{-1} in the spectrum of PSf/PANI membranes: A, B, E and F. The band at 3371 cm^{-1} is attributed to the elongation vibration of the O-H bonds of water, probably retained inside the pores during membrane formation. The band at 1640 cm^{-1} can be attributed to the deformation vibration of the O-H bond of water.

3.2 Membrane hydrophilicity measurement

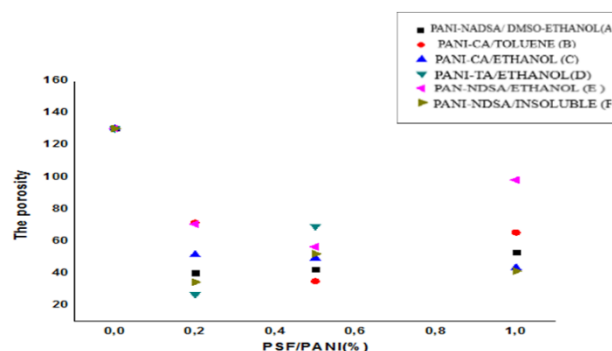
The contact angle is an important parameter to evaluate the hydrophilic/hydrophobic nature of the surface. Contact angle results of pristine and composite membranes are given in Figure 5 and Figure 6. The results show that the contact angles of PSf/PANI(B), PSf/PANI(D), and PSf/PANI(F) membranes decreased to 59.1°, 46.4° and 59.7° respectively, as compared to that of pristine membrane and PSf. PSf/PANI: A, C and E membranes, which is 76.4°, 75.1°, 60.6° and 63.8°, respectively; this is probably due to the increase in porosity of PSf/PANIs: D, B and F membranes. However, the water uptake of PSf/PANI membranes increased with PANI concentration (1%), which is consistent with contact angle measurements (Fig. 5). The N-H bond of PANI is apparently responsible for the decrease in hydrophilicity. This polar bond allows it to form hydrogen bonds with water, thus increasing the hydrophilicity of PSf/PANI membranes.

**Fig.5.** Water drop shape on the surface of the PSf and PSf/PANI membrane.**Fig.6.** Dynamic contact angle results of the membranes PSf/PANI at 1% of PANIs.**Fig.7.** Water uptake (%) of PSf/PANI membranes.

As shown in Fig. 7, the water uptake for the membranes decreases for the different PSf/PANI membranes. This decrease is due to the presence of PANI which appears to be trapped on the membrane surface, thus reducing the average pore size and porosity of membranes.

3.3 Mean pore radius and porosity measurements

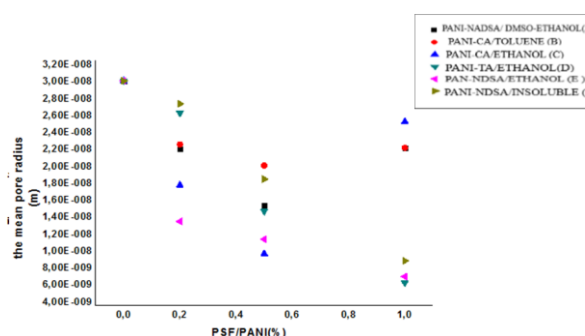
The porosity is an important characteristic which influences the permeability of the membrane. It depends on the membrane formation mechanism. According to the literature, a membrane can only be porous if the phase separation is instantaneous, slow, and dense [37, 38]. Porosity results for pristine and composite membranes are given in Fig. 8.

**Fig.8.** Porosity (%) of PSf/PANI membranes.

At a composition of 0.2 %, the porosity of the PSf/PANIs membranes (A, B, C, D, E, and F) record a decrease compared to the pristine membrane. This decrease can be attributed to the presence of PANI particles on the surface of the membranes, which has the effect of reducing the average pore size of the membrane. From 0.2 to 0.5 %, a further decrease in the porosity of the PSf/PANIs membranes (A, C, D and F) is observed, while that of the PSf/PANIs membrane (B and E) has increased. Between 0.5 and 1 %, it is noted that when the PANI composition reaches 1 %, the membrane porosity tends to increase (the porosity of PANI membranes (D and E) is higher than that of PANIs (A, B, C, and F)). The PSf/PANI membranes have a larger and more significant pore size due to the incorporation of PANI and the rate of diffusion between solvent NMP in the distilled water (non-solvent) bath increases during phase inversion which results in the formation of microvoids and porosity of the membrane increases. The increased porosity of the membrane can be due to the increased pore size of the membrane.

Moreover, as shown in Fig.9, the increased PANI composition from 0.2 to 0.5% decreases the mean pore radius of the PSf/PANI membranes compared to the pristine PS membrane. At 1 % PANI composition, a continuous decrease in the average pore radius is observed for the PSf/PANIs membranes (D, E, and F), unlike the PSf/PANIs membranes (A, B, and C) which record a remarkable increase in their average radius. This can be caused by the fast solvent-non-solvent exchange between the coagulation bath and the polymer solution during phase inversion due to the hydrophilic character of PANI.

Porous membranes may be classified by pore diameter: microporous ($d_p < 2 \text{ nm}$), mesoporous ($2 < d_p < 50 \text{ nm}$), and macroporous ($d_p > 50 \text{ nm}$) [39, 40]. According to this definition, all of the elaborated PSf/PANI membranes are classified as mesoporous membranes, with pore radii ranging from 6 to 30 nm.

**Fig.9.** Mean pore radius of PSf/PANI membrane.

3.4 Pure water flux

The pure water flux results given in Fig. 10 show that increasing the concentration of PANI additive in pristine PSf membranes improves water permeation. The enhancement of water permeation may be due to the improvement of hydrophilicity as noted for contact angle (Fig. 6) and water uptake (Fig. 7). Moreover, the addition of PANI reduces progressively the surface tension between water and the membrane, because the

N-H bond is a polar bond that allows it to create hydrogen bonds with water. This reduction increases the absorption rate of water molecules on the membrane.

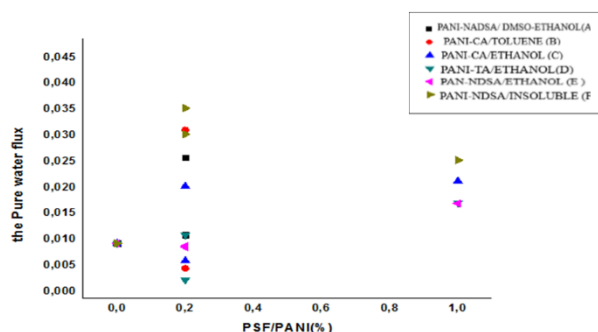


Fig.10. Mean pore radius of PSF/PANI membrane.

3.5 Rejection studies of PSF/PANI membranes

In order to determine the rejection process of all the elaborated PSF/PANI membranes, the membranes giving the best permeability were selected and tested with solution of meaning at a concentration of 20 mg.L^{-1} at pressure of 3 Bar. Maximum rejection was observed for PSF/PANI (D and E) membranes (97 %) and (95 %), respectively (Fig. 11). The performance of rejection declined gradually and reached 40% for PS membranes. The increase on rejection is attributed to the hydrophilic membrane's structures (Fig. 6), and the mean pore radius's smaller (Fig. 8). The membrane with the smallest mean pore radius will provide better reject properties. Also, the electrostatic effect between PANI which carries positive charges and methylene blue which is a chloride salt that carries positive charges, generates repulsive forces between the support PSF/PANI membrane and the solute MB.

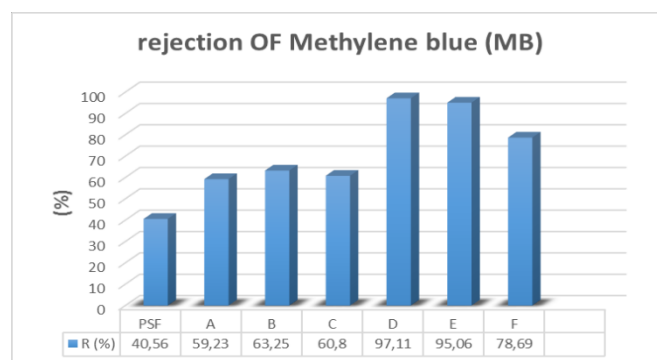


Fig.11. Rejection (MB) of the PSF/PANI membrane at 1% PANIs.

4 Conclusion

In this study, PSF/PANI membranes were elaborated using via the phase inversion method using six polyaniline (PANI) synthesized with the same procedure, but in the presence of different proton dopants and solvents. We studied the changes of structural properties by infrared spectroscopy (FTIR), optical spectroscopy by UV-Visible spectroscopy of different PANI synthesized with the same protocol, the only difference is focused on the type of used solvent (ethanol, toluene, DMSO) as well as the used protonic dopant (NDSA, cinnamic acid, tartaric acid). The infrared spectrum (FTIR), has allowed to characterize the protonated form of the PANIs, in particularly by displacement of some bands, that allowed to estimate the oxidation degree for each of the PANIs studied. The found values confirmed the structure of emeraldine base (with $\gamma=0.5$). UV-Visible spectroscopy has allowed confirming protonation of PANIs and obtaining a PANIs in the emeraldine salt form. Observing the FTIR absorption bands of the different membranes (PSf/PANI), we find that these bands do not change although PANI was diluted by NMP in the presence of PEG and PSf. This shows that there is no interaction between PANI and PSf during the inversion phase.

The results showed that the concentration and type dopant of PANI influenced the hydrodynamic and morphological properties of PSF/PANI membranes. The decrease contact angle from 76° to 46° and increase water uptake exhibited increase of the PSF membrane hydrophilic nature with an addition on PANI at 1%, The permeability was improved 80 % for the membrane PSF/PANI (D). The porosity results and ray showed that PANI concentration influenced the morphological properties of the membranes.

addition of the PANIs (D and E) improved up the rejection property of methylene a blue (mb) to 95%. The work realized allowed to highlight of the importance of the selection of the dopant and the concentration of the PANI to elaborate the PSF/PANI membranes. We cannot confirm that a doping is better than another one because that depends on the application targeted.

Declaration of Competing 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.

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