Al-Khwarizmi Engineering Journal

Al-Khwarizmi Engineering Journal
ISSN (printed): 1818 – 1171, ISSN (online): 2312 – 0789
Vol. 22, No. 1, March, (2026), pp. 1- 13

Preparation, Characterization, and Evaluation of Electrospun Copolymer Nanofiber Membranes for Kerosene Removal from Aqueous Solution

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(Received 21 September 2025; Revised 15 November 2025; Accepted 15 December 2025; Published 1 March 2026)
https://doi.org/10.22153/kej.2026.12.010

Abstract

The petroleum sector generates significant amounts of oily wastewater requiring proper treatment before being released into the environment. The high stability, hydrophilicity, and large surface areas of membranes provide great opportunities for treating oily wastewater. For this work, electrospun nanofiber (ESNF) membranes of polyacrylonitrile (PAN) and polysulfone (PSU) were manufactured and evaluated for their separation capabilities. Multiple concentrations of the PAN and PSU polymers were used to optimize the copolymer nanofiber (NF) membranes. The water flux, oil rejection, and porosity evaluation of the membranes were compared. In addition, the surface and profile of the ESNF membranes were evaluated in terms of morphology and roughness, wettability, and tensile strength, and the results of scanning electron microscopy, atomic force microscopy, contact angle, and tensile tests were incorporated. The ESNF membranes provided positive results in the separation of oily wastewater. In particular, the best results were observed for the membranes composed of 75% PAN and 25% PSU, which reached 99.7% oil rejection, a maximum water flux of 400 L/m^2.h, and 96% porosity, demonstrating great potential for wastewater treatment on an industrial scale. However, this study focused primarily on short-term performance evaluation. The long-term mechanical and chemical stability of ESNF membranes under continuous operational conditions remains to be examined.

Keywords: *Polyacrylonitrile; Polysulfone; Copolymer; Electrospinning; Nanofiber membranes; Oily wastewater*

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1. Introduction

Many industries, including food processing, textiles, mining, petrochemical and oil processing, pharmaceuticals, and metal finishing, discharge large volumes of oily wastewater into natural water bodies (e.g., rivers and seas) on a daily basis. When released into surface waters, these contaminants pose serious threats to aquatic life and the surrounding environment. The increasing levels of such pollutants have become a significant environmental concern [1].

Oily wastewater typically consists of two phases: a dispersed phase (suspended oil droplets) and a continuous phase (the surrounding water medium). Oil droplets can be categorized into three forms based on their diameter: free-floating (>150 µm), dispersed (20–150 µm), and emulsified (<20 µm). While physical separation methods generally work well for treating free and dispersed oils and oils that can easily be separated by flotation, they tend to be inefficient for emulsified oils due to the small size and instability of the droplets [2], [3].

The conventional treatment of emulsified oily wastewater by adsorption, biological processes, and even gravity separation is plagued by recontamination, corrosion, and high operational costs [4], [5]. In comparison, membrane separation methods are less operationally complex, smaller, and offer better eco-efficiency [2].

Various techniques, such as track-etching, sintering, phase inversion, and stretching, are used to fabricate membranes, and these methods influence the essential membrane properties [6]. Among them, electrospinning (ES) has become one of the most versatile and efficient methods for producing nanofiber (NF) or microfiber (MF) membranes with wide aspect ratios, due to its simplicity and convenient operation [7], [8]. ES uses a high-voltage electrostatic field and various polymer solutions. Owing to their small diameters (as low as a few nanometers) and properties such as pore size distribution, high porosity, and large surface areas, electrospun fibers are ideal for separation applications [9]. Furthermore, compared with other methods, ES is a simple, inexpensive process that is applicable to various raw material types and allows easy adjusting of NF alignment [10].

Given the rapid development of science and technology, new membrane materials for effective oily wastewater treatment are needed. The most common polymers are polyacrylonitrile (PAN), polysulfone (PSU), polyethersulfone, polyvinyl alcohol (PVA), and polyvinylidene fluoride (PVDF). PSU has been widely used due to its outstanding thermal stability, mechanical properties, porosity, surface area, and conductivity. However, it is hydrophobic and does not enhance water permeation. For these issues, hydrophilic materials should be added to increase the wettability of the membrane [11]. PAN, a hydrophilic and chemically stable polymer, can be successfully electrospun, making it suitable for use as an ESNF. It has high molecular weight, thermal stability, good mechanical properties, and very good compatibility with other materials [12].

These properties justify the use of PAN for modified surface hydrophilicity and functionality in PSU-based membranes [13]. For example, in previous studies, 8% PAN NF mats modified with various concentrations of boron nitride (BN) nanoparticles (1wt%–4 wt%) were prepared [13]. The BN/PAN membrane showed excellent properties, with 99% oil rejection, 79% porosity, improved mechanical properties, and smooth to rough surface morphologies due to the decrease in fiber size [9]. Diwan et al. (2023) studied ESNFs and cast thin-film PAN/DMF membranes of different polymer concentrations (10%–14%). The 14% electrospun PAN/DMF membrane showed significantly higher performance than the same composition of cast membrane in water flux, oil rejection, surface roughness, and porosity [14].

Although plasma technologies have been evolving, scant research has been conducted on the effect of different PAN/PSU ratios on oily wastewater separation. The present study attempts to address this issue by determining how varying ratios of PAN/PSU copolymer affect membrane functionality. Membranes were fabricated by blending varying concentrations of PAN and PSU and then ES.

The prepared membranes were characterized using scanning electron microscopy (SEM), atomic force microscopy (AFM), mechanical testing, porosity measurements, and contact angle (CA) analysis. Their performance in oily wastewater separation was evaluated using a bench-scale experimental setup to identify an optimal formulation for industrial application. This study is the first systematic evaluation of PAN/PSU copolymer ratios (100:0, 75:25, 50:50, 25:75, 0:100) fabricated under identical ES conditions. No additives or surface modifiers were used, highlighting the intrinsic synergistic effect between PAN and PSU.

2. Materials and Methods

2.1. Materials

PAN (purity: 99%, molecular weight: 150,000 g/mol) and PSU (molecular weight: 35,000 g/mol) were purchased from Shandong Natural Micron Pharm Tech Co., Ltd. N,N-dimethylformamide (DMF) (chemical formula: $HCON(CH_3)_2$, molecular weight: 73.10 g/mol) was supplied by THOMAS BAKER (India). Kerosene ($C_{12}H_{26}$–$C_{15}H_{32}$), with a density of 0.81 g/cm³ at 25 °C and an average molecular weight of 175 g/mol, was obtained from the Midland Iraqi Refineries Company. Tween-80 ($C_{64}H_{124}O_{26}$), a non-ionic surfactant with a density of 1.07 g/cm³ at 25 °C and a molecular weight of 604.81 g/mol, was purchased from Hopkin & Williams, Ltd. All chemicals were used as received.

2.2. Fabrication of nanofiber membranes

Five PAN/PSU precursor solutions were prepared by dissolving a total polymer concentration of 15 wt.% in DMF, which served as the solvent at 85 wt.%. The specific mass ratios of

PAN to PSU in each ESNF formulation are presented in Table 1.

Table 1,
Mass ratios of the dissolved PAN/PSU in DMF in the electrospun nanofiber membranes

Membrane	PAN (wt.%)	PSU (wt.%)
M_1	100	0
M_2	75	25
M_3	50	50
M_4	25	75
M_5	0	100

A total of 20 g of each prepared dope solution was transferred into a glass beaker, heated to 60 °C, and stirred at 600 rpm for a minimum of 6 h to ensure complete dissolution of PAN and PSU in the DMF solvent. Following dissolution, the solutions were left undisturbed overnight to allow for degassing and removal of air bubbles.

The ES setup used to fabricate the ESNF membranes is illustrated in Fig. 1. The system comprised a syringe pump for controlled delivery of the polymer solution, a 5 mL disposable plastic syringe fitted with a 23G needle (inner diameter ~0.33 mm), a high-voltage power supply, and a rotating metal drum collector covered with a sheet of thin aluminum foil. The distance between the needle tip and the collector surface was maintained at 14 cm. For NF membrane production, the prepared polymeric solution was mounted onto the syringe pump. The polymer solution was ejected through the needle and directed toward the rotating collector drum under the following conditions: a flow rate of 1 mL/h and an applied voltage of 15 kV. The NF membranes were collected directly on the aluminum foil affixed to the drum (15 cm × 25 cm). ES was carried out at ambient temperature and humidity. Upon completion, the membranes were carefully peeled from the foil surface and dried at room temperature until further use.

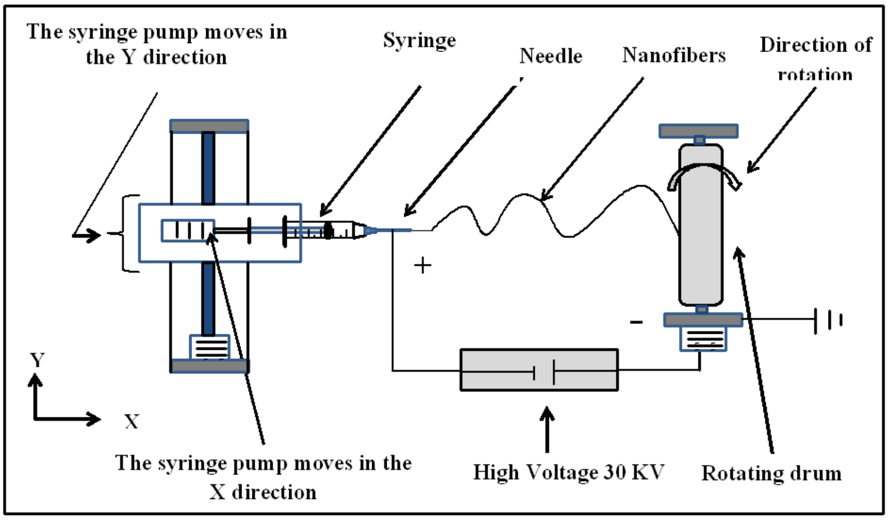


Fig. 1. Schematic diagram of the electrospinning system [14].

2.3. Preparation of the emulsified oil solution

An oil-in-water emulsion with a concentration of 500 ppm was prepared by dissolving 0.5 g of kerosene in 1,000 mL of deionized water, followed by the addition of 0.05 g Tween-80 as a surfactant and emulsifying agent. The mixture was homogenized using a high-shear emulsifier device (Model: SRH-S Lab High-Shear Emulsifier; 220 V, 50 Hz, 50 W) for 10 min at 10,000 rpm. The resulting oil emulsion was used immediately in the membrane filtration experiments to ensure stability and uniformity [15]. Emulsification using the emulsifier device is illustrated in Fig. 2. The oil concentrations in both feed and permeate were determined using UV–vis spectrophotometry at a wavelength of 290 nm, utilizing a UV-9200 spectrophotometer (Biotech Engineering Management Co., Ltd., UK). To ensure emulsion stability, we measured the oil droplet sizes using LOMO Laboroscope AL-2000 microscope.

Fig. 2. Preparation of the oil-in-water emulsion using the high-shear emulsifier device.

2.4. Membrane Characterization Tests

The surface morphology of the ESNFs was analyzed using SEM (TESCAN MIRA3, France) and AFM (Core AFM 2023, Nanosurf AG, Switzerland). These tools were used to assess fiber structure, pore size, and topographical uniformity. Surface wettability of the membranes was assessed using a goniometer (Theta Lite TL-100 and TL-101, Ryts Instruments, Bangkok, Thailand) to perform CA measurements. Samples were prepared (2 cm × 2 cm) for each membrane mat, and to guarantee precision and uniformity, the samples were dried at room temperature before measurement.

Porosity is one of the major characteristics affecting membrane performance since it indicates the volume portion of the membranes that is taken up by the pores [8]. The porosity (Ƹ) of the ESNF membranes was determined by its wet and dry weight using the gravimetric method [16]. Square membrane samples were cut into 2 cm × 2 cm sections. For each dry sample, the weight (W_{dry}) was taken, and then the sample was submerged in isopropyl alcohol for 5 min to achieve full liquid penetration. The weight was taken again (W_{wet}) to measure the sample after the 5 min soak. The porosity of the sample was then calculated using the following equation:

$$\mathcal{E} = \frac{\frac{Wwet - Wdry}{pIPA}}{V} \times 100\% \qquad \text{...(1)}$$

where W_{wet} and W_{dry} are the wet and the dry masses of a sample (gm), respectively; p_{IPA} is the density of isopropyl alcohol (0.785 gm/cm^3), and V is the geometrical volume of a sample in (cm^3).

2.5. Membrane performance test

The performance of the fabricated membranes was evaluated using a cross-flow filtration arrangement (Fig. 3) containing a rectangular cross-flow membrane test cell (6 cm × 12 cm). A 500 mL beaker containing an oil-water emulsion was used to feed the solution, and the beaker was connected to a feed pump that passed the solution through the membrane cell. For all the experiments, the pressure was set to 0 bar and was controlled using a valve. A feed flow rate of 100 mL/min was maintained.

Filtration tests were conducted for 3 h, with each test divided into six 30-minute episodes. Membrane performance was evaluated on two criteria: water flux and oil rejection [17]. The water flux (J_w) in L/m^2.h (LMH) was calculated based on the formula:

$$Jw = \frac{V}{A\,t} \qquad \text{... (1)}$$

where V is the filtration volume (L), A is the effective area of the membrane (m^2), and t is the filtration time (h) [18].

The oil rejection rate (R), which represents the percentage of oil retained by the membrane, was calculated using Eq. (2):

$$R = \frac{Cf - Cp}{Cf} \times 100\% \qquad \text{...(2)}$$

where C_f is the concentration of oil in the feed (mg/L), and C_p is the concentration of oil in the permeate (mg/L).

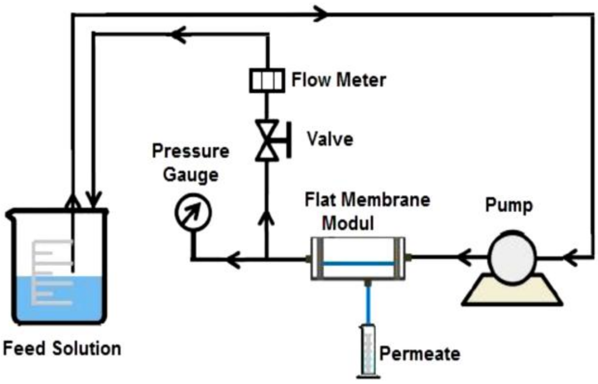


Fig. 3. Schematic diagram of the cross-flow membrane filtration setup.

3. Results and Discussion

3.1. Emulsified Oil Characterization

Figure 4 presents the digital image of the prepared emulsion solution, which contains oil droplets in the range of 1.5–12 µm, confirming the successful preparation of the solution. The average droplet size was less than 10 µm for more than 48 h. Thus, the prepared emulsions in our experiments can be classified as emulsions. The changes in droplet sizes and agglomerations in time are sufficiently slow, so their effects on the time scale of the filtration experiments can be disregarded .

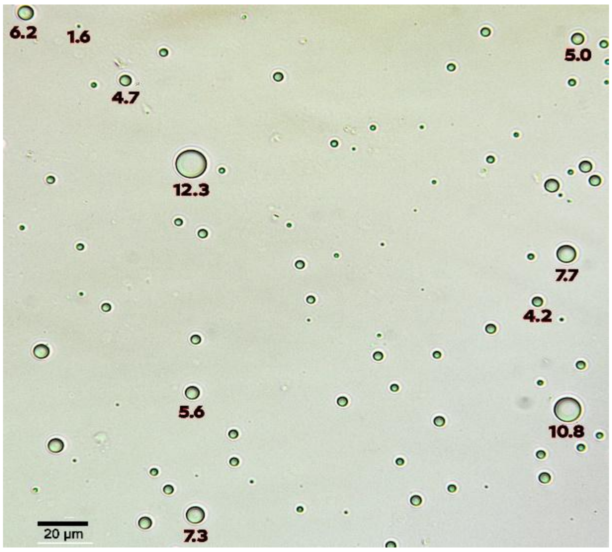


Fig. 4. Digital image of the oil droplet in the prepared emulsions (500 mg/L) at 25 °C

3.2. Membrane Characterizations

3.2.1. Morphology of the ESNFs

The changes in surface morphology of the ESNFs are shown in Fig. 5. In general, the properties and morphologies of the ESNFs are mainly determined by the composition of the ES polymeric dope solution [16]. Thus, we can see from Fig. 5a that M_1 produced straight, uniform, and continuous NFs compared with M_5, which are non-homogeneous NFs with numerous beads (Fig. 5e). The results in bead formation can be explained by the increase in PSU polymer viscosity, which increased with the increase in PSU concentration in the dope solution [19]. In addition, the beads became bigger when the viscosity of the dope solution increased, and vice versa. This viscosity can be defined as the relationship between a specific polymer and a solvent in the dope solution [20]. Meanwhile, Figs. 5b–d illustrate an increase in the crosslinking between fibers with the increase of PAN concentration compared with the PSU concentration in the dope solution. This result indicates a good combination between PAN and PSU polymers, with the stability of the polymer solution increasing with the addition of a copolymer through ES [20]. For this reason, the rise in water flux and oil rejection is affected by the addition of a larger amount of PAN in the dope solution. Thus, M_2 gave the best selective properties obtained.

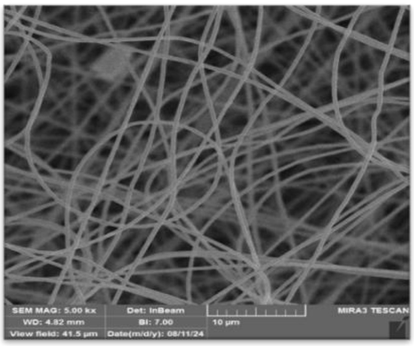
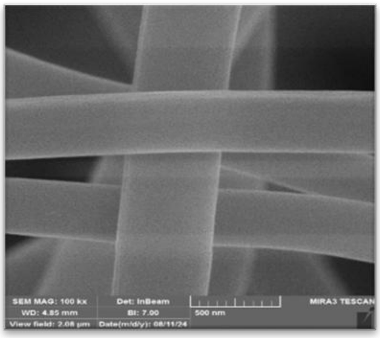
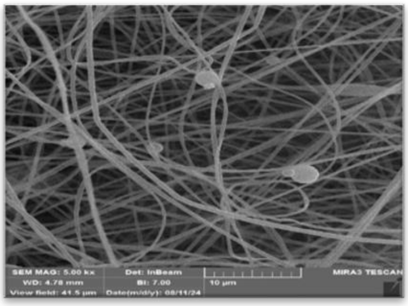
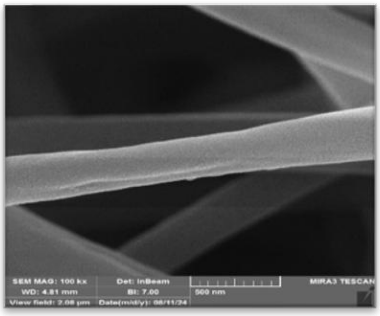
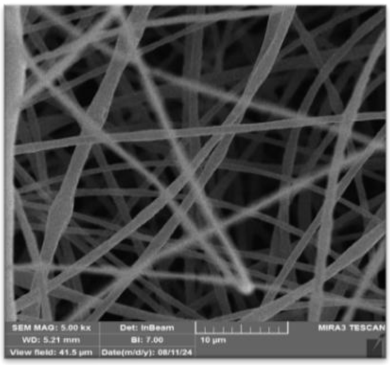
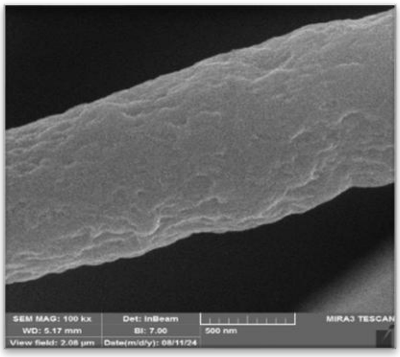
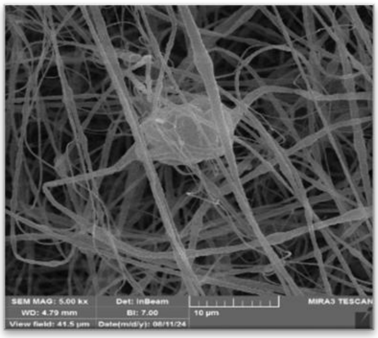
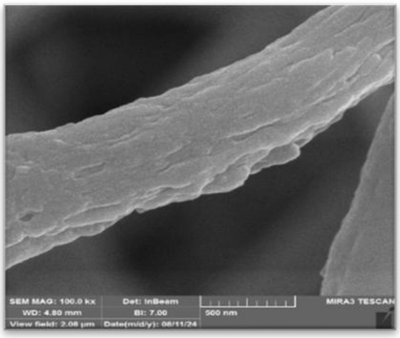
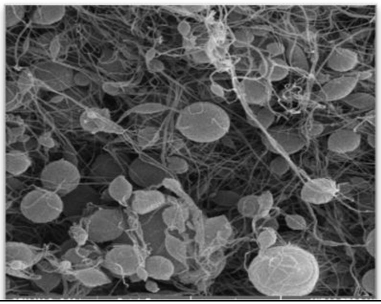
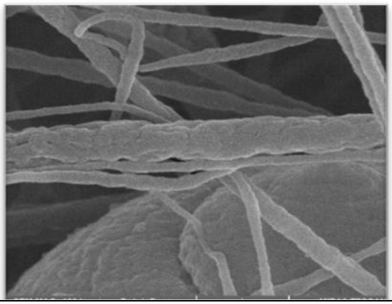
M1		
M2		
M3		
M4		
M5		

Fig. 5. SEM images of the electrospun nanofiber membranes.

3.2.2. AFM Analysis

The AFM technique gives evidence of the change in surface roughness of a membrane. Surface roughness is known to increase the hydrophobicity of a membrane [21]. Fig. 6 shows the surface AFM images of the ESNFs obtained and the values of root mean square height (Sq) and arithmetic mean height (average roughness) (Sa) that can be calculated from the roughness profile.

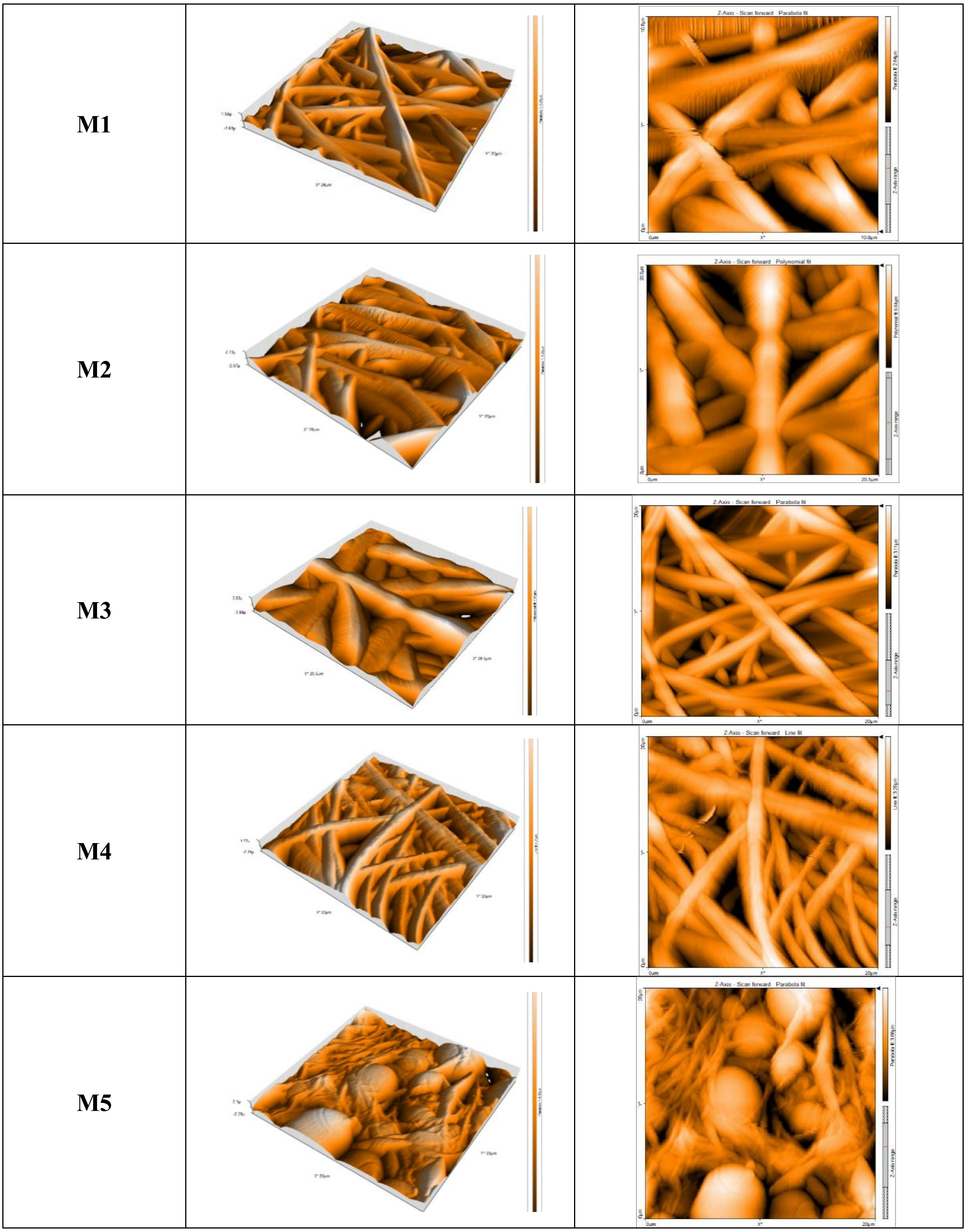


Fig. 6. AFM of the electrospun nanofiber membranes.

The Sa values for M_1 and M_5 were 587.2 and 669.7 nm, respectively. These results mean that M_5 is rougher than M_1.

On the other hand, for the copolymers M_2, M_3, and M_4, the Sa values were 785.4, 1,214.0, and 503.5 nm, respectively, indicating that the surface of the ESNFs became rougher with the increase of PSU concentrations compared with the PAN concentration in the dope solution. These results are recorded in Table 2.

Table 2,
AFM results of the prepared ESNFs

Membrane	Root mean square height (Sq) (nm)	Arithmetic mean height (Sa) (nm)
M_1	696.7	587.2
M_2	976.7	785.4
M_3	1508	1214
M_4	633.2	503.5
M_5	851.7	669.7

3.2.3. Contact Angle

The changes in CA of the ESNFs are shown in Fig. 7. As expected, the lowest CA obtained for M_1 is 79.5°, which displays a hydrophilic nature compared with M_5 (138°). The increase in PAN concentration compared with PSU in the dope solution displays a decrease in CA achieved. Accordingly, increasing the PAN content in ESNFs enhanced their hydrophilicity, as evidenced by the decreasing values of CA: 92.4° for M_2, 98.2° for M_3, and 105.8° for M_4. The hydrophilic nature of the membranes indicates enhanced wettability compared with hydrophobic membranes, increasing water flux, and improving oil removal in the filtration system. However, more details should be measured carefully.

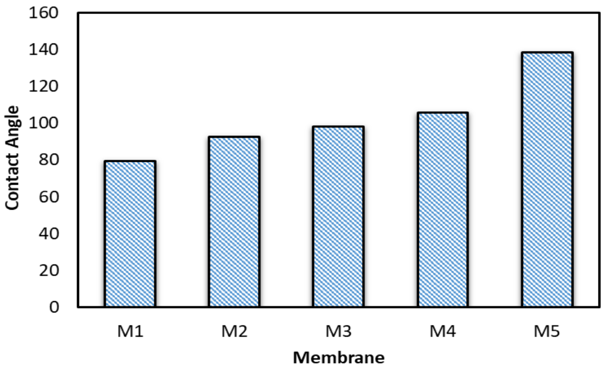


Fig. 7. Contact angles of the ESNFs prepared under different concentrations of dope solutions.

3.2.4. Porosity

NFs with high porosity can be considered a potential material for use in oil–water separation [8]. According to the experimental results (Fig. 8), of the five ESNFs prepared, the porosity of M_2 was around 96%, while this value was down to 93% and 92% for M_3 and M_4, respectively. This outcome can be explained by the increased interference between the fibers in M_2 compared with M_3 and M_4. The values of the three copolymers are higher than the single PAN ESNFs and the single PSU ESNFs. However, mixing copolymers with different polar properties can directly produce effective membranes with high porosity [20]. These results indicate that as the PAN increased in the dope solution, the porosity of the ESNFs increased. The high-water flux was then attributed to the high porosity and surface area.

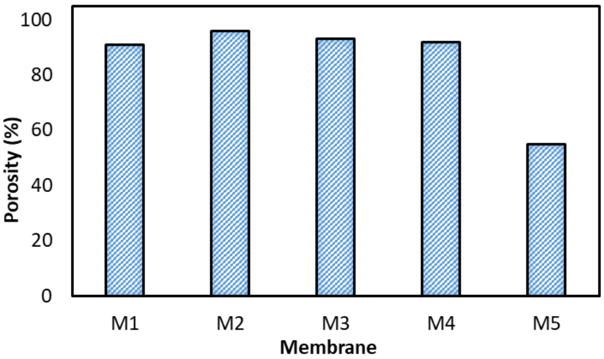


Fig. 8. Porosity of the ESNFs

3.2.5. Mechanical Properties

The mechanical properties of the ESNFs were measured, and the results are labeled in Table 3. Basically, the tensile test continues until tensile strength, which is the maximum stress before breaking, is reached. This method measures the force at break divided by the cross-sectional area of the sample.

The mechanical properties of NFs can be improved by adding other polymers. Thus, the tensile strength for M_2 was 2 MPa and decreased to 1.7 and 1.0 MPa for M_3 and M_4 copolymers, respectively.

On the other hand, the tensile strength for M_1 and M_5 was around 0.8 and 0.0525 MPa, respectively. These results exhibit improved mechanical properties for the copolymer ESNFs compared with the single PAN ESNFs and single PSU ESNFs. The increasing tensile strength is due to the decrease in fiber diameter, the molecular orientation in the amorphous phase of the polymer, and the arrangement of NFs in the mat.

Table 3,
Mechanical properties of the ESNFs

Membrane	Stress (MPa)	Elongation at Break (%)
M_1	0.8	1.6
M_2	2	4
M_3	1.72	3.4
M_4	1	2
M_5	0.053	0.11

3.3. Membrane performance results

The water flux and oil rejection efficiency of the ESNF membranes were evaluated using a 500 ppm oil-in-water emulsion. Performance tests were conducted using a cross-flow filtration system, and the corresponding results are presented in Fig. 9. As shown, the water flux of the membranes we tested started at 400 LMH for M_2 and finished with a water flux of 173 LMH for M_5, demonstrating a gradual decline of water flux.

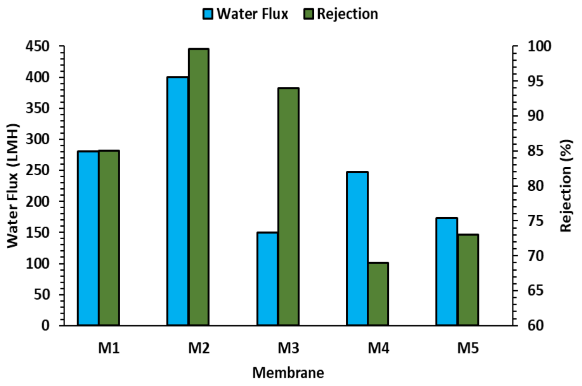


Fig. 9. Water flux and oil rejection of the prepared ESNFs.

M_2 outperformed all fabricated membranes in each parameter. Moreover, the separation time obtained aligned with previous results [22], [23]. M_2 had the highest oil rejection rate (~99.7%) and was therefore highly efficient in separation. This superiority was due to the increased hydrophilicity of ESNFs and the strong interfacial cross-linking between fibers, providing an effective method for separating oil from water. Table 4 summarizes the membrane performance changes and CA for each of the five ESNFs fabricated. Results show that the CA decreased linearly with an increased PAN concentration in the ES dope solution.

Table 4,
Comparison of the ESNFs

Membrane	Water flux (LMH)	Oil rejection (%)	Contact angle (°)	Porosity (%)
M_1	254	91%	79.5	91
M_2	400	99.7%	92.4	96
M_3	362	98.3%	98.2	93
M_4	297	95%	105.8	92
M_5	173	83%	138.5	55

A comparison of the pristine 14 wt.% PAN/DMF NF membrane with some previously reported electrospun PAN membranes tested in oil removal can be found in Table 5. The table also highlights the differences in filtration methods employed in these studies. Notably, no additives were used to enhance flux and oil rejection in this work, but this could be a topic for future research. Our membrane showed higher water flux and comparable oil rejection compared with the previous research that utilized cross-flow systems.

Table 5,
Recent progress in ESNFs for oil–water separation

Membrane type	Polymer concentrations (wt.%)	Water flux (LMH)	Oil rejection (%)	Porosity (%)	Contact angle (°)	References
PAN	12%	1850			20	[23]
PS	18%	209			113	
	22%	1332			116	
	26%	1679			125	
	30%	1841			126	
PVDF		180	>98		109.7	[24]
PVDF–Graphene nanofiber membranes	1.5%:15%	210			87.9	
PAN	8%		98	73.3	69.1	[9]
BN/PAN	8%:1%			73.7		
BN/PAN	8%:2%			75.1		
BN/PAN	8%:3%		99.8	78.9	82.4	
BN/PAN	8%:4%			79.0		
PAN	10%	120	96	92	57	[14]
	12%	210	96.5		36	
	14%	250	97	96	31	
PAN/CA	1%:3%	502.9–857.2	>99.9		0	[13]
CNTs–PAN	[(8 + 5) wt.% PAN (double layer heat-pressed) + (0.2 wt% CNTs and 0.05 wt% PVA) and 0.05 wt% Glutaraldehyde (GA)]	60	96			[22]
PAN/PSU	100%:0%	281	85	92	79	This work
	75%:25%	400	99.6	96	92	
	50%:50%	150	94	93	96	
	25%:75%	247	69	92	105	
	0%:100%	254	73	55	138	

4. Conclusions

The need for effective oily wastewater treatment resulted in the development of novel ESNFs with tunable properties. ES PAN and PSU were achieved at five varying concentrations, and the end ESNFs were characterized by SEM, AFM, CA, mechanical strength, and porosity. Experimental results indicated that combining PAN and PSU copolymers can significantly enhance water flux, oil rejection, wettability, mechanical strength, and porosity. Furthermore, increasing PAN concentration in the dope solution clearly achieves optimal membrane properties.

These findings suggest that the membrane with 75% PAN/25% PSU (M_2) holds significant potential for industrial-scale oily wastewater treatment. It reaches the optimal permeate flux value of 400 LMH, a maximum separation efficiency of 99.7%, and maximum porosity, making it an excellent oil–water separation membrane for safety and environmental protection.

Future work will focus on evaluating the long-term mechanical and chemical stability of the PAN/PSU membranes under continuous operational conditions to confirm their industrial viability.

Conflict of Interest

The authors declare that they have no conflicts of interest.

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تحضير وتوصيف وتقييم أغشية ألياف نانوية من البوليمر المغزول كهربائيًا لإزالة الكيروسين من المحاليل المائية

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المستخلص

يُنتج قطاع البترول كميات كبيرة من مياه الصرف الصحي الزيتية التي تتطلب معالجة مناسبة قبل إطلاقها في البيئة. تُوفر الأغشية عالية الاستقرار، والمحبة للماء، والمساحات السطحية الكبيرة فرصًا واعدة لمعالجة مياه الصرف الصحي الزيتية. في هذا العمل، صُنعت أغشية ألياف نانوية مغزولة كهربائيًا (ESNFs)من بولي أكريلونيتريل (PAN) وبولي سلفون (PSU) ، وقُيِّمت قدراتها على الفصل. لتحسين أغشية الألياف النانوية البوليمرية المشتركة(NFs) ، استُخدمت تراكيز متعددة من بوليمرات PAN وPSU .قُورنت الأغشية في تقييم تدفق الماء، ورفض الزيت، والمسامية. بالإضافة إلى ذلك، قُيِّم سطح وشكل أغشية الألياف النانوية المغزولة كهربائيًا من حيث الشكل والخشونة، وقابلية البلل، وقوة الشد، وأُدرجت نتائج اختبارات المجهر الإلكتروني الماسح(SEM) ، ومجهر القوة الذرية (AFM) ، وزاوية التلامس (CA)، واختبارات الشد. وقد أظهرت أغشية الألياف النانوية المغزولة كهربائيًا نتائج إيجابية في فصل مياه الصرف الصحي الزيتية. وعلى وجه الخصوص، تم ملاحظة أعظم النتائج بالنسبة للأغشية M2 التي كان تركيبها ٧٥٪ PANو٢٥٪ PSUوالتي وصلت إلى رفض الزيت بنسبة ٩٩,٧٪، وتدفق المياه الأقصى ٤٠٠ LMH، ومسامية ٩٦٪، وبالتالي، إمكانات أكبر لمعالجة مياه الصرف الصحي على نطاق صناعي.