EEET ECOLOGICAL ENGINEERING & ENVIRONMENTAL TECHNOLOGY

Ecological Engineering & Environmental Technology, 2025, 26(12), 238–249
https://doi.org/10.12912/27197050/214187
ISSN 2719–7050, License CC-BY 4.0

Received: 2025.10.20
Accepted: 2025.11.15
Published: 2025.12.01

Electrospun nanofiber polymer membranes for sustainable and efficient wastewater purification

Rianyza Gayatri[1*], Wirach Taweepreda[2], Rara Eka Dyla Putri[3], Muzafar Zulkifli[4], Ahmad Naim Ahmad Yahaya[4]

[1] Department of Chemical Engineering, Faculty of Engineering, Universitas Sriwijaya, Jalan Palembang-Prabumulih KM 32, Indralaya, Ogan Ilir 30662, South Sumatera, Indonesia
[2] Polymer Science Program, Division of Physical Science, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90110, Thailand
[3] Industrial Chemical Technology Study Program, Chemical Engineering Department, Sriwijaya State Polytechnic, Jl. Srijaya Negara, Bukit Lama, Ilir Barat, Palembang, South Sumatra, Indonesia
[4] Green Chemistry and Sustainability Cluster, Universiti Kuala Lumpur, Branch Campus Malaysian Institute of Chemical and BioEngineering Technology, 78000 Alor Gajah, Melaka, Malaysia
* Corresponding author's e-mail: rianyzagayatri@ft.unsri.ac.id

ABSTRACT

Nanofiber membranes made of polyvinylidene fluoride (PVDF) and titanium dioxide (TiO_2) were investigated using the electrospinning method. Polyvinylpyrrolidone (PVP) was used as a polymer substance in this study. The utilisation of electrospinning as a technique for producing nanofibers and other one-dimensional nanostructures is considered a straightforward and adaptable approach. The experiment involved the electrospinning of fibrous membranes. The electrospinning process was conducted with a voltage of 16 kV and a feeding rate of 1 ml/h. The distance between the syringe needle tip and the filter membrane was 10 cm. The membrane's wettability and morphology were assessed through contact angle and scanning electron microscopy (SEM) measurements. Fourier transform infrared spectroscopy (FTIR) was employed to examine the membranes' functional groups and elemental composition to ascertain their effectiveness in water filtration. Bovine Serum Albumin (BSA) was utilized as a standard solution. The results show that the synthesized PVDF-TiO_2-PVP nanofiber has great potential for water filtering applications. The PVDF-TiO_2-PVP membrane's contact angle was lowered to 117 degrees. As a result, the PVDF-TiO_2-PVP membrane achieved its target water flux of 1806.14 L/(m^2.h), which is an improvement over previous tests. Compared to a polymeric membrane made of pure PVDF, the results suggest that adding additives improves filtration performance.

Keywords: PVDF, TiO_2, PVP, nanofibers, electrospinning, filtration, BSA.

INTRODUCTION

Straightly membrane separation methods have shown widespread interest (Du et al., 2021). Using sieving techniques with diverse particle sizes and the coalescence process plays a pivotal role in addressing the interface challenge encountered in membrane separation, as highlighted by Bai et al. (Bai et al., 2020), Membranes exhibiting distinct wettability characteristics, such as hydrophilic-oleophobic or hydrophobic-oleophilic properties, can facilitate the passage of water or oil, respectively, while impeding the transport of the other component selectively. This attribute enables the effective accomplishment of oil-water separation objectives (Tian and Ma, 2020; Ying et al., 2020).

Over the years, researchers have developed a keen interest in fibrous structures. Regarding oil/water emulsion separation, the electrospun nanofibrous membrane stands out because of its specific surface area, high porosity, stability, and customizable function (Zhang et al., 2020). The diameter of a nanofiber is smaller than one millimetre (Nasir et al., 2015). Electrospun nanofibers are developing into a versatile material with many uses thanks to their many desirable properties, include their small fibre diameter, adjustable porosity, affordability, high surface-to-volume ratio, capacity for structural manipulation, lightweight nature, and minimal energy requirements (Sharma et al., 2021). Their adaptability is demonstrated by different morphologies, such as hollow nanofiber tubes and nanoribbons, which can be generated from the same synthesis process by adjusting the relevant parameters.

Electrospinning is a straightforward and versatile methodology for producing nanofibers and other nanostructures with one-dimensional characteristics (Bolong and Saad, 2021). The production of fibres within an electrospinning configuration is significantly influenced by many parameters that can be categorised into three distinct groups: the procedural aspects, the composition of the solution, and the limitations imposed by the surrounding environment. The voltage, the tip and the collector distance, and the feeding rate are process parameters. Temperature and humidity are environmental characteristics, while viscosity, conductivity, volatility, molecule weight, and surface tension are examples of solution properties (Bhardwaj and Kundu, 2010).

Polymers like polytetrafluoroethylene (PTFE), polyether sulfone (PES), polyvinylidene difluoride (PVDF), and polyester (PET) are commonly employed for membrane separation, and they are typically hydrophobic (Pagliero et al., 2020). As a result of materials' inherent hydrophobicity, membrane fouling and membrane densification have become pressing issues (Tofighy et al., 2021). One potential approach to effectively mitigate membrane pollution is to enhance the hydrophilic properties of separation membranes (Zhang et al., 2020). The construction of nanocomposite membranes involves the amalgamation of nanoparticles and polymers, resulting in the formation of a rough and hydrophilic surface that improves the polymer membranes' mechanical characteristics, antibacterial and anti-fouling properties (Li et al., 2020). Researchers have experimented with adding additives and fillers to the polymeric solution blend to create more useful fibres. The nanocomposite membranes were designed to enhance the mechanical, anti-fouling, and antibacterial properties of polymer membranes by combining nanoparticles with polymers to create a durable and hydrophilic surface (Du et al., 2021; Zhang et al., 2020; Tofighy et al., 2021).

One filler material that has received much attention for its potential application in photocatalysis is titanium dioxide (TiO_2) (Abed and Waisi, 2024). To improve performance, it is recommended to use thinner fibres, which increases their surface area (Kaplan et al., 2016). By electrostatically spinning together two layers, Cheng et al. (2020) developed a composite nanofiber membrane that exhibits asymmetry. This membrane consists of a single layer of cross-linked active oligomers, forming a multi-hydrophilic functional network. The oil-water separation process exhibits a high efficiency of 99.2%, with a corresponding flux loss of approximately 2%. The membrane's hydrophilicity can be vastly enhanced by including certain hydrophilic polymers and inorganic nanoparticles. Acknowledging that the limited adhesive strength between the functional layer and the substrate membrane poses challenges regarding the hierarchical rough structure's ability to withstand the erosive and flushing impacts exerted by external forces (Lv et al., 2020; Ma et al., 2016).

Electrospun polyethersulfone (PES) fibre mats were shown to be promising ion-exchange materials after being sulfonated (Kwak et al., 2013). Electrospinning was used to create a composite of SiO_2-TiO_2-PVDF (Nasir et al., 2015). SEM analysis confirmed that the average diameter of SiO_2-TiO_2-PVDF nanofibers is 350 nm and that their surfaces are smooth with no beads on the nanofiber strings. The nanofiber membrane exhibits efficient oil-water separation capabilities owing to its notable characteristics, including a significantly large specific surface area, substantial porosity, and uniform distribution of apertures (Zhang et al., 2020). PVDF is particularly notable within the realm of polymers for its efficacy in membrane separation and membrane distillation due to its advantageous characteristics, including its affordability, exceptional chemical resistance, and remarkable thermal stability (Xiang et al., 2020). Electrostatic spinning produces a membrane that looks and feels like non-woven felt, with fibres loosely packed. This results in little mechanical strength and little practical use. Yuan et al. (2020) demonstrated that low surface energy makes it susceptible to oil contamination.

Polyvinylpyrrolidone (PVP) is a synthetic polymer characterised by its notable hydrophilic properties, rendering it highly soluble in aqueous PVP is commonly dissolved and subsequently eliminated using either non-solvent-induced phase separation (NIPS) or thermally induced phase separation (TIPS) techniques, thereby fulfilling the role of a pore-forming agent (Du et al., 2021; Ilyas et al., 2020). The modified membrane maintains respectable hydrophilic and antifouling performance even when exposed to prolonged external stress, as shown by Du et al. (2021). This occurs as a result of the PVP dissolving and roughening the fiber's surface, which causes the TiO_2 NPs to disperse randomly throughout the fiber. The membrane's mechanical qualities and endurance were enhanced by incorporating PVP and TiO_2, which also gave it stable hydrophilic and antifouling properties. The surface morphological changes, filtration performance and antifouling properties of electrospinning nanofiber membranes were observed and assessed by changing the quantity of the additives in the spinning fluid (Du et al., 2021).

Earlier researchers included PVP in the spinning solution to counteract the fibre breaking brought on by PVDF's high crystallinity (Jabbarnia et al., 2016). Electrostatic spinning fibre holes can also be made by immersing PVP. PVP was added to the polymeric solution to make up for the conductivity loss due to PVDF's high crystallinity, and it also improves the polymer's electro-spinnability as presented in (Jabbarnia et al., 2016) study. The remaining PVP enhances the hydrophilicity of the membrane in the fibre due to its abundance of carbonyl functional groups (Tofighy et al., 2021). These holes also facilitate the loading of inorganic nanoparticles like TiO_2 (Du et al., 2021). Using nano TiO_2 as a catalyst or catalyst carrier is highly promising owing to its exceptional attributes, such as elevated thermal stability, resistance to chemical degradation, and efficient photocatalytic properties (Lee et al., 2018). In contrast to other metal oxides at the nanoscale, titanium dioxide nanoparticles exhibit significant surface activity. They can serve as adsorption carriers due to a polar titanium-oxygen (Ti-O) bond. In addition, after absorbing water, they will ionize and create several hydroxyl groups due to polarization. A study by Venkatesh et al., (2020) conducted a study which revealed that incorporating TiO_2 nanoparticles (NPs) into a polymer membrane can improve its hydrophilicity, self-cleaning properties, resistance to fouling, and ability to inhibit bacterial growth. The preparation of hydrophilic and anti-fouling PVDF nanofiber composite membranes and a simple and practical method were disclosed. In this research, nanofiber PVDF-TiO_2-PVP membranes were created by electrospinning. Excellent mechanical characteristics and chemical resistance are two of PVDF's many notable features (Yang et al., 2020). Hydrophilic nanoparticles like titanium dioxide (TiO_2) have increased membrane wettability and anti-fouling effectiveness. In addition to its low cost, low toxicity, high hydrophilicity, and antibacterial properties, TiO_2 nanoparticles have been employed in membrane modification (Kaplan et al., 2016). PVP, a water-soluble polymer, can mechanically reinforce the membrane and simplify polymer solution processing. The membrane's mechanical qualities and endurance were enhanced by incorporating PVP and TiO_2, giving it stable hydrophilic and anti-fouling properties. Electrospun nanofiber membranes had their surface morphology, filtration efficiency, and anti-fouling property studied by varying the concentration of additives in the spinning solution. This research aimed to investigate the structure and morphology of PVDF-TiO_2-PVP nanofiber composites synthesized using electrospinning. As-prepared composite membranes' flux and separation efficiency were studied in depth. This study also aimed to evaluate the potential of the fabricated nanofiber membranes for effective wastewater treatment applications through performance assessment in filtration and antifouling tests.

MATERIALS AND METHODS

Polymer-based membranes made from polyvinylidene fluoride (PVDF) Kynar®740 pellets were purchased from Arkema Inc. USA. Merck provided N, N-dimethylacetamide (DMAc, >99%). Nanoparticles of titanium dioxide (Degussa P25) were shipped from Germany's Evonik GmbH. Bovine serum albumin (MW = 66,000 g/mol) and polyvinylpyrrolidone (PVP) (MW = 40,000) were procured by Sigma Aldrich.

Electrospun membrane fabrication

The electrospinning was used to create the nanofiber membranes. The PVDF pure and PVDF-TiO_2-PVP membrane dope solutions were created for this experiment. The dope solution was made by dissolving the PVP additive in the DMAc solvent and then adding the necessary amount of TiO_2 to the solution, after which the PVDF pellets were pre-dried (24 hours). After dissolving the PVDF (16 g), the solution was heated to 60 °C and agitated for 24 hours (Gayatri, et al., 2023b). To eliminate any last traces of air bubbles, the dope was ultrasonically agitated for half an hour after it had completely dissolved. Electrospinning machinery received the dope solution. Fibrous membranes were electrospun in this experiment at a voltage of 16 kV and a feeding rate of 1 ml/h, with the distance between the syringe needle tip and the filter membrane fixed at 10 cm. The membrane's BSA rejection, permeate flow, and pure water flux were measured in order to assess its efficiency.

Measurement of nanofiber membrane efficiency

Permeability tests were performed on the membranes in a flat sheet membrane testing device to determine their filtration performance. At a concentration of 1000 g/L, Bovine Serum Albumin was fed to the feed tank. The membrane was then placed in a membrane cell. After 5 minutes, the permeation volume was measured for 25 minutes at 1 bar (Du et al., 2021). The pure water flows in the experimental setup were determined using Equation 1 (Gayatri,, et al., 2023a):

$$J = \frac{Q}{A \times t} \quad (1)$$

where: J is the flux (L/m^{2}h), V is the permeate volume (L), t is the collection period (hour), and A is the membrane surface area (m^2).

$$R = 1 - \frac{Cp}{Cf} \times 100 \quad (2)$$

Permeate concentration (C_p) and feed concentration (C_F) in parts per million, and rejection rate (R) in percent. The content of BSA in the permeate and feed was measured using a UV-Vis spectrophotometer with a maximum wavelength of 278 nm.

Nanofiber membrane characterizations

Characterization was carried out on the recovered nanofibrous membranes. The membrane morphology was investigated using scanning electron microscopy (SEM). Polymer structural and functional group evolution can be visualized using FTIR. The surface morphology and topographical characteristics of membrane materials can be effectively characterized at the nanoscale using atomic force microscopy (AFM). The hydrophilicity of membranes was also evaluated using contact angle analysis. The Drop Metre A-100 contact angle system was used to measure the membrane's static contact angle in order to describe its wetting behavior.

RESULTS AND DISCUSSION

Nanofiber membrane characterization

Morphology of nanofiber membranes

In Figure 1, 800x and 1000x magnifications of the SEM result of the two produced nanofiber membranes were shown. Figure 1 demonstrates a pure PVDF nanofiber's rough surface and curled diameter. Hydrophobic polymers, like PVDF, have a negative reaction to water. For this reason, the PVDF membrane does not readily let the passage of water molecules (Tofighy et al., 2021). Nanofibers made of PVDF-TiO_2-PVP are straight, uniform, and beadless with a smooth surface. TiO_2 nanoparticles and PVP molecules are hydrophilic (water-loving), which explains why this is the case. These particles and molecules draw in water molecules, forming a hydrated layer on the membrane's surface. The membrane's surface area is increased, and its hydrophilicity is enhanced by the hydrated layer, making it more permeable to water molecules.

The optimal polymer concentration and spinning parameter have been attained on nanofiber production, corroborated by the finding that the surface morphology of composite nanofiber of SiO_2-TiO_2-PVDF was smooth without a bead on nanofiber copolymer string (Nasir et al., 2015) Electrospun fibres' low mechanical strength and susceptibility to fracture, when subjected to external forces, are caused by the stress concentration phenomenon caused by the fibre's beaded structure. Furthermore, a wide aperture distribution of those holes will result from an unequal fibre diameter distribution. As a result, electrospun fibres must have a uniform and orderly morphology (Du et al., 2021).

The surface roughness of electrospun fibres is positively correlated with the PVP content, as PVP exhibits high water solubility. Consequently, the dissolution of PVP occurs during the soaking process in clean water, thereby contributing to the increased roughness, as studied by Du et al. (2021). The average diameter of PVDF-TiO_2-PVP nanofibers was 65.9 nm, while that of pure PVDF

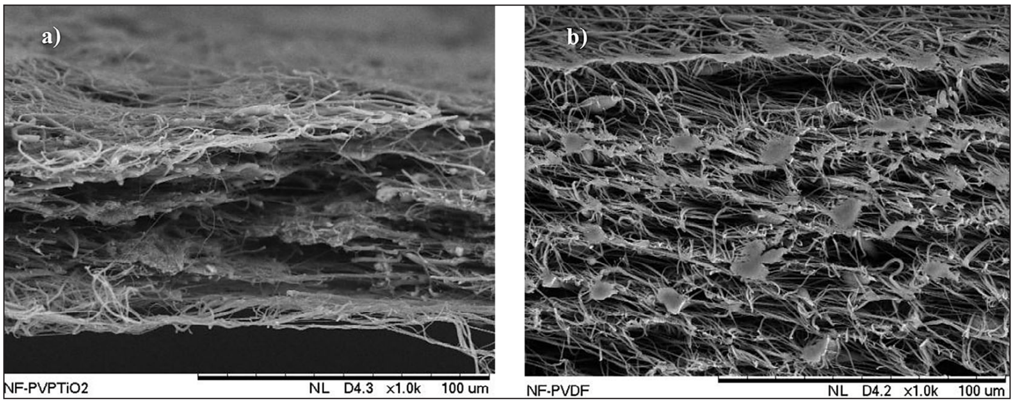


Figure 1. SEM Results a) PVDF-TiO_2-PVP nanofiber composite membrane and b) Pristine PVDF membrane

was 200 nm. Nanofibers with consistent diameter and ordered morphology can be produced by regulating factors like polymer content and flow rate (Sharma et al., 2021). The nanofibers' diameter grows in step with the polymer concentration.

FTIR of nanofiber membranes

Figure 2 presents the FTIR spectra of PVDF and PVDF-TiO_2-PVP nanofiber membranes, which reveal important details about the

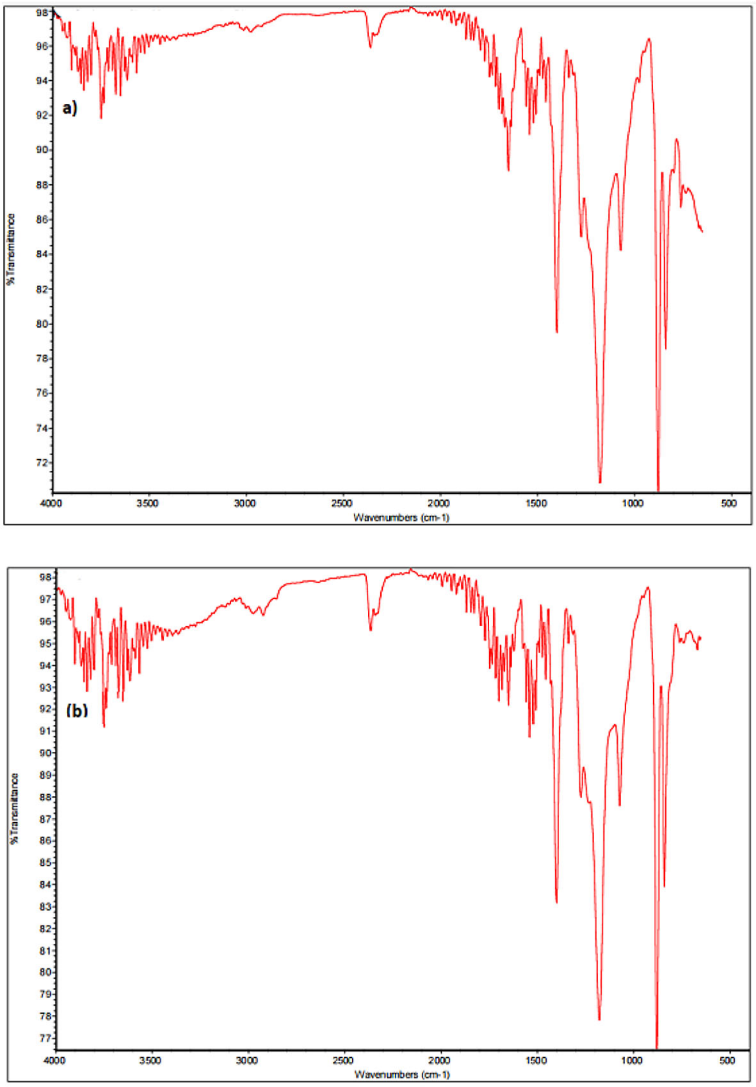


Figure 2. FTIR Spectra of (a) PVDF-TiO_2-PVP nanofiber; (b) PVDF Pristine nanofiber

membranes' chemical composition and structure. Knowledge of these membranes' qualities will aid in developing new membranes with specialized characteristics for various uses. The following distinctive peaks can be seen in th PVDF and PVDF-TiO_2-PVP nanofiber membranes FTIR spectra. Absorption bands were found at 1400, 1170, and 870 cm^{-1} for the pure PVDF and PVDF-TiO_2-PVP hybrid membranes. These absorption bands are commonly cited as the PVDF crystal's signature spectra (Ma et al., 2016). C-F bonds in the PVDF cause peaks at 1400 cm^{-1}, 1274 cm^{-1}, and 1179 cm^{-1} (Gayatri, et al., 2023a; Tian and Ma, 2020). The C-H linkages in the PVDF polymer cause the peak at 877 cm^{-1} (Nasir et al., 2015). The PVP polymer displays a peak at 840 cm^{-1} due to the stretching vibration of the -CH_2 group. The -OH stretching vibration of the PVP polymer is responsible for the peak at 1065–1070 cm^{-1}. The CO stretching vibrational peak in the TiO_2 nanoparticles causes the peak at 1650 cm^{-1}. The presence of PVP inside the composite membrane architecture is evidenced by the observation of three distinct peaks at wavenumbers 1674 cm^{-1}, 1399 cm^{-1}, and 1241 cm^{-1}, which correspond to the stretching vibrations of the carbonyl (C=O), carbon-hydrogen (C-H), and carbon-nitrogen (C-N) functional groups, respectively. These spectral features align with the expected vibrational modes of PVP (Ghobadi Moghadam and Hemmati, 2023). The peaks created in the spectrum of a PVDF-TiO_2-PVP membrane are almost in the same place as those formed in a spectrum of pure PVDF. The minute quantities of PVP and TiO_2 in the membrane matrix account for this occurrence.

These peaks verify that PVDF, PVP, and TiO_2 are present in the nanofiber membranes. The amounts of each component in the membranes can be estimated from the peaks' relative strengths. Due to molecular chain tangles during the electronic blending spinning process, PVP and PVDF are distributed randomly throughout the fibre. According to prior research by Du et al. (Du et al., 2021), the lingering PVP serves as an internal binder, strengthening the nanofiber membrane's resistance to water.

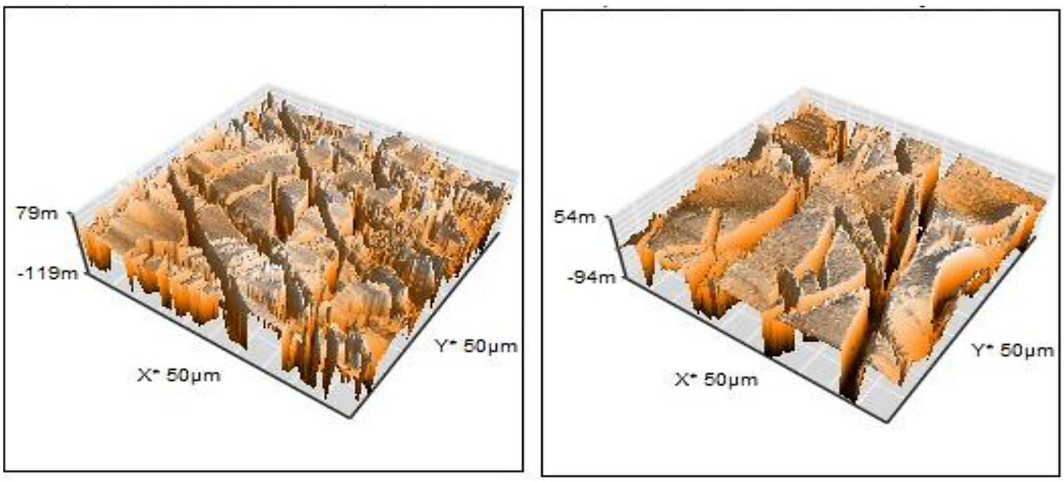
FTIR spectra of PVDF-TiO_2-PVP composite nanofibers show the significant spectral modifications are typically observed – such as broadening around 3400 cm^{-1} (O–H stretching from TiO_2 or PVP), a new band near 1660 cm^{-1} (C=O stretching of PVP), and potential shifts in the C–F bands—confirming successful incorporation and interaction of TiO_2 nanoparticles and PVP within the PVDF matrix. These shifts and intensity changes reflect possible hydrogen bonding, coordination interactions, or physical entrapment of additives, which can modulate crystallinity, hydrophilicity, and functional performance of the nanofibrous membranes. Thus, FTIR serves as a critical tool for verifying composite formation and understanding molecular-level interactions among the membrane constituents.

AFM of nanofiber membranes

Atomic Force Microscopy (AFM) provides critical insights into the nanoscale surface morphology of electrospun PVDF-TiO_2-PVP nanofiber membranes. Mean surface roughness (Ra) and surface AFM images of PVDF-TiO_2-PVP and PVDF pristine nanofiber membrane are shown in Figure 3. The dark area in a 3D AFM image indicates troughs, while the bright area shows the highest point on the membrane surface (Arif et al., 2019). The comparative AFM analysis of pristine PVDF nanofiber membranes (Figure 3b) and PVDF-TiO_2-PVP composite membranes (Figure 3a) reveals a significant transformation in surface topography upon the incorporation of TiO_2 nanoparticles and PVP. The pristine PVDF membrane exhibits a relatively smoother and less complex surface profile, with height variations ranging from –94 nm to +54 nm, indicating moderate nanoscale roughness and a more uniform fiber distribution. In contrast, the PVDF-TiO_2-PVP membrane (Figure 3a) shows a markedly more rugged surface, with height fluctuations extending from –119 nm to +79 nm, and a noticeably denser and more heterogeneous topographical pattern.

The surface roughness, quantified as the root mean square roughness (Sq) of PVDF-TiO_2-PVP nanofiber membranes, was measured to be 558.48 nm, while the pure PVDF membrane root mean square roughness (Sq) was only 489.05nm. This relatively high Sq value reflects the presence of micro- and nanoscale features, likely arising from heterogeneous fiber diameters, bead formation, and TiO_2 nanoparticle agglomeration on or within the PVDF-PVP matrix. Roughness at this scale can enhance membrane functionalities such as surface wettability and active surface area, all of which are critical for applications in filtration and catalysis (Arif et al., 2019).

This increase in surface roughness is attributable to the successful integration of TiO_2



(a) (b)

Figure 3. AFM analysis of (a) PVDF-TiO_2-PVP nanofiber; (b) PVDF Pristine nanofiber

nanoparticles and the phase interaction with PVP, which likely disrupted the uniformity of the electrospun fibers and introduced additional surface features. Applications requiring more surface area, like membrane filtration, photocatalysis, and biomolecule adsorption, benefit from this morphological improvement. The enhanced roughness of the composite membrane may also contribute to improved hydrophilicity and fouling resistance, as supported by previous studies that correlate topographical complexity with functional performance. These findings underscore the synergistic effects of TiO_2 and PVP in tuning the nanoscale architecture of PVDF membranes for advanced application potential.

In comparison to previous studies, our measured roughness is notably higher. For instance, (Arif et al., 2019) reported an Sq value of ~250 nm for electrospun PVDF membranes without TiO_2, suggesting that the incorporation of TiO_2 nanoparticles significantly contributes to topographical complexity. The findings show that surface roughness decreases as TiO_2 concentration rises, while the PVDF membrane with the highest loading has the maximum roughness, which is probably caused by aggregated particles (Zhao et al., 2013).

As can be observed from Tofighy et al., (2021) study, The pristine PVDF membrane has a Ra of roughly 64.3 nm and high hydrophobic properties. The PVDF matrix's surface roughness is successfully increased by the addition of GONRs and PVP. With a Ra of roughly 196.3 nm, the PVDF/(0.5GONRs)/PVP (M7) nanocomposite membrane had the maximum surface roughness. The prepared nanocomposite membrane becomes rougher as a result of the synergistic effects of GONRs and PVP on hydrophilicity enhancement, which raise the non-solvent and solvent exchange rate during the phase inversion process (Zhao et al., 2017). Higher roughness can directly increase the flux of pure water by improving the filtration area's efficiency (Tofighy et al., 2021).

The present work, employing a mixed matrix system (PVDF-TiO_2-PVP), appears to promote synergistic interactions among the components, contributing to enhanced phase separation and rougher surface morphology. Additionally, PVP being a hydrophilic polymer may induce phase segregation during electrospinning, further enhancing roughness via differential solidification dynamics. This elevated surface roughness can be advantageous for membrane-based applications. Specifically, in hydrophilic/hydrophobic balance tuning, a rougher surface can facilitate improved water flux and fouling resistance, as described by Ahmad et al., (2022), who linked nanomembrane roughness to antifouling performance. Furthermore, the heterogeneous topography may serve as a platform for enhanced photocatalytic activity, where surface roughness increases the light-scattering capability and active site availability of TiO_2. According to surface morphology, a rougher surface has a higher tendency for fouling, whereas a smoother surface is less likely to clog with the foulant (Zhao et al., 2016).

In hydrophilic membranes, high surface roughness creates voids and increases the membrane surface area, which improves the antifouling and pure water flux. Conversely, high surface roughness results in poor antifouling qualities. The ultrafiltration membrane's permeability and anti-fouling qualities are enhanced by its hydrophilicity (Ahmad et al., 2022). In conclusion, the AFM analysis of the PVDF-TiO_2-PVP membrane indicates a significantly roughened surface compared to PVDF-only or binary composites. The synergistic impact of TiO_2 and PVP enhances not only the morphological complexity but potentially the membrane's performance in real-world applications.

Contact angle measurement of nanofiber membranes

Due to its simplicity and ease of use, water contact angle measurement is far more commonly utilized in practical applications to assess the hydrophilicity of membrane surfaces (Ahmad et al., 2022). The contact angle measurements (Figure 4) demonstrate a marked improvement in the hydrophilicity of the PVDF-TiO_2-PVP nanofiber membrane compared to the pristine PVDF membrane. The experimental findings indicate that the contact angle of the PVDF-TiO_2-PVP membrane measured 118 degrees, whereas the contact angle of the pure PVDF membrane was determined to be 134 degrees. Pure electrospun PVDF membranes are highly hydrophobic since their chemical structure comprises C-F linkages and no hydrophilic groups (Du et al., 2021). Hydrophobic PVDF, a polymer with a low affinity for water, is to blame for this. This suggests that the membrane's surface can become more hydrophilic by incorporating TiO_2 nanoparticles into the PVDF material (Gayatri, et al., 2023b). Since TiO_2 nanoparticles have a high surface energy and may interact with water molecules, they are responsible for the enhanced hydrophilicity of PVDF-TiO_2-PVP nanofiber membranes. Hydrophilicity can also be bestowed upon the fiber membrane by adding PVP.

The PVDF-TiO_2-PVP nanofiber membranes have enhanced hydrophilicity, making them more applicable in fields requiring a high-water flux, such as water purification. A membrane's wettability can be evaluated by measuring its contact angle, which is why it is an essential feature. A membrane that exhibits hydrophobic properties is characterised by a significantly elevated contact angle, resulting in the repulsion of water molecules. Conversely, a hydrophilic membrane displays a notably reduced contact angle, attracting water molecules towards its surface. As a result of their elevated contact angle, membranes made from electrospun PVDF and PVDF-TiO_2-PVP fibres are perfect for uses that call for water repellency. Better antifouling qualities can be achieved using a membrane surface that is more hydrophilic (Ma et al., 2016).

The pure PVDF membrane exhibits a high contact angle of approximately 134°, indicating a strongly hydrophobic surface, which is characteristic of PVDF due to its non-polar fluorinated structure. Upon incorporation of hydrophilic PVP and TiO_2 nanoparticles, the composite membrane contact angle significantly decreases to 118° reflecting enhanced surface wettability. This improvement can be attributed to the existance of polar functional groups from PVP and the intrinsic hydrophilic properties of TiO_2, which introduce hydrogen bonding sites and surface oxygen species capable of interacting with water molecules.

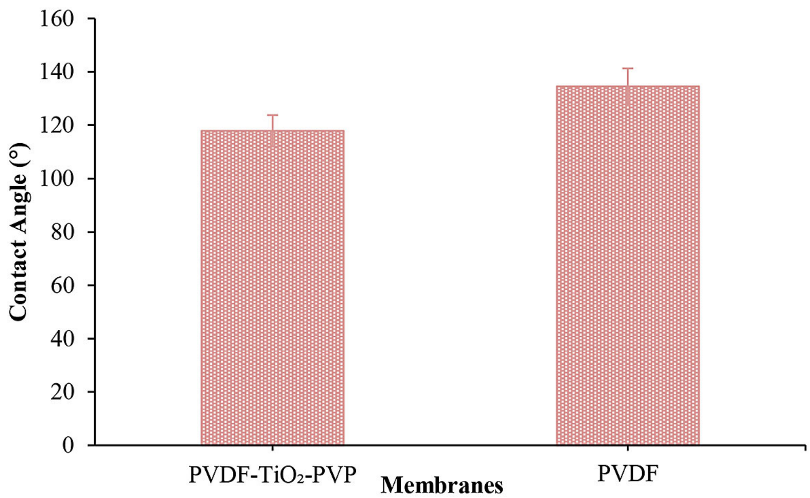


Figure 4. Nanofiber membranes contact angle

Enhanced hydrophilicity is crucial in membrane applications, as it promotes water permeation, reduces the likelihood of pore blockage, and minimizes fouling caused by organic and biological contaminants. These results are consistent with the observed increases in BSA flux and reduced flux decline, confirming that surface modification through TiO_2 and PVP incorporation effectively tailors the membrane interface for superior filtration performance. Such hydrophilic enhancement is highly desirable in next-generation membranes targeting efficient and sustainable water purification technologies.

Evaluation of nanofiber membrane function

The flux performance comparison between pristine PVDF and PVDF-TiO_2-PVP nanofiber membranes reveals a significant enhancement in both pure water flux and bovine serum albumin (BSA) solution flux for the modified membrane. Figure 5 portrays the water flux and BSA flux measurements used to illustrate the permeability performance results provided. Water flux and BSA flux were positively correlated, with the PVDF-TiO_2-PVP membrane outperforming the PVDF Pristine membrane in both cases.

PVDF-TiO_2-PVP membrane maintains a high-water flux of 1806 L/m^2h and demonstrates a notably improved BSA flux of 1694 L/m^2h, reflecting its superior antifouling properties. In contrast, the pristine PVDF membranes have a water flux of about 1715 L/m^2h, which decreases to around (1245 L/m^2h) when filtering BSA, indicating a considerable flux decline due to fouling and protein adsorption.

The synergistic effect is responsible for this performance enhancement of TiO_2 nanoparticles and hydrophilic PVP, which enhance surface hydrophilicity and reduce protein adhesion. Additionally, the increased surface roughness and porosity observed in AFM analysis likely contribute to better permeation characteristics and reduced pore blocking. The reduced flux decline upon BSA filtration in the PVDF-TiO_2-PVP membrane highlights its potential in high-performance separation processes, especially in protein-rich wastewater or biomedical filtration applications. These findings demonstrate the effectiveness of nanocomposite engineering in tailoring membrane surface properties for enhanced filtration efficiency and fouling resistance, aligning with the design criteria for next-generation ultrafiltration membranes.

Increased surface hydrophobicity leads to a drastic decrease in water permeability, which follows the same pattern as the contact angle result (Bolong and Saad, 2021). TiO_2 was observed to boost the membrane's hydrophilicity, which in turn raised the membrane's water flux and BSA flux, as previously mentioned in the study.

The membrane composed of PVDF-TiO_2-PVP exhibits a reduced rejection rate (4.42%) for BSA compared to the pure PVDF membrane (8.55%), as illustrated in Figure 6. This facilitated the obstruction of the pores of the PVDF membrane by BSA. The thick surface can increase filtration resistance and retain more BSA simultaneously (Gayatri, et al., 2023a; Gebru and Das, 2017).

The high pure water flux of PVDF-TiO_2-PVP membranes is influenced by a number of factors. First, water molecules can interact with

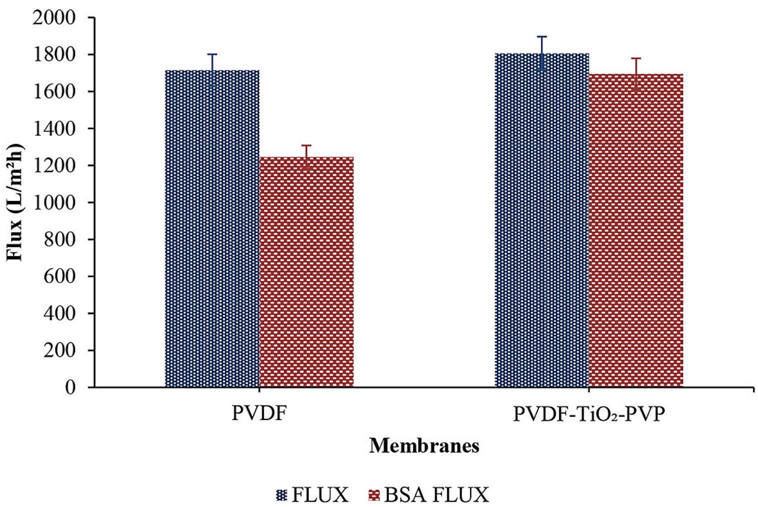


Figure 5. Water flux and BSA flux of nanofiber membranes

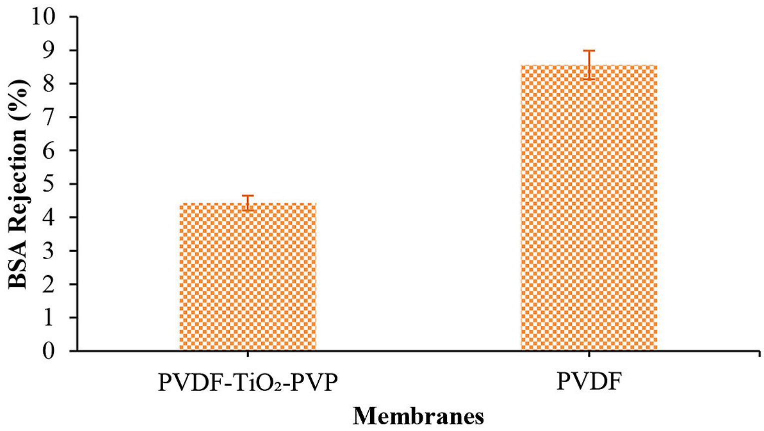


Figure 6. BSA rejection of nanofiber membranes

the membrane thanks to the hydrophilic surface that the TiO_2 nanoparticles give. Furthermore, the membrane's PVP polymer opens up the pores, which makes it easier for water molecules to move through them. This study demonstrates the feasibility of using nanofiber electrospun PVDF-TiO_2-PVP membranes in applications demanding high water flux and BSA rejection.

CONCLUSIONS

This present study demonstrates that electrospinning is an effective method for fabricating PVDF–TiO_2–PVP nanofibrous membranes with enhanced wettability, antifouling properties, and separation performance. Optimized membranes containing 2 wt% PVP and 1 wt% TiO_2 exhibited uniform morphology and improved hydrophilicity, making them appropriate for a variety of uses, such as wastewater purification, water treatment, and biomedical diagnostics. These findings highlight the potential of TiO_2- and PVP-modified PVDF membranes as multifunctional platforms for advanced filtration technologies. SEM confirmed the formation of uniform, bead-free, and continuous nanofibers in the PVDF–TiO_2–PVP composite membranes. FTIR validated the successful incorporation of PVDF, PVP, and TiO_2 through the identification of characteristic functional group vibrations. The inclusion of TiO_2 nanoparticles and PVP significantly enhanced PVDF nanofibers surface hydrophilicity, indicating their potential for advanced membrane-based separation and filtration applications. AFM analysis of the PVDF-TiO_2-PVP membrane indicates a significantly roughened surface compared to PVDF-pristine. The PVDF-TiO_2-PVP membrane outperformed the PVDF Pristine membrane in terms of flux (water flux of 1806 L/m^2h and a BSA flux of 1694 L/m^2h). This opens up a wide range of potential uses for the resulting fibers, including water purification and separating biomolecules like protein.

Acknowledgements

The authors acknowledge Universitas Sriwijaya.

REFERENCES

1. Ahmad, N. N. R., Mohammad, A. W., Mahmoudi, E., Ang, W. L., Leo, C. P., Teow, Y. H. (2022). An overview of the modification strategies in developing antifouling nanofiltration membranes. In *Membranes 12*(12). MDPI. https://doi.org/10.3390/membranes12121276
2. Abed, I. A., Waisi, B. I. (2024). Performance enhancement of polyethersulfone-based ultrafiltration membrane decorated by titanium dioxide nanoparticles for dye filtration. *Ecological Engineering and Environmental Technology*, *25*(5), 265–273. https://doi.org/10.12912/27197050/186182
3. Arif, Z., Sethy, N. K., Kumari, L., Mishra, P. K., Verma, B. (2019). Antifouling behaviour of PVDF/TiO_2 composite membrane: a quantitative and qualitative assessment. *Iranian Polymer Journal (English Edition)*, *28*(4), 301–312. https://doi.org/10.1007/s13726-019-00700-y
4. Bai, W., Chen, K., Chen, J., Xu, J., Lin, H., Lin, Y., Xu, Y., Lin, J. (2020). Natural Highly-hydrophobic urushiol@TiO_2 coated cotton fabric for effective oil–water separation in highly acidic alkaline and salty environment. *Separation and Purification Technology*, *253*. https://doi.org/10.1016/j.

seppur.2020.117495

5. Bhardwaj, N., Kundu, S. C. (2010). Electrospinning: A fascinating fiber fabrication technique. In *Biotechnology Advances* 28(3), 325–347. https://doi.org/10.1016/j.biotechadv.2010.01.004
6. Bolong, N., Saad, I. (2021). Optimizing fabrication of electrospinning nanofiber membranes for water filtration using response surface methodology. *Jurnal Kejuruteraan*, *33*(3), 763–771. https://doi.org/10.17576/jkukm-2021-33(3)-34
7. Cheng, X., Sun, Z., Yang, X., Li, Z., Zhang, Y., Wang, P., Liang, H., Ma, J., Shao, L. (2020). *Supporting information In Situ Asymmetry Engineering Constructing Superhydrophilic Hierarchical Polyacrylonitrile Nanofiber Membrane for Unprecedently Ultrafast Oil-Water Emulsion Separation*.
8. Du, C., Wang, Z., Liu, G., Wang, W., Yu, D. (2021). One-step electrospinning PVDF/PVP-TiO_2 hydrophilic nanofiber membrane with strong oil-water separation and anti-fouling property. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, *624*. https://doi.org/10.1016/j.colsurfa.2021.126790
9. Gayatri, R., Fizal, A. N. S., Yuliwati, E., Hossain, M. S., Jaafar, J., Zulkifli, M., Taweepreda, W., Ahmad Yahaya, A. N. (2023a). Preparation and characterization of PVDF–TiO_2 mixed-matrix membrane with PVP and PEG as pore-forming agents for BSA rejection. *Nanomaterials*, *13*(6), 1023. https://doi.org/10.3390/nano13061023
10. Gayatri, R., Yuliwati, E., Fizal, A. N. S., Zailani, M. Z., Jaafar, J., Zulkifli, M., Taweepreda, W., Yahaya, A. N. A. (2023b). Effect of TiO_2 concentration in PVDF-TiO_2-PVP mixed matrix membrane performance using ultrafiltration process. *Materials Today: Proceedings*, 1–8.
11. Gebru, K. A., Das, C. (2017). Removal of bovine serum albumin from wastewater using fouling resistant ultrafiltration membranes based on the blends of cellulose acetate, and PVP-TiO_2 nanoparticles. *Journal of Environmental Management*, *200*, 283–294. https://doi.org/10.1016/j.jenvman.2017.05.086
12. Ghobadi Moghadam, A., Hemmati, A. (2023). Improved water purification by PVDF ultrafiltration membrane modified with GO-PVA-NaAlg hydrogel. *Scientific Reports*, *13*(1), 8076. https://doi.org/10.1038/s41598-023-35027-5
13. Ilyas, A., Mertens, M., Oyaert, S., Vankelecom, I. F. J. (2020). Synthesis of patterned PVDF ultrafiltration membranes: Spray-modified non-solvent induced phase separation. *Journal of Membrane Science*, *612*. https://doi.org/10.1016/j.memsci.2020.118383
14. Jabbarnia, A., Khan, W. S., Ghazinezami, A., Asmatulu, R. (2016). Investigating the thermal, mechanical, and electrochemical properties of PVdF/PVP nanofibrous membranes for supercapacitor applications. *Journal of Applied Polymer Science*, *133*(30). https://doi.org/10.1002/app.43707
15. Kaplan, R., Erjavec, B., Dražić, G., Grdadolnik, J., Pintar, A. (2016). Simple synthesis of anatase/rutile/brookite TiO_2 nanocomposite with superior mineralization potential for photocatalytic degradation of water pollutants. *Applied Catalysis B: Environmental*, *181*, 465–474. https://doi.org/10.1016/j.apcatb.2015.08.027
16. Kwak, N. S., Jung, W. H., Park, H. M., Hwang, T. S. (2013). Electrospun polyethersulfone fibrous mats: Sulfonation, its characterization and solution-phase ammonium sorption behavior. *Chemical Engineering Journal*, *215–216*, 375–382. https://doi.org/10.1016/j.cej.2012.10.065
17. Lee, C. G., Javed, H., Zhang, D., Kim, J. H., Westerhoff, P., Li, Q., Alvarez, P. J. J. (2018). Porous electrospun fibers embedding TiO_2 for adsorption and photocatalytic degradation of water pollutants. *Environmental Science and Technology*, *52*(7), 4285–4293. https://doi.org/10.1021/acs.est.7b06508
18. Li, D., Sun, X., Gao, C., Dong, M. (2020). Improved water flux and antifouling properties of cardo poly(aryl ether ketone) ultrafiltration membrane by novel sulfobetaine polyimides additive. *Separation and Purification Technology*, *251*. https://doi.org/10.1016/j.seppur.2020.117144
19. Lv, Y., Ding, Y., Wang, J., He, B., Yang, S., Pan, K., Liu, F. (2020). Carbonaceous microsphere/nanofiber composite superhydrophilic membrane with enhanced anti-adhesion property towards oil and anionic surfactant: Membrane fabrication and applications. *Separation and Purification Technology*, *235*. https://doi.org/10.1016/j.seppur.2019.116189
20. Ma, W., Rajabzadeh, S., Shaikh, A. R., Kakihana, Y., Sun, Y., Matsuyama, H. (2016). Effect of type of poly(ethylene glycol) (PEG) based amphiphilic copolymer on antifouling properties of copolymer/poly(vinylidene fluoride) (PVDF) blend membranes. *Journal of Membrane Science*, *514*, 429–439. https://doi.org/10.1016/j.memsci.2016.05.021
21. Nasir, M., Juliandri, Prihandoko, B. (2015). Fabrication of SiO_2-TiO_2/PVDF copolymer nanofiber composite by electrospinning process. *Procedia Chemistry*, *16*, 184–189. https://doi.org/10.1016/j.proche.2015.12.034
22. Pagliero, M., Bottino, A., Comite, A., Costa, C. (2020). Novel hydrophobic PVDF membranes prepared by nonsolvent induced phase separation for membrane distillation. *Journal of Membrane Science*, *596*. https://doi.org/10.1016/j.memsci.2019.117575
23. Sharma, A., Pathak, D., Patil, D. S., Dhiman, N., Bhullar, V., Mahajan, A. (2021). Electrospun PVP/TiO_2 nanofibers for filtration and possible protection from various viruses like COVID-19. *Technologies*,

9(4). https://doi.org/10.3390/technologies9040089
24. Tian, Y., & Ma, H. (2020). Solvent-free green preparation of reusable EG-PVDF foam for efficient oil-water separation. *Separation and Purification Technology*, *253*. https://doi.org/10.1016/j.seppur.2020.117506
25. Tofighy, M. A., Mohammadi, T., Sadeghi, M. H. (2021). High-flux PVDF/PVP nanocomposite ultrafiltration membrane incorporated with graphene oxide nanoribbones with improved antifouling properties. *Journal of Applied Polymer Science*, *138*(4). https://doi.org/10.1002/app.49718
26. Venkatesh, K., Arthanareeswaran, G., Bose, A. C., Kumar, P. S. (2020). Hydrophilic hierarchical carbon with TiO_2 nanofiber membrane for high separation efficiency of dye and oil-water emulsion. *Separation and Purification Technology*, *241*. https://doi.org/10.1016/j.seppur.2020.116709
27. Xiang, S., Guo, Z., Wang, Y., Liu, H., Zhang, J., Cui, Z., Wang, H., Li, J. (2020). The microstructure regulation, strengthening, toughening and hydrophilicity of polyamide6 in fabricating poly (vinylidene fluoride)-based flat membrane via the thermally induced phase separation technique. *European Polymer Journal*, *126*. https://doi.org/10.1016/j.eurpolymj.2020.109568
28. Yang, J., Wang, L., Xie, A., Dai, X., Yan, Y., Dai, J. (2020). Facile surface coating of metal-tannin complex onto PVDF membrane with underwater Superoleophobicity for oil-water emulsion separation. *Surface and Coatings Technology*, *389*. https://doi.org/10.1016/j.surfcoat.2020.125630
29. Ying, T., Su, J., Jiang, Y., Ke, Q., Xu, H. (2020). A pre-wetting induced superhydrophilic/superlipophilic micro-patterned electrospun membrane with self-cleaning property for on-demand emulsified oily wastewater separation. *Journal of Hazardous Materials*, *384*. https://doi.org/10.1016/j.jhazmat.2019.121475
30. Yuan, X. T., Xu, C. X., Geng, H. Z., Ji, Q., Wang, L., He, B., Jiang, Y., Kong, J., Li, J. (2020). Multifunctional PVDF/CNT/GO mixed matrix membranes for ultrafiltration and fouling detection. *Journal of Hazardous Materials*, *384*. https://doi.org/10.1016/j.jhazmat.2019.120978
31. Zhang, G., Zhan, Y., He, S., Zhang, L., Zeng, G., Chiao, Y. H. (2020). Construction of superhydrophilic/underwater superoleophobic polydopamine-modified h-BN/poly(arylene ether nitrile) composite membrane for stable oil-water emulsions separation. *Polymers for Advanced Technologies*, *31*(5), 1007–1018. https://doi.org/10.1002/pat.4835
32. Zhang, Y., Hu, Y., Zhang, L., Wang, Y., Liu, W., Ma, C., Liu, S. (2020). Porous SiO_2 coated AlxFeyZr1-x-yO_2 solid superacid nanoparticles with negative charge for polyvinylidene fluoride (PVDF) membrane: Cleaning and partial desalinating seawater. *Journal of Hazardous Materials*, *384*. https://doi.org/10.1016/j.jhazmat.2019.121471
33. Zhao, C., Xu, X., Chen, J., Yang, F. (2013). Effect of graphene oxide concentration on the morphologies and antifouling properties of PVDF ultrafiltration membranes. *Journal of Environmental Chemical Engineering*, *1*(3), 349–354. https://doi.org/10.1016/j.jece.2013.05.014
34. Zhao, L., Zhang, M., He, Y., Chen, J., Hong, H., Liao, B. Q., Lin, H. (2016). A new method for modeling rough membrane surface and calculation of interfacial interactions. *Bioresource Technology*, *200*, 451–457. https://doi.org/10.1016/j.biortech.2015.10.055
35. Zhao, X., Li, J., Liu, C. (2017). Improving the separation performance of the forward osmosis membrane based on the etched microstructure of the supporting layer. *Desalination*, *408*, 102–109. https://doi.org/10.1016/j.desal.2017.01.021