

Enhancing the Performance of Electrospun Nanofiber Membranes in Water and Wastewater Treatment Processes Using Omniphobic Surface Modification

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Abstract

The necessity for advantageous advancements in filtering technology has resulted in limited attention towards emerging materials, specifically nanofiber membranes, for water purification. The issue of organic material and the collection of residual organics in wastewater is a significant challenge since existing treatments like coagulation/flocculation and chlorine treatment have proven insufficient in achieving satisfactory outcomes. Further treatment and disposal steps are required for the additional amount of sludge produced by these methods. The field of nanotechnology has remarkable promise in filtration applications owing to its capacity to fabricate materials with exact structural control to meet specific needs. Membrane distillation (MD) has emerged as a potentially viable method for treating industrial wastewater. However, the existing hydrophobic MD membranes encounter substantial challenges related to wetting, mostly caused by the presence of pollutants often found in wastewater. This work presents a novel approach for the production of a Poly Vinylidene Fluoride - Hexa Fluoro Propylene Electrospun Nanofiber Membrane (PVF-HFP-ENM) that exhibits enhanced resistance to wetting by reduced surface-tension materials. The PVF-HFP-ENM membrane, which possesses an inherent re-entrant structure, has been fluorinated using 1H, 1H, 2H, 2H-Per Fluoro Decyl Trichlorosilane (PFDT) by Vapor Deposition (VD) without the need for surface activation. The fluorinated membrane had exceptional surface omniphobicity, as evidenced by its high contact angles with water and ethanol, measuring $148 \pm 0.4^\circ$ and $120 \pm 0.8^\circ$, respectively. The fluorinated membrane exhibited exceptional stability regarding omniphobicity and mechanical qualities, even when subjected to extreme circumstances such as ultrasonic disinfection, boiling water, and acidic and base exposure. The present work focuses on utilizing ENM in the context of industrial wastewater treatment and the alteration of nanomembranes to mitigate clogging problems and enhance the efficiency of wastewater treatment processes.

Keywords: Membrane distillation, Electrospun Nanofiber Membrane, omniphobicity, surface modification, water treatment.

Introduction

Humans use less than 1% of global freshwater resources. Most of Earth's freshwater is in icecaps, glaciers, and mountains. Freshwater makes up 2.6% of Earth's water, while saltwater makes up 97.5%. According to the source, irrigation uses 72% of freshwater, domestic consumption 8%, and businesses 21% [1]. Safe drinking water is unavailable to 1.3 billion people worldwide. Around 1.8 million people die from diarrheal diseases caused

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by contaminated water yearly. By 2052, the global population will grow by over 3.1 billion. About 2.8 billion people in developing nations suffer economic consequences from inadequate water resources [2].

Every person is entitled to clean water, essential to public health and sustainable development. Pollution, overexploitation, and climate change threaten this vital resource. Additionally, polluted wastewater releases into natural aquatic systems pose ecological and health risks. Advanced water and wastewater treatment materials and technology are needed to address these issues. Electrospinning, a flexible manufacturing method, is increasingly used to make nanofiber membranes for water treatment [3].

An electric field is applied to a polymer solution or melt to create nanofibers gathered into a non-woven membrane. The above procedure allows precise regulation of membrane fiber diameter, porosity, and surface chemistry, making it versatile for water treatment. Different methods have been used to reduce water contaminants. Some solutions are effective and suitable but have limits [4].

The growing global need for clean, drinkable water and effective wastewater treatment has spurred research into new water purification materials and technologies [5]. Due to their large surface area, adjustable pore size, and easy manufacturing, ENMs are considered a solution. Nanofiber membranes show promise in filtering and separation applications, offering a solution to water scarcity and environmental contamination. Fouling, low flow rates, and chemical resilience have hindered the implementation of these concepts.

ENMs purify water from textile waste like dyes, pigments, and colorants. ENMs remove dust, sand, and particulates and treat effluents. ENMs are essential for water desalination, heavy metal removal, pathogen elimination, and microorganism and bacteria reduction [6]. ENM selection depends on water contaminants. The nanomembranes discussed are efficient and come in polyvinylidene fluoride, nylon-6, polyacrylonitrile, and functional polymeric membranes.

Fouling and accumulating organic substances, minerals, or microbes on the membrane surface reduces ENM efficacy. Membrane fouling reduces permeate flux, increases energy use, and increases repair and replacement needs. These factors prevent the widespread use of these membranes for water treatment. Scholars have used omniphobic surface modification methods to reduce fouling in ENM [7]. Omniphobic surfaces resist aqueous solutions, oils, and organic solvents. Surface chemistry and micro/nanostructuring prevent liquid droplets and impurities from sticking to the surface, causing repellent behavior.

This study aids the development of sustainable and effective water and wastewater treatment methods. It addresses global water issues and promotes environmental stewardship. The following sections will examine the concepts, methodology, and experimental methods used to alter omniphobic surfaces. After presenting the results, the implementation implications will be discussed.

Related Works

Researchers have recently focused on surface modification methods to overcome these obstacles and improve ENM. Omniphobic surface modification research is promising. This method makes membrane surfaces repel many liquids, including oils, organic solvents, and aqueous solutions. This method increases membrane fouling resistance and simplifies mixture separation, opening new water and wastewater treatment possibilities.

Wu et al. (2022) create a PVDF/MAF-4 composite membrane for membrane distillation. Implementation requires a composite membrane engineered for high flux and scaling resistance [8]. The defined membrane performs better in membrane distillation. It improves scaling resistance and water flow. This method improves membrane efficiency. It's important to consider the drawbacks. These include scaling up production issues and cost concerns.

Asadolahi and Fashandi (2023) used a durable, nanoparticle-free, single-step omniphobic alteration on Poly (vinylidene fluoride) electrospun membranes to improve membrane distillation. The method involves modifying the membrane surface to make it omniphobic [9]. The membrane is modified and tested for durability and distillation performance. The output values include the membrane with enhanced omniphobic properties and membrane distillation potential.

Zhou et al. (2021) systematically examine high-flux electrospun nanofiber membrane distillation methods for aquaculture wastewater treatment. A comprehensive review and concise synthesis of pre-existing scholarly literature on high-flux membrane distillation methods using electrospun nanofibers is suggested [10]. Implementation involves collecting and analyzing research data to determine high-flux approach efficacy. The output values examine high-flux methods and their use in aquaculture wastewater treatment with ENMs. Consolidating information in this field may help researchers choose the best high-flux tactics. However, this method may limit review comprehensiveness.

Nguyen et al. (2022) briefly overview membrane distillation's non-fluoroalkyl functionalized hydrophobic surface modifications to promote environmentally sustainable applications [11]. The suggested method synthesizes a comprehensive overview of MD surface alterations research without fluoroalkyl functionalization. Implementation involves reviewing relevant academic material and assessing these improvements' environmental and financial benefits.

Madalosso et al. (2021) review current membrane surface modification methods for membrane distillation. The methods include electrospinning, coating, and plasma treatment. The proposed method examines several membrane surface modification methods and their uses in membrane distillation. Implementation involves integrating research findings and identifying progress and challenges in these methodologies [12]. The output values examine several membrane surface modification strategies and their suitability for membrane distillation.

Tijing et al. (2019) examine nanofibers in water and wastewater treatment, focusing on membrane distillation [13]. The suggested method synthesizes a comprehensive overview of nanofiber water treatment developments. Implementation involves critical analysis of nanofiber-based membrane distillation research. The output values cover nanofibers' role in water and wastewater treatment, including membrane distillation. The inclusion of nanofiber usage data is a benefit of this study. The lack of distillation application data is a drawback.

Et al. (2018) suggest amphiphobic surface modification on ENMs to improve membrane distillation wetting resistance. The technology alters ENM surface properties to make them amphiphobic [14]. The implementation phase evaluates modified membranes' anti-wetting properties in membrane distillation. The amphiphobic membranes improve distillation wetness resistance. This technology's wetting resistance is useful in many applications. However, maintaining performance over time may be difficult.

Tlili and Alkanhal (2019) studied ENMs' viability in membrane distillation processes for water and wastewater treatment. A comprehensive study of electrospun nanofibers in various water treatment methods is suggested [15]. Implementation requires a concise synthesis of membrane distillation research. The results comprehensively analyze electrospun nanofibers in water and wastewater treatment, highlighting their importance in membrane distillation. Variety is a benefit of nanofibrous membranes. However, the narrow distillation applications may be a drawback.

Wu et al. (2020) proposed vapor deposition for ENM omniphobic surface modification. In membrane distillation, this modification improves membrane anti-wetting [16]. This study creates omniphobic ENM surfaces using vapor deposition. This study's implementation

phase evaluates modified membranes' anti-wetting properties in distillation procedures. Output values include altered membranes and their increased wetting resistance.

This study makes a valuable contribution to the continuous endeavors to create sustainable and effective approaches for water and wastewater treatment. By doing so, it tackles worldwide water-related issues and advocates for the responsible management of the environment. The subsequent sections of this paper will explore the fundamental concepts and methodology underlying the modification of omniphobic surfaces. Additionally, it will provide a detailed account of the experimental procedures employed, present the obtained results, and discuss the potential implications for practical applications.

Performance Enhancement of ENMs in Water and Wastewater Treatment Processes

The primary aim of this study is to examine and advance omniphobic surface modification methodologies for ENMs in the context of water and wastewater treatment technologies. This study aims to investigate several approaches to omniphobic alteration of surfaces, encompassing chemical procedures, surface patterns, and coatings.

Electrospinning

It is a technique used in the field of nanotechnology to produce nanofibers. The process of electrospinning polymer nanofibers is depicted in Fig. 1. An elevated voltage to induce melting or dissolution of a polymeric solution generates an intensified electrostatic field, hence facilitating the formation of nanofibers. At the terminal end of a capillary tube, the solution or polymer melts manifest due to the influence of its surface tension. Moreover, the presence of an electric field may be ascribed to the introduction of a significant charge inside the liquid, resulting in the emergence of forces that are seen to be amplified due to the repulsion between like charges. This repulsion, in turn, leads to a reduction in surface tension. It is important to acknowledge that applying an electric field to the semicircular surface of the solution results in the elongation of the summit of the capillary tube. This elongation forms a distinct structure known as the Taylor cone.

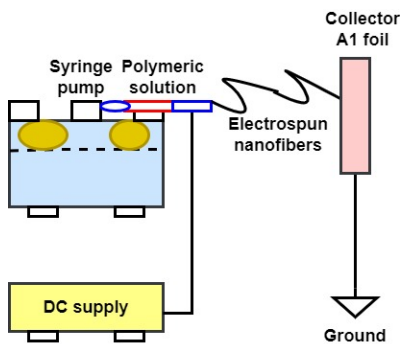


Figure 1: Process of electrospinning polymer nanofibers

The electrospinning process produces a fibrous membrane that exhibits both flexibility and a large surface area, as well as a porous structure that allows for effective filtering. These membranes are commonly known as ENMs. Multiple investigations have demonstrated that electrospun membranes have a notable combination of high flux and low transmembrane pressure. One issue related to ENMs is the electrostatic charge within the membrane. This charge results from using an electric field during the electrospinning process, and its intensity rises with the thickness of the membrane.

Furthermore, the effectiveness of ENMs is contingent upon providing supplementary support mechanisms to enhance their structural integrity. Consequently, most applications involving ENMs in membrane separation technology in contemporary times rely on a hybrid system. In the context of these systems, it is common practice to position nanofibers either on a support (also referred to as a substrate), to interpose them between distinct

layers, or to combine them with fibers of a larger scale, known as micro-sized fibers. To effectively mitigate the handling challenges associated with electrospun nanofibers, it is imperative to employ a direct spinning approach onto a robust and inflexible support, such as a collector screen. Typically, heat treatment is employed to process electrospun nanofibers to eliminate any remaining solvents and facilitate the development of crystalline structures.

In the electrospinning process, the nanofibers exhibit a random orientation, forming an open pore structure that is well-suited for membrane applications. The structural integrity of ENM can be enhanced by a heat treatment process performed below the melting point of the constituent material. During this treatment, the randomly oriented and overlapping fibers of ENM tend to fuse, resulting in improved cohesion and facilitating convenient handling. Furthermore, the application of heat treatment on ENMs facilitates the enhancement of crystallinity, subsequently leading to an improvement in their mechanical strength.

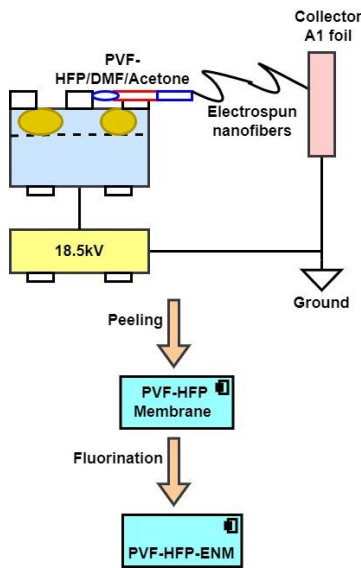


Figure 2: Fabrication of PVF-HFP-ENM

The omniphobic nanofiber membranes were initially produced by electrospinning, as seen in Fig. 2. Subsequently, a VD method was employed to fluorinate the membranes. To create a PVF-HFP dope solution, PVF-HFP tablets with a weight percentage of 16% were dissolved in a combination of DMF or acetone, with a weight ratio of 4:1. This dissolution process was carried out at room temperature for 24 hours, with constant stirring. A solution of PVF-HFP was enhanced in terms of its spinnability by adding LiCl at a concentration of 0.03 wt%.

Before electrospinning, the solution known as the "dope" underwent a degassing procedure lasting two hours. The degassed solution was then placed into a 13-mL syringe equipped with a 23G needle. The solution's flow rate has been controlled at 1 mL h⁻¹ using a syringe pump. The experimental setup involved setting the voltage at the needle tip and separating the needle tip and the grounded spinning drum clad with an aluminum foil to 18.5 ± 0.5 kV and 17 cm, respectively. The manipulation of electrospinning duration has regulated the thickness of the membrane. The humidity and temperature within the electrospinning container have been determined to be 45-55% and 22-23 °C, respectively. Before fluorination, the PVF-HFP-ENM in its original state was subjected to a vacuum oven at a temperature of 65°C for an extended period to eliminate any remaining solvent.

Surface Alteration by the Process of Fluorination

A 155 x 115 mm membrane was positioned onto a sample holder within a desiccator. Subsequently, the desiccator that had been exposed was placed into a glovebox; after that,

it underwent a purging process with nitrogen gas for approximately one hour. A volume ranging from 55 to 410 μL of FDTS was introduced into a container underneath the membrane. Subsequently, the desiccator was hermetically sealed using a cover coated with vacuum grease within a glovebox environment filled with N_2 gas. Subsequently, the desiccator was removed from the glovebox and transferred to a vacuum oven for a duration ranging from 5 to 40 hours. The vacuum level was set at 20 kilopascals, while the temperature was between 60 and 110 degrees Celsius. The membranes fluorinated from the PVF-HFP membrane were designated as the PVF-HFP-ENM, as seen in Fig. 2.

Characterization of Membranes

The investigation of the membranes involved the examination of their top surface and cross-sectional morphology, as well as the analysis of elemental mappings. This was accomplished using a Scanning Electron Microscopy (SEM) instrument, the Merlin Zeiss Gemini 2 model, in conjunction with an Energy Dispersive X-ray Spectra (EDS) system. The specimens were sputtered using Iridium for 55 seconds before being seen using a SEM.

The attenuated total reflection technique was employed to examine the membranes' surface chemical structure and functional categories before and after fluorination. The Fourier Transform Infrared (FTIR) spectrometer utilized in this study operates within the 4100-750 cm^{-1} wavelength range. The X-ray photoelectron spectrometer was utilized to ascertain the component elements' chemical structures and valence states. The scan spectra were calibrated using the binding energy of the carbon 1s electron (285.2 eV), which corresponds to graphitic carbon. The evaluation of the surface morphology and hardness of the membranes was conducted using a 3100 Atomic Force Microscope (AFM) with a scanning area measuring $3.5\mu\text{m}\times 3.5\mu\text{m}$. The arithmetic mean roughness (R) was computed over the scanned region.

The wall thickness of the membrane was ascertained by analyzing the cross-SEM picture of the membrane. The determination of the varied pore sizes of the membranes was conducted using a Porometer 3G equipment. Before the experiment, the sample was moistened using Porofil, a wetting liquid with a low surface tension of 17 mN m^{-1} . Following that, nitrogen gas (N_2) was used to examine the wet and dry curves, which can be used to infer the dimensions of the pores as well as their distribution. The porosity of the membranes was assessed using the gravimetric approach, which involved determining the pore volume of the membrane and dividing it by the total volume of the membrane. This calculation was performed using the following equation:

$$\gamma = \frac{(w_{wet} - w_{dry}) / \rho_{IP}}{(w_{wet} - w_{dry}) / \rho_{IP} + (w_{dry}) / \rho_m} \quad (1)$$

where w_{wet} and w_{dry} are the weights of wet and dry membranes, respectively. ρ_{IP} and ρ_m are the density of isopropanol and material, respectively.

To examine the wettability characteristics of the membranes, the contact angles of several substances, including deionized (DI) water, a 3.5% NaCl solution with 0.4 mM SDS, n-decane, and ethanol, were determined using a KSV tensiometer contact angle device. The slender drop method was employed for measuring these contact angles. The droplets were captured in digital form using a camera, and the contact angle measurements were afterward examined using the curve fitting technique. The average contact angle was determined based on the data obtained from a minimum of three distinct locations for each membrane. Liquid Entry Pressure (LEP) was evaluated using a cylindrical pressure dead-end Filtration Cell (FC). The FC had a practical area of 10.2 cm^2 and was linked to a pressurized air bottle through a pressure regulator and a gauge. A filter cell held a dry PVF-HFP or PVF-HFP-ENM material at the bottom. Subsequently, 100 mL of DI water was pressurized with air and introduced as the feed. The initial pressure was 20 kPa, then incremented by 10 kPa every 2 minutes. The LEP was defined as the pressure at which the initial water droplet became visible on the permeate side of the pressure FC. The calculation of LEP may be determined using the following equation:

$$LEP = [4G.S.\cos\theta]/D_{max} \quad (2)$$

Where G and S are the geometric pore coefficient and surface tension of the solution to be tested, respectively. θ is the contact angle and D_{max} is the maximum pore size of the membrane.

Stability Tests for Omniphobicity

The durability of the PVF-HFP-ENMs omniphobicity was assessed through various rigorous conditions. These conditions included subjecting the membrane to a 65-minute treatment in an ultrasonic bath, with a frequency of 50 kHz and power of 100W, while being immersed in water. Additionally, the membrane was exposed to a 55-minute treatment in boiling water (DI water at 100 °C) and treatments with 1M HCl solution and 0.5M NaOH solution at room temperature for the same duration. Following the completion of the treatment, the membranes underwent measurement of the Water Contact Angle (WCA) and Ethanol Contact Angle (ECA).

Results and Discussion

The omniphobic membrane that was produced demonstrated strong resistance to wetting when exposed to a saline feed containing Sodium Dodecyl Sulfate (SDS) at a concentration of 0.5 mM during an 8-hour dynamic Direct Contact Membrane Distillation (DCMD) test. In the context of DCMD testing, a dynamic configuration is employed to replicate and evaluate the performance and efficacy of membranes in the DCMD process, which is a thermally-induced separation technique. During the process of DCMD, a solution with elevated temperature (often comprising impurities or solutes) is brought into direct contact with one side of the membrane. Simultaneously, a cooler permeate (usually consisting of pure water or a less concentrated solution) is maintained on the other side of the membrane. Thermal energy is utilized to induce the transition of the feed solution from its liquid state to a vapor phase, facilitating the vapor's passage through the membrane's pores. This process results in the accumulation of concentrated brine or residue on the side where the feed solution is present. The permeate side of the system is responsible for gathering the vapor that has undergone purification. This vapor subsequently undergoes condensation, separating solutes or pollutants from the original feed solution.

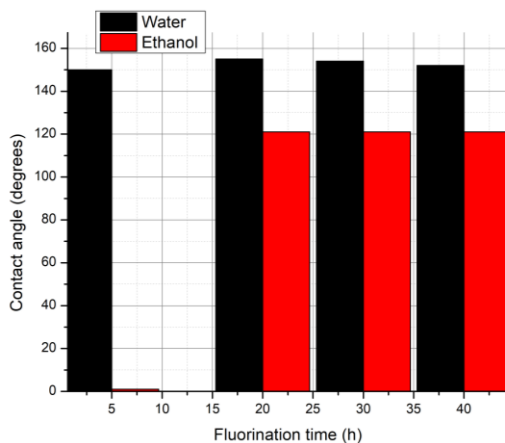


Figure 3: Impact of fluorination time (h) on WCA and ECA in degrees

The impact of fluorination time on omniphobicity is shown in Fig. 3. The PVF-HFP-ENM, with a modification time of 5 hours, may be readily dissolved by ethanol while having a high WCA. This suggests that the 5-hour duration is insufficient for the complete evaporation of the liquid PFDT. When the modification duration reached 20 hours, the PVF-HFP-ENM demonstrated the ability to achieve omniphobicity by effectively repelling water and ethanol, preventing wetness. The extension of the fluorination duration did not significantly alter the WCA and ECA, suggesting that the VD process may have already reached completion after 20 hours.

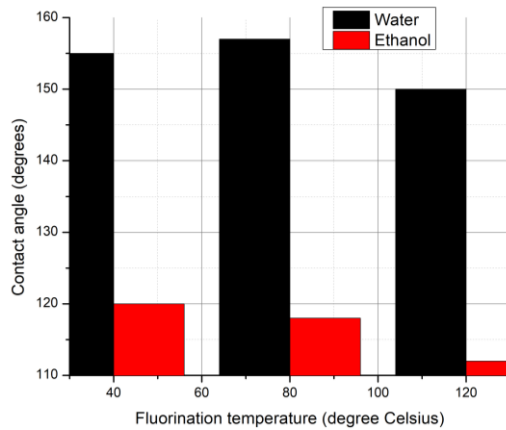


Figure 4: Impact of fluorination temperature ($^{\circ}\text{C}$) on WCA and ECA in degrees

Fig. 4 illustrates the impact of fluorination temperature on the omniphobicity of the PVF-HFP-ENM. The results indicate that the temperature's influence on membrane omniphobicity is negligible due to the comparatively low evaporation temperature of PFDT at 50°C . Nevertheless, the membrane's omniphobicity was less stable at 50°C than at higher temperatures.

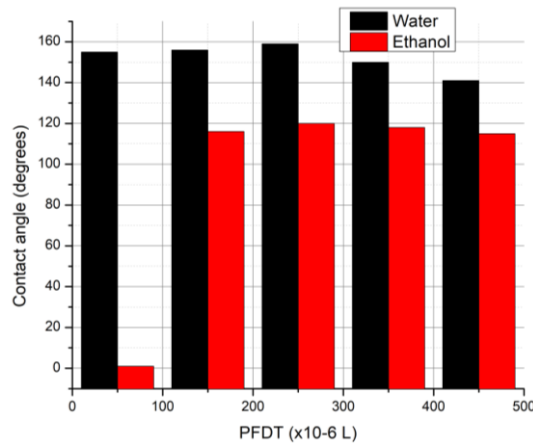


Figure 5: PFDT quantity (μL) on WCA and ECA in degrees

The impact of the PFDT quantity was of utmost importance, as evidenced by the findings presented in Fig. 5. When the PFDT was insufficient to coat the surface with a volume of $50\ \mu\text{L}$ thoroughly, the resulting PVF-HFP-ENM exhibited a lack of resistance to ethanol. Omniphobicity was effectively attained by increasing the quantity of FDTS to $250\ \mu\text{L}$. The alteration of the membrane surface's surface energy may be ascribed to the total encapsulation of PVF-HFP nanofibers by PFDT. The contact angle between the WCA and ethanol remained nearly constant even when the PFDT was increased to $450\ \mu\text{L}$. This indicates that a thicker PFDT layer on the nanofiber did not provide any extra impact on the omniphobicity of the membrane. Based on the findings mentioned above, to achieve a state of steady and satisfactory omniphobicity, the duration of fluorination, the temperature at which it is conducted, and the quantity of PFDT employed were established as 20 hours, 80°C , and $250\ \mu\text{L}$, respectively.

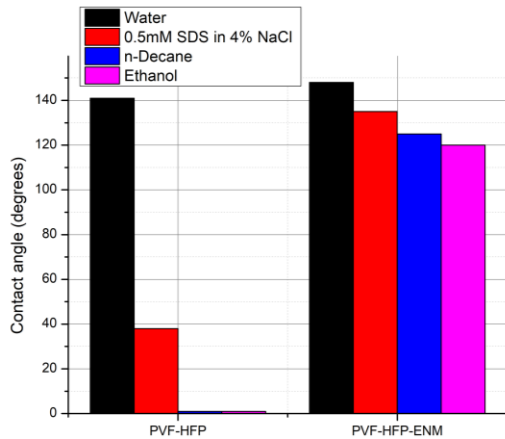


Figure 6: Contact angles of water, n-decane, ethanol, and 0.5mM SDS in 4% NaCl for PVF-HFP and PVF-HFP-ENM membranes

Fig. 6 provides contact angle measurements for several liquids on two distinct membrane types: PVF-HFP and PVF-HFP-ENM. Contact Angles of Water, n-Decane, Ethanol, and 0.5mM SDS in 4% NaCl for PVF-HFP and PVF-HFP-ENM Membranes have been given. Contact angles are a quantitative indicator of a material's wettability, whereby more extensive contact angles correspond to greater hydrophobicity and less affinity towards the liquid. Within the framework of this particular context:

The PVF-HFP membrane demonstrates a contact angle of $141 \pm 0.2^\circ$ when in contact with water, suggesting its hydrophobic characteristics. The PVF-HFP-ENM membrane has a higher degree of omniphobicity, as evidenced by WCA and ECA of $148 \pm 0.4^\circ$ and $120 \pm 0.8^\circ$, respectively. Under the experiment's conditions, adding 0.5mM SDS in 4% NaCl resulted in increased wettability of both membranes. Specifically, the PVF-HFP membrane exhibited a contact angle of $38 \pm 0.1^\circ$, while the PVF-HFP-ENM membrane had a lower contact angle of $135 \pm 0.8^\circ$. This observation implies that the inclusion of SDS in a saline solution enhances the wetting properties of the membranes. The contact angles of non-polar liquids, such as n-decane and ethanol, are very low (1°) when using PVF-HFP and PVF-HFP-ENM membranes. This observation suggests that these membranes strongly attract non-polar liquids, rendering them well-suited for many applications that involve the separation or interaction of such substances.

In general, the contact angle measurements presented in this study offer significant contributions to understanding membrane surface properties, including their hydrophobic characteristics and interactions with diverse liquids. These features are pivotal in many membrane-based separation processes and applications.

Conclusion

This study introduces an innovative method for fabricating a nanofiber membrane composed of Poly Vinylidene Fluoride - Hexa Fluoro Propylene (PVF-HFP) that demonstrates improved resistance to wetting by materials with lower surface tension. The PVF-HFP-ENM membrane, characterized by its intrinsic re-entrant structure, was subjected to fluorination utilizing 1H, 1H, 2H, 2H-Per Fluoro Decyl Trichlorosilane (PFDT) by VD without the requirement of surface activation. The fluorinated membrane exhibited remarkable surface omniphobicity, as indicated by its very high contact angles with water and ethanol, measuring $148 \pm 0.4^\circ$ and $120 \pm 0.8^\circ$, respectively. The fluorinated membrane demonstrated remarkable durability in terms of omniphobicity and mechanical properties, even when exposed to severe conditions such as ultrasonic disinfection, boiling water, and acidic and alkaline environments. This study primarily examines the application of ENM in industrial wastewater treatment. It specifically investigates the modification of nanomembranes to address issues related to clogging and improve the overall efficiency of wastewater treatment procedures.

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