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Electrospun Membrane with Ultrafine Fibers for Oil/Water Separation Application

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ABSTRACT

Environmental pollution has become an urgent concern for both nature and human beings because of oily wastewater spills from industries and household appliances. Therefore, the filtration of industrial oily wastewater is now a major problem in the present world. Many types of experiments are being conducted to find a solution for this issue, and researchers are still looking for a cheaper and better solution. A promising response to this issue can be membrane-interfaced oil-water filtration. And the application of Electrospun membranes can successfully solve this matter. It is found that Polyvinylidene Fluoride-based membranes are being used for this process because of their resistance to chemicals and good mechanical strength. Also, Titanium Dioxide particles are a suitable choice because of their non-hazardous properties and solubility with polymer solutions. In this study, Titanium Dioxide nanoparticles were first modified by regulating their pH, and then Electrospun Nanofibrous membranes were produced by adding those modified particles with Polyvinylidene Fluoride. A unique preparation method was used to decrease the particle diameter with alkaline agents, which also results in decreased fiber diameter of membranes. The produced membranes showed improved oleophilic properties and hydrophobicity. Finally, membranes were applied and can be associated with the progress of Oil/Water separation purposes, which also can sustain the recycling process of hazardous chemicals and ensure the contribution to a safe environment.

Keywords : Modification, Nanoparticles, Diameter, Electrospinning, Membrane, Oil/Water Filtration

I. INTRODUCTION

Both environment and humans are facing a great threat because of oily wastewater spilled from industrial production or household sewage [1,2]. It has become an urgent task of human beings for oil removal or oil/water filtration from marine oil spills and industrial wastewater [3,4]. Therefore it is strongly needed for us to produce materials with effective oil/water separating capabilities [5,6]. To deal with hazardous oily wastewater, various traditional methods i.e. gravity separation, adsorption, slag removal, and biological treatments were being used [7,8]. But these widely used methods have many limitations like incomplete separation, low efficiency, expensive, and complicated processes [9].

Recently, the science of applied materials and interfaces is being studied, and many experiments are being conducted to produce materials that have opposite oil-water wettability instead of traditional oil-water separation methods [10]. Various oil adsorption or oil/water separation materials have been prepared by scientists, a good candidate among which are Nanofibrous membranes because of their larger surface area, permeability, flexibility, etc. [11,12] Also, polymer membranes are widely experimented with and applied because of their low cost of and high efficiency [13,14]. One simple and cost-effective method for this is Electrospinning as it can prepare nanometer-scale fibers.

Electrospun fabricated nanofiber yarns are flexible and have better application properties in tissue engineering, smart textiles, wearable sensors, energy storage devices, etc. It is also a cheap and scalable manufacturing technique that is widely used for producing continuous nanofibers of various sources like natural and synthetic polymers, polymer-derived carbon, ceramics, metals, metal oxides, etc. [15,16]. One of the most widely used polymers in Electrospinning is Polyvinylidene fluoride (PVDF) because of its excellent mechanical and anti-oxidation properties, thermal stability, resistance to chemicals, and irradiation [17,18]. Many experiments have been conducted on the preparation of PVDF [19,20]. Ahmed et al. [21] added PVDF with HFP (hexafluoropropylene) to increase mechanical strength. Also, inorganic nanoparticles were mixed with the polymer solution to improve their properties. Lalia et al. [22] prepared a series of PVDF-HFP membranes, which explains the connection between polymer concentration and resultant structure and morphology. Although, these PVDF membranes can be easily penetrated by water [23] and also causes the penetration of the solution into a membrane, which is a severe disadvantage to the requirement of a filtration process.

Titanium dioxide (TiO_2) has also been widely used in experiments because of its self-cleaning and photocatalytic properties [24,25] which are major requirements for oil/water filtration [26]. The Spinnability and entanglement of PVDF chains can be lowered by adding TiO_2 in a polymer solution, which results in decreased diameter, and also lead to better water repellence and improved air-trapping [27]. It also shows unique properties such as non-hazardous, cheap, and easy processing control. These characteristics make Titanium Dioxide a popular choice in self-cleaning surfaces, such as pigments, solar cells, and applications for environmental purification [28].

The objective of this study is to develop a membrane that will improve the progress in oily wastewater filtration. For that, a unique preparation method should be taken which will include the Modification of Titanium Dioxide (TiO_2) nanoparticles, and will decrease their particle diameter. Then the modified particles shall be added with PVDF particles together for Electrospinning, and the produced membrane should also have decreased fiber diameter. Finally, the membrane is to be used for Oil/Water Separation for comparison and practical application.

II. METHODS AND MATERIAL

A. Materials

Titanium Dioxide (TiO_2), Sodium Hydroxide (NaOH 95%), Sodium Carbonate (Na_2CO_3), Sodium Bicarbonate ($NaHCO_3$), Tetrahydrofuran (THF), Polyvinylidene Fluoride (PVDF), and Dimethylacetamide (DMAC) was brought from Shanghai Macklin Biochemical Co. Ltd. N,N-Dimethylformamide (DMF), Dichloromethane (DCM) and Sodium Chloride (NaCl) was brought from Hangzhou Gaoijing Fine Chemical Industry Co. Ltd. And Hydrochloric Acid (HCl 38%) was brought from Hangzhou Shuanglin Chemical Reagent Co. Ltd. All chemicals and reagents were used as purchased without further purification.

B. Modification of TiO_2 Nanoparticles

For the preparation of Titanium Dioxide samples, three different methods were followed using three different alkaline agents. This also resulted in three different modified particles. Firstly, 3 g TiO_2 was added into 5 g NaOH (10%) solution and magnetically stirred at room temp for 24h. Then the gel is mixed with 20mL THF and again stirred at room temperature for 48h. After that, the solution is dried to vapor at 60°C for 4h and then cooled down to room temp by keeping for 1 week. Similarly, two more particles were modified following the same procedures, with exception of using Na_2CO_3 (10%) and $NaHCO_3$ (10%) instead of NaOH.

C. Preparation of PVDF/TiO_2 Membranes

As mentioned above, the three modified TiO_2 particles were mixed with PVDF to make five different Nanofibrous membranes by using the electrospinning method. From NaOH modified particle, 15% TiO_2 was added into a 1:1 mixture of THF and DMF with a total of 15mL solution, then 15% PVDF was added to the solution. The mixture was magnetic stirred at 60°C for 12h. Then 8mL of the solution was taken into a 10mL syringe, and an electrospinning setup was built with a "Kinco Electrospinning Machine". The liquid flow of the solution was set at $1mlh^{-1}$, and the produced nanofibers were collected in an aluminum foil wrapped in a cylinder. The distance between the needle and cylinder was 12cm. The electric volt between them was 12kV. And the rotation speed of the roller was 200 rotations per minute. After the experiment, the produced membrane was collected and dried at 60°C for 2h to remove any additional particles. Similarly, two more membranes were made with Na_2CO_3 and $NaHCO_3$ modified particles using the same parameters. And another two were also made, using Dimethylacetamide instead of DMF for comparison. The four membranes are named Na_2CO_3(i), $NaHCO_3$(i) and Na_2CO_3(ii), $NaHCO_3$(ii) respectively. Finally, five different membranes were produced for a more acceptable experimental result. For clarification, the NaOH modified particle couldn't be used with DMAC because from our experiment it is found that, NaOH doesn't make a homogeneous solution when added with DMAC.

D. Characterization

The diameter of both modified TiO_2 nanoparticles and membranes was characterized by FE-SEM. An Ultra 55 SEM machine manufactured by Carl Zeiss SMT Pte Ltd. was used to take microscopic pictures of the samples. A JEC-3000FC auto fine coater machine by JEOL was used to make a layer of Gold plating on the surface of membrane samples before inserting them into the SEM machine. The obtained images were processed and the diameter was carefully measured by using the ImageJ software. An average of 20 samples were taken from each picture to have an accurate measurement.

The membranes were also tested for their WCA (Water Contact angle). For that, a Kruss Contact Angle testing machine was used to take images of the droplets. Each sample membrane was attached to a glass slide and inserted into the machine. Then water droplets (5 µL) were placed on the membrane surface before taking images from the machine.

E. Gravity driven Oil-Water Separation

The modified PVDF/TiO_2 membranes were cut into a length/width of 6cm and were fixed between a glass funnel and a conical flask for the experiment of oil-water separation. The effective permeation area was 14.44 cm^2. Into the upper glass vessel, a mixture of 50 mL Dichloromethane (DCM) and 50 mL water including different alkali, salt, and acid solution was poured. The oil and water were colored with 3B red and 4R blue respectively. The only driving force to achieve the separation was gravity. The Separation

Efficiency (η) was calculated by the following equation [32]:

“η (%) = $V_1/V_0 \times 100$”

Where V_0 represents the oil volume before and V_1 represents the oil volume after the separation. The flowing rate (L/m^2h) or Flux during the oil-water separation was calculated by the following equation [32]:

“$q = V/St$”

Where V is the volume of permeated oil (L), S is the valid area (m^2) of the membrane, and t refers to the time (s) of the separation.

III. RESULTS AND DISCUSSION

A. pH measurement of modified TiO_2 Nanoparticles

The impact of different alkali agents used on TiO_2 particles was shown in Figure 1. The initial pH of the Unmodified TiO_2 was measured as 7.07 which is close to neutral. Then three different samples of TiO_2 particles were modified with three different Alkali agents: $NaHCO_3$, Na_2CO_3, and NaOH. It increased the pH of TiO_2 particles as a result of contact with alkaline chemicals. The pH of the three TiO_2 particles modified with $NaHCO_3$, Na_2CO_3, and NaOH were 9.4, 9.51, and 11.04 respectively. This showed that the alkaline nature of $NaHCO_3$ and Na_2CO_3 is the same as they have almost similar pH values.

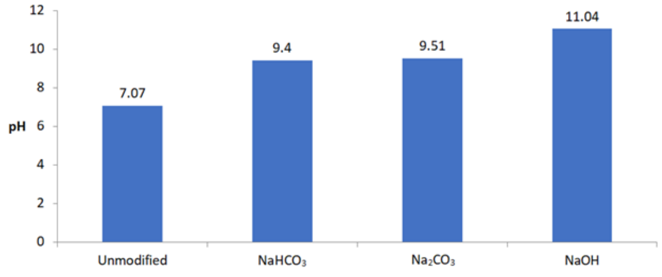
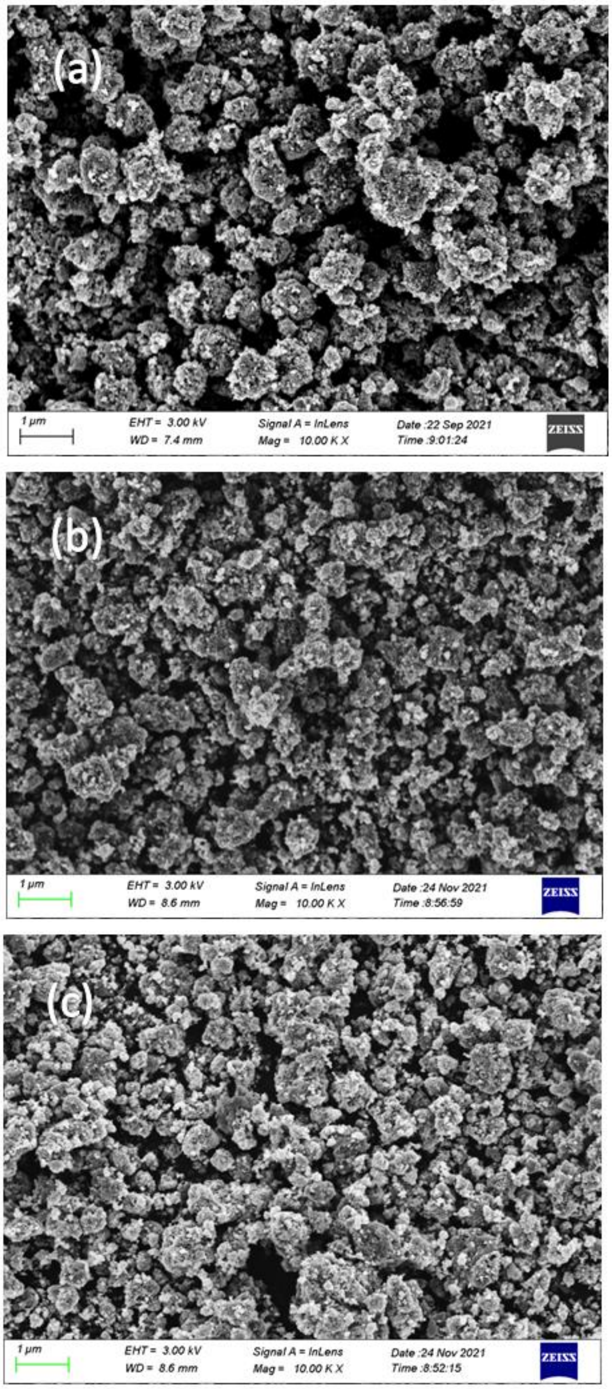


Fig 1. pH measurement of TiO_2 particles with different alkali agents.

B. Characterization of modified TiO_2 Nanoparticles

Figure 2 shows the FE-SEM images of modified TiO_2 nanoparticles. Here, three different particle samples were characterized alongside unmodified TiO_2 obtained from commercial industries. The particle diameter of pH 7.07, 9.4, 9.51, and 11.04 are 639.54, 522.28, 540.86, and 393.89 nm respectively. This indicates that at a neutral pH the particles showed relatively larger size, also showed that the influence of $NaHCO_3$ and Na_2CO_3 on TiO_2 is the same as they have almost similar particle sizes. But because NaOH has a bigger impact on increasing the pH of the particles; its diameter is also much smaller than the previous two.



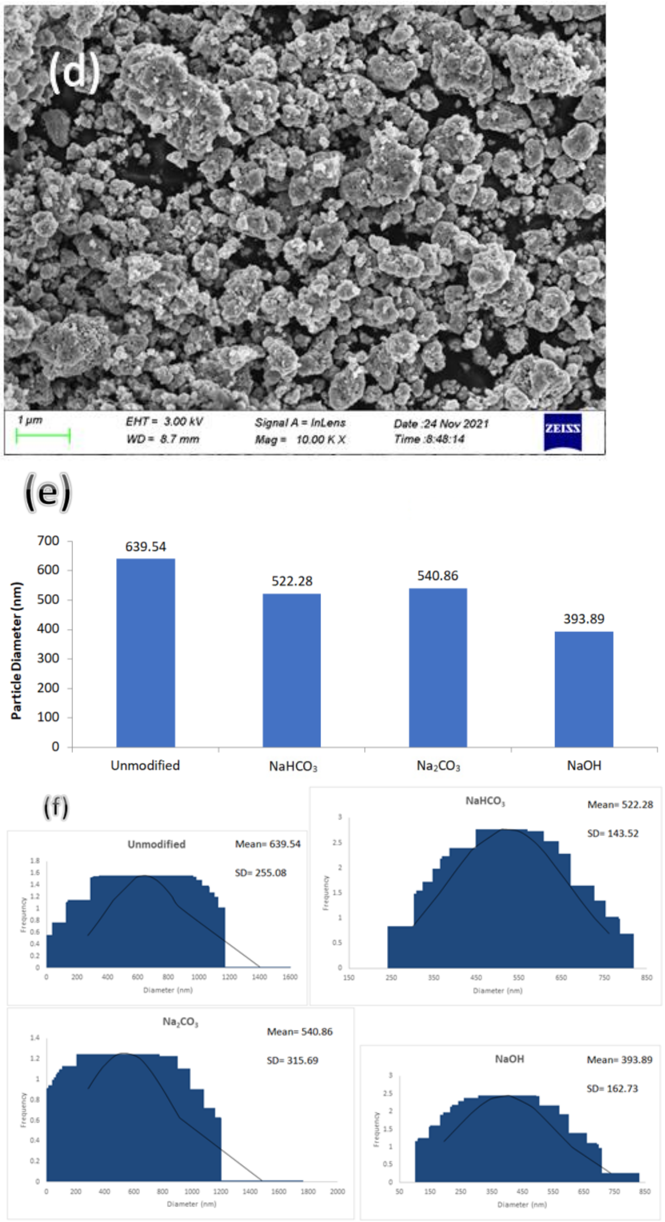
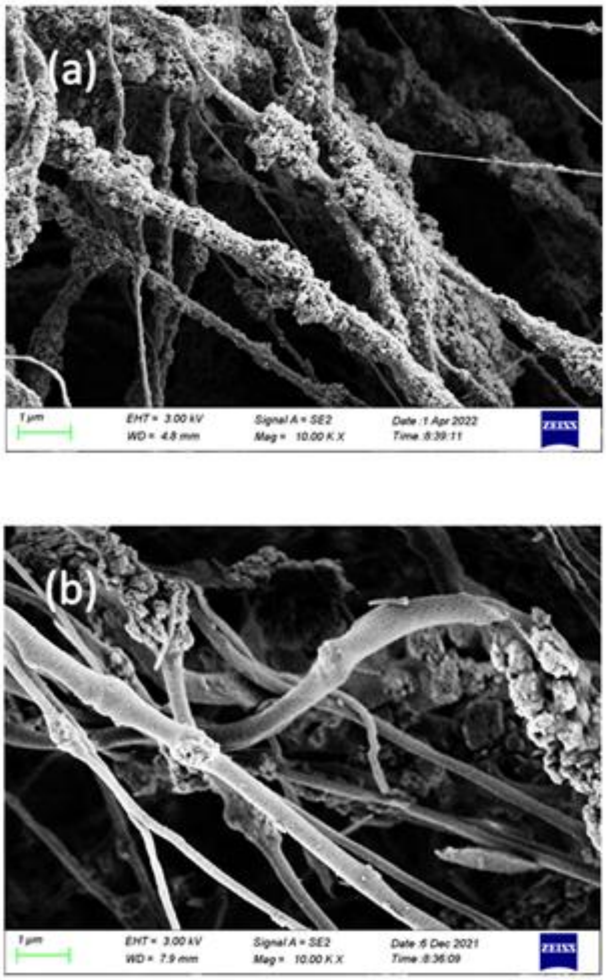


Fig 2. FE-SEM images of (a) Unmodified TiO_2, and TiO_2 particles modified with (b) $NaHCO_3$, (c) Na_2CO_3, and (d) NaOH, (e) Comparison of measured diameters against increased pH of modified particles (average of 20 samples), (f) Normal distribution of particle diameters.

C. Characterization of PVDF/TiO_2 Electrospun Membranes

The FE-SEM images of PVDF/TiO_2 ENMs (Electrospun Nanofibrous Membranes) were shown and compared in Figure 3. A phase inversion method was followed to fabricate the membranes [17]. Here, 15% TiO_2 (unmodified) and 15% PVDF were added with a 1:1 ratio of THF and DMF to fabricate the membrane for reference. Figure 3(a) shows the surface image of Unmodified TiO_2 ENM, it is found that PVDF/TiO_2 nanofibers before modification have an average diameter of 660.27 nm. Then, the characterization of five modified membranes can be also observed in Figure 3(b-f). From the measurements, it is found that the fiber diameters of $NaHCO_3$(i), $NaHCO_3$(ii), Na_2CO_3(i), Na_2CO_3(ii), and NaOH membranes are 147.44, 178.08, 110.11, 154.36 and 52.84 nm respectively. This determines that under the same magnification, all five modified membranes show decreased fiber diameter compared to similar TiO_2 membrane before modification. Figure 3(g) shows the comparison of measured fiber diameters of all sample membranes. This indicates that the particle diameter also influences the fiber diameter of electrospun membranes prepared from them.



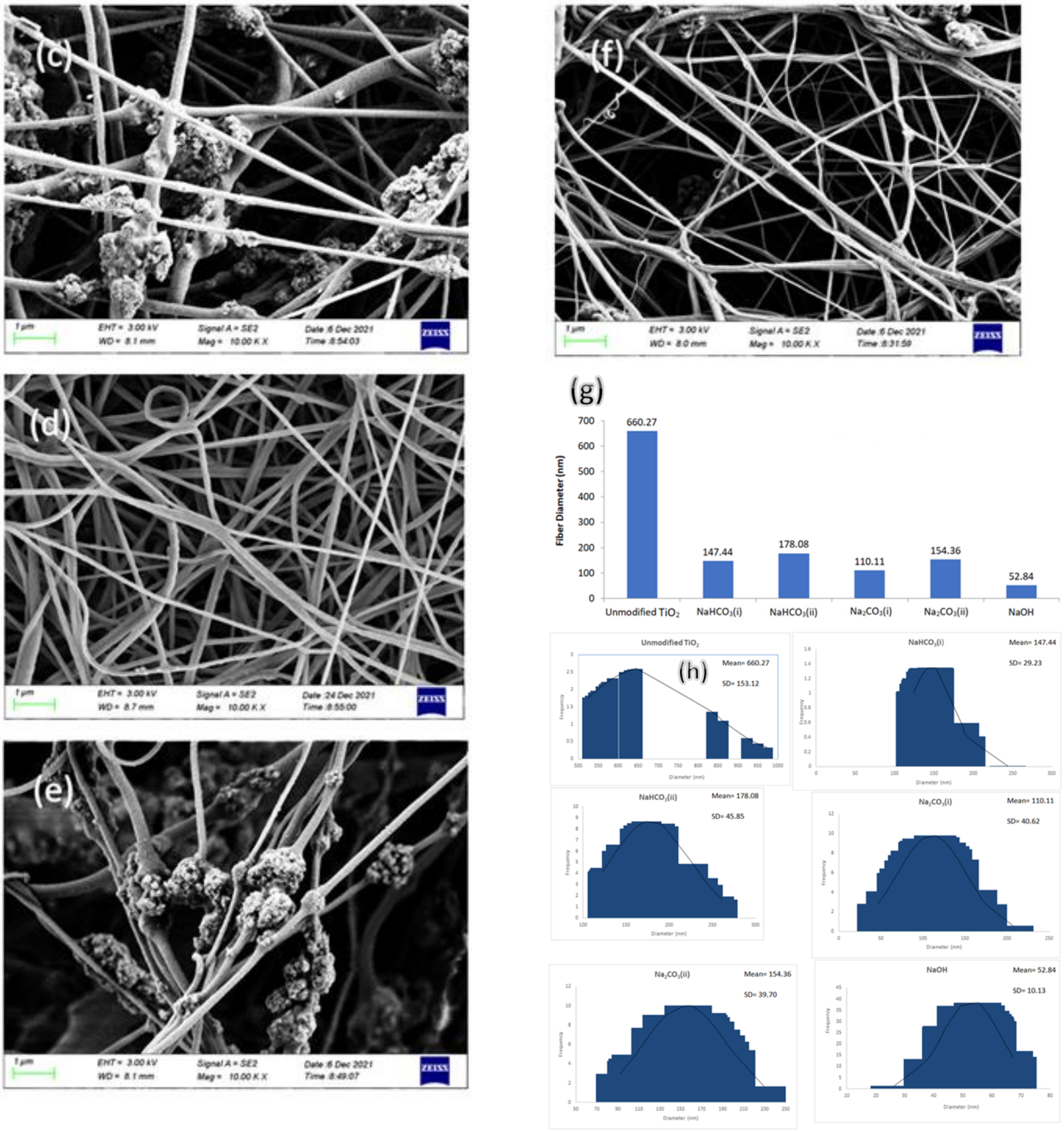
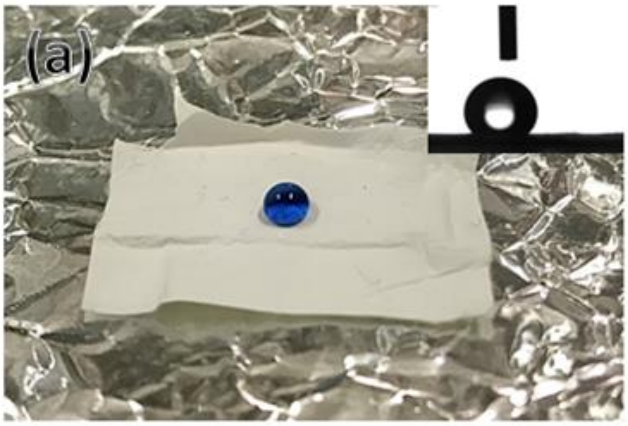


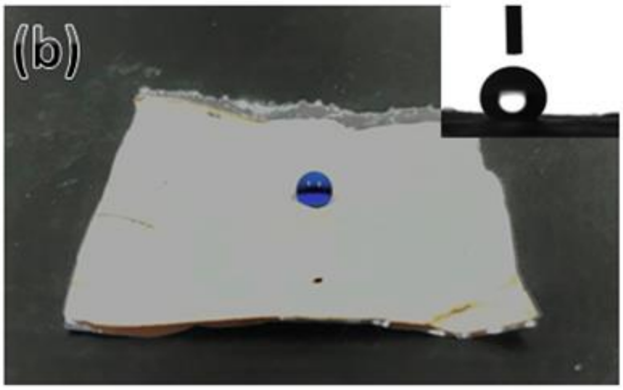
Fig 3. Fiber diameter measurement of sample membranes produced from (a) PVDF/TiO_2 (before modification), (b) $NaHCO_3$(i) with DMF, (c) $NaHCO_3$(ii) with DMAC, (d) Na_2CO_3(i) with DMF, (e) Na_2CO_3(ii) with DMAC, and (f) NaOH membranes added with Modified TiO_2 particles, (g) Comparison of measured diameters of all sample membranes (average of 20 samples), (h) Normal distribution of fiber diameters.

D. Contact Angle Measurement of Modified Membranes

The WCA (Water Contact Angle) of the five modified membranes, alongside the Unmodified TiO_2 membrane for reference, were shown and compared in Figure 4. Water droplets dyed with 4R Blue were placed on the sample membranes for physical images, also similar membranes were used to test their WCA properties. It is visible from the figures that all samples showed a higher contact angle than membrane made from the same TiO_2 particles before modification. This indicates better hydrophobicity which is a major requirement for Oil/Water filtration. Here, we also found that the NaOH-modified membrane shows the highest WCA among all membranes. This also has the smallest fiber diameter as mentioned in our previous section, and the result indicates the connection between fiber diameters with the hydrophobicity of prepared membranes. And from the other compared samples it is also proved that the smaller fiber diameters are, the higher its WCA. Therefore, the modification method used in this study has improved the hydrophobicity of all sample membranes significantly. And that fiber diameter influences the hydrophobicity of membranes prepared from them.



$NaHCO_3$(i)



$NaHCO_3$(ii)

Na_2CO_3(i)

Na_2CO_3(ii)

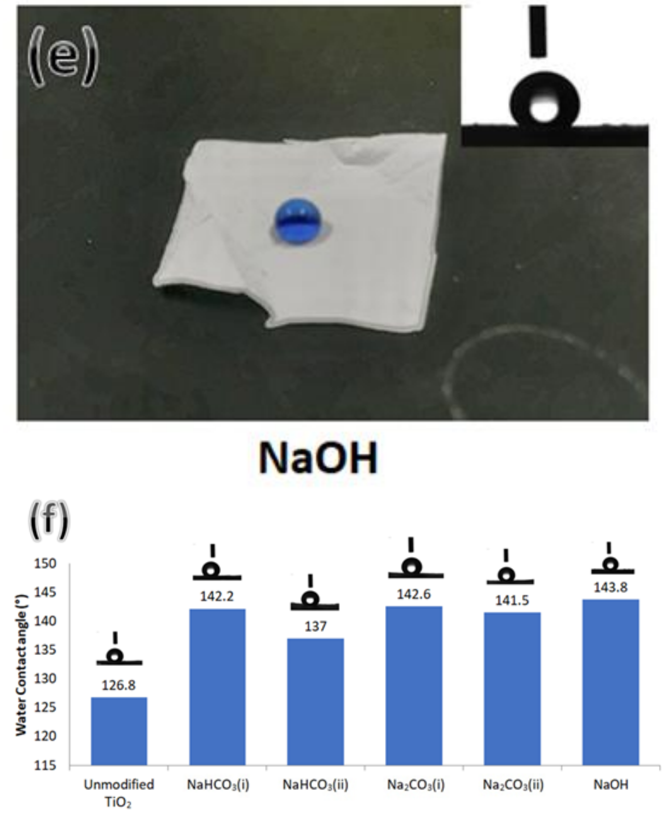
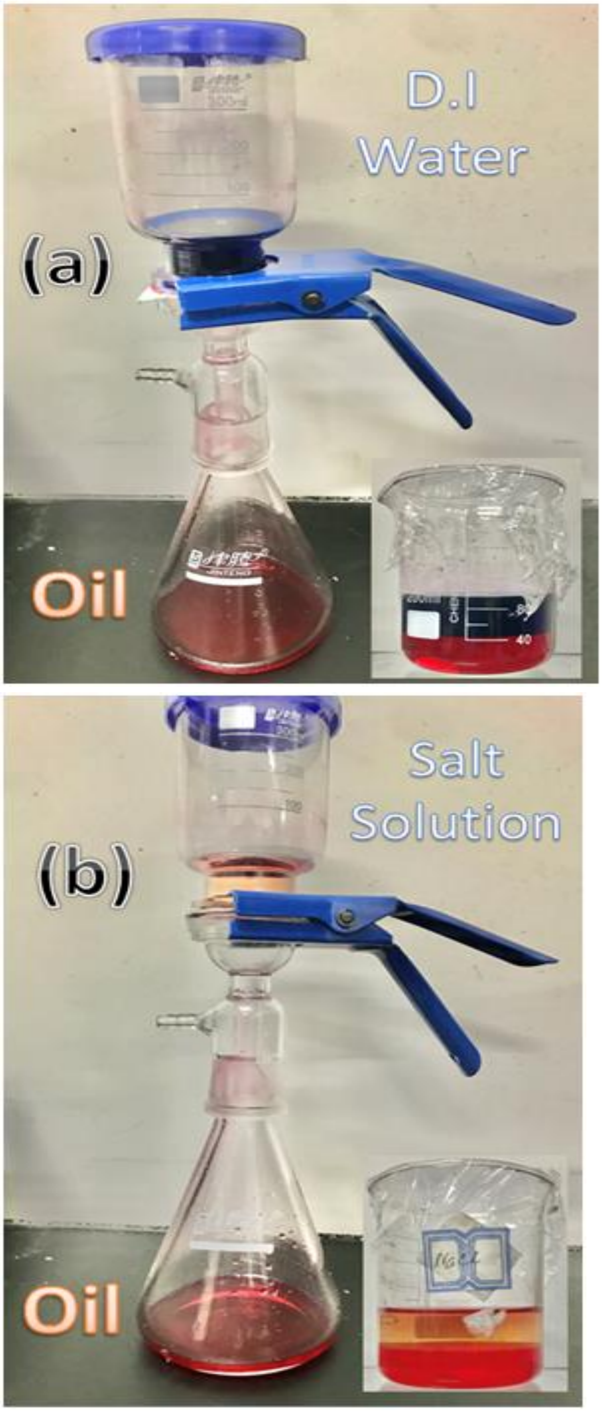


Fig 4. Display of Hydrophobic properties, (a-e) Images of D.I water droplets dyed with 4R Blue on modified membranes, insight shows the corresponding contact angle, (f) Water Contact Angle comparison of five modified membranes against Unmodified TiO_2 membrane.

E. Gravity driven Oil-Water Separation

A liquid separation setup consisting of a flask and funnel was used to test the oil/water filtration capability of Electrospun Nanofibrous PVDF/TiO_2 membranes. The modified membranes can separate oil and water from the mixture, also known as gravity driven oil-water separation. As shown in Figure 5(a), the membrane was fixed between a glass funnel and conical flask, and a 100 mL liquid mixture containing 50 mL Dichloromethane (DCM) and 50 mL water dyed with 3B Red and 4R Blue respectively, was poured onto the surface of the membrane. Once the membrane surface was contacted by the oil-water mixture, an instant separation was observed. Where the oil possessed a strong affinity and quickly permeated through the membrane, and dropped into the conical flask due to the effects of gravity force and oleophilicity of the membrane. Also, the water was repelled and stayed above because of the hydrophobicity of modified membranes. This result was achieved only by gravity and no external driving force was used during the filtration. The oil adsorption and diffusion into the Nanofibrous membrane are achieved by the oleophilicity that determines good chemical compatibility between the membrane and oily pollutants [29].



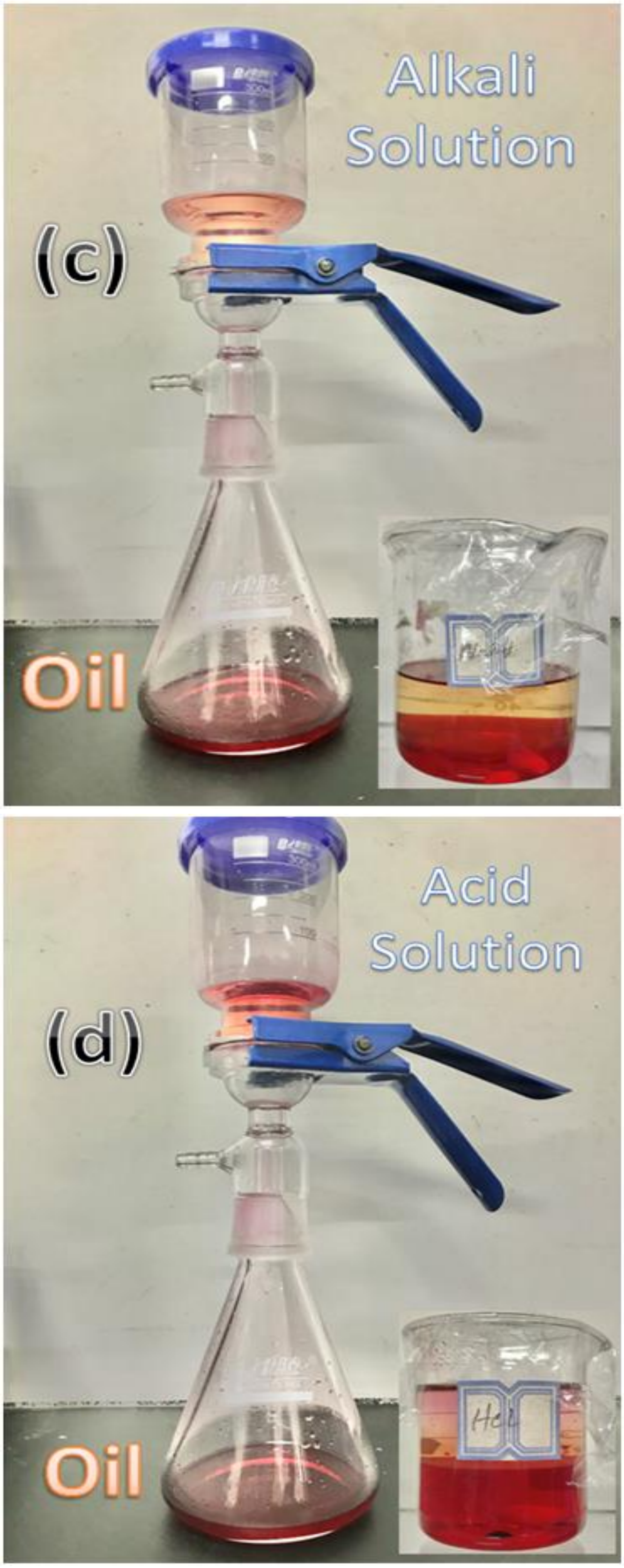
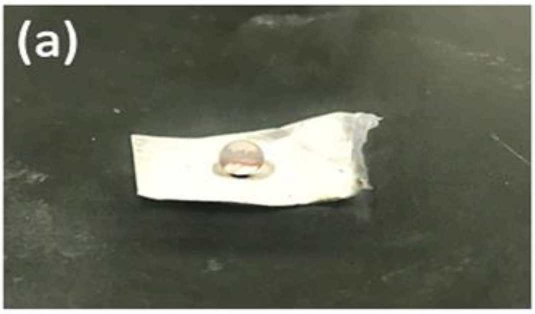
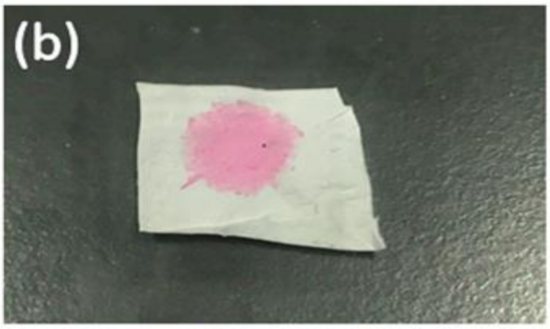


Fig 5. Filtration performance of a sample NaOH-modified membrane using mixtures of Dichloromethane and (a) Water, (b) NaCl, (c) NaOH, (d) HCl. Insight shows the corresponding mixtures before filtration.

It is found in many kinds of research that the membrane hydrophobicity/oleophilicity becomes weaker when the materials are contacted with strong corrosive chemicals, which is a severe limitation in real oil/water separation. And because of polluted wastewater contents, it is important to test the filtration performance in different harsh environments, especially in alkali, salt, and acidic conditions [30,31]. Figure 6 shows representative images of corrosive droplets and oil on the membrane surface. The water droplets of acid, salt, and alkali showed perfect spherical shapes, proving that the samples are hydrophobic not only towards pure water but also corrosive droplets. It is also observed that only the oil droplet is spreading over the membrane, which indicates that the Nanofibrous membranes achieved steady and excellent anti-corrosion to many different chemicals. And at the same time retained their affinity towards oil, as it was able to quickly wet and further diffuse into the membrane.

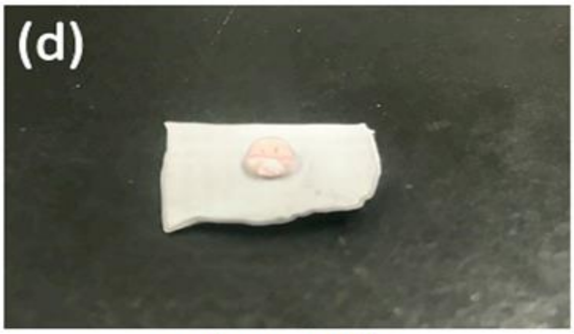


Salt Solution



Oil droplet

Alkali Solution

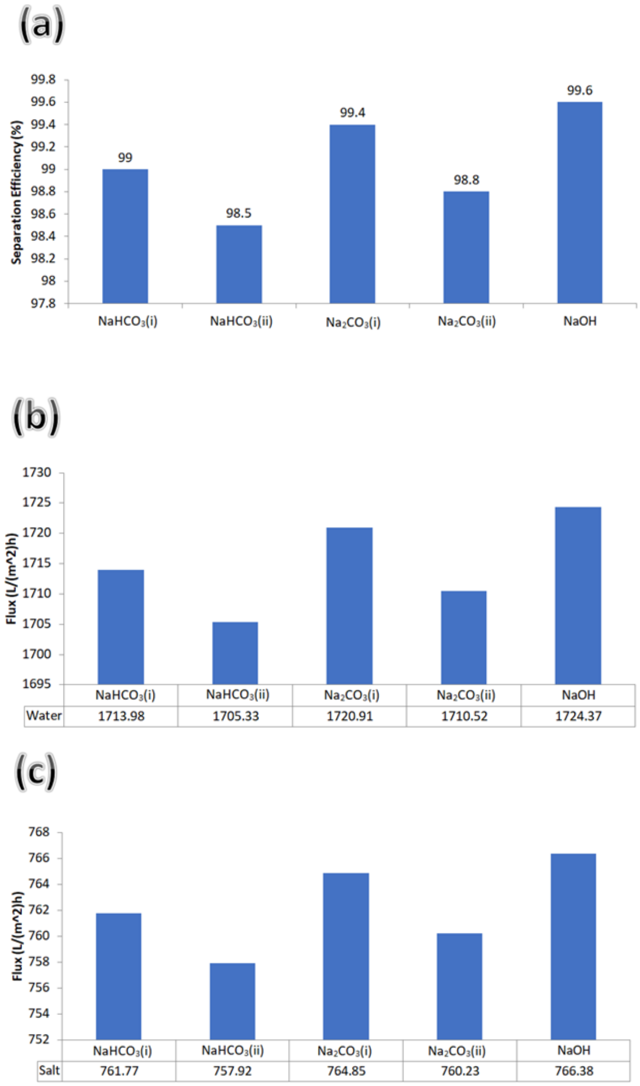


Acid Solution

Fig 6. Demonstration of Oil and Different Corrosive Solution droplets on the surface of a sample NaOH-modified membrane.

Since the membranes are corrosion resistant, their application in oil-water filtration could also be used in harsh environments. Figure 5(b-d) demonstrates the separation of the same membrane in alkaline, salt, and acidic pollutant solutions. It is found that the modified membranes with PVDF/TiO2 composition were able to separate the oil (Dichloromethane) from NaOH, NaCl, and HCl. During every different experiment, the oil quickly penetrated through the membrane towards the bottom of the conical flask regardless of the solution pH, while the water containing different chemical solutions stayed above the membrane. This showed excellent separation performance with the corrosive solution and displays a high oil/water separation performance.

The performance calculation of five modified membranes for filtration of Oil with different corrosive chemicals (Water, Salt, Alkali, and Acid) was shown and compared in Figure 7. For determining the separation performance, Figure 7(a) summarizes the separation efficiency of nanofiber membranes. From our experiments, it is found that the NaOH-modified membrane shows the best separation efficiency. This indicates the relevance of filtration performance with fiber diameter and hydrophobicity. It is also found that the efficiency of a sample membrane against any water, salt, alkaline or acidic solution shows the same. This means the nature of different corrosive chemicals does not affect the modified properties, and the same membrane can be applied against all types of pH to separate from the oil, indicating its effectiveness for real-life Oil-Water filtration. The mean value of membrane separation efficiency found is 99.06.



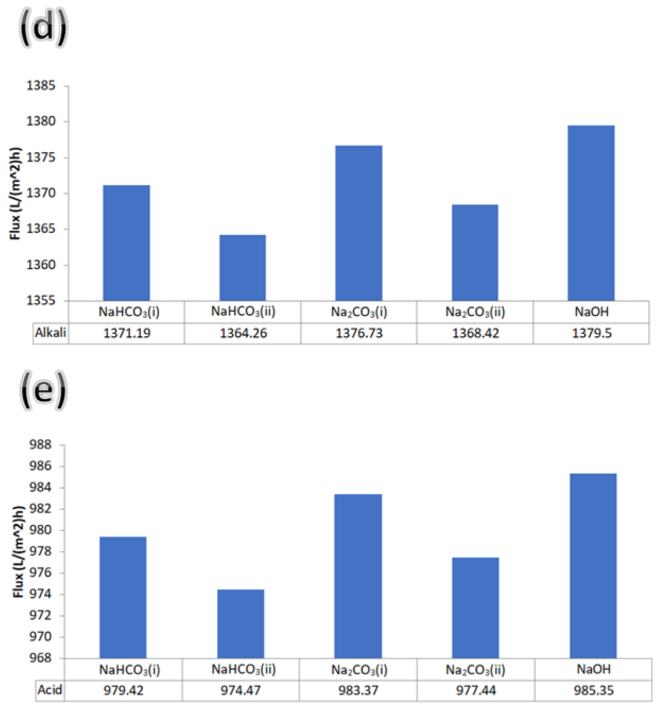


Fig 7.(a) Separation efficiency comparison for oil-water separation of five sample membranes, (b-e) Flow rate (flux) of modified membranes against different corrosive mixtures.

Figure 7(b-e) represents the Flow rate (Flux) of the modified membranes against different Water, Salt, Alkali, and Acid mixtures. It explains that although the separation efficiency of a particular membrane for different corrosive mixtures is similar, the slight time difference in separating oil from water, salt, alkali, and acid creates a larger difference between filtration flow rates. From property comparison, separation efficiency, oil flux, and comprehensive performance, it is found that the NaOH-modified membrane is most suitable in all aspects as shown in previous results. From this study, it is also determined that the hydrophobicity/oleophilicity and anti-corrosion properties made the modified membrane a good candidate for oil/water separation.

IV. CONCLUSION

A unique preparation method was followed to develop Electrospun nanofibrous membranes from modified TiO_2 particles and was applied in Oil/Water filtration. In this study, different types of alkaline (NaOH, Na_2CO_3, and $NaHCO_3$) were used to modify the pH of TiO_2 particles. And by increasing the pH value, this method also showed a decrease in the particle diameter which was confirmed by FE-SEM morphology analysis. After that, these particles were added with PVDF to produce several nanofiber membranes by Electrospinning. And the modified membranes were again characterized by FE-SEM, where all membranes showed smaller fiber diameters. Also, the Water Contact Angle was tested. Compared with Unmodified TiO_2, the membranes showed increased hydrophobicity. Then, the Oil Filtration test of each membrane was conducted with four different methods. Here all membranes showed high efficiency with Water, Salt, Alkaline, and Acid. Finally, the best performance was chosen from all properties to find the suitable material. The produced membrane can be used in industries and other household applications where oily wastewater is a bigger problem. By helping a significant factor in the pollution treatment of industrial waste, this nanomaterial will be useful to contribute to green nature.

V. ASSOCIATED CONTENT

Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

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Notes

The authors declare no competing financial interest.

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SUPPORTING INFORMATION

Figure S1: pH measurement of (a) Unmodified TiO_2, and TiO_2 particles modified with (b) $NaHCO_3$, (c) Na_2CO_3 and (d) NaOH.

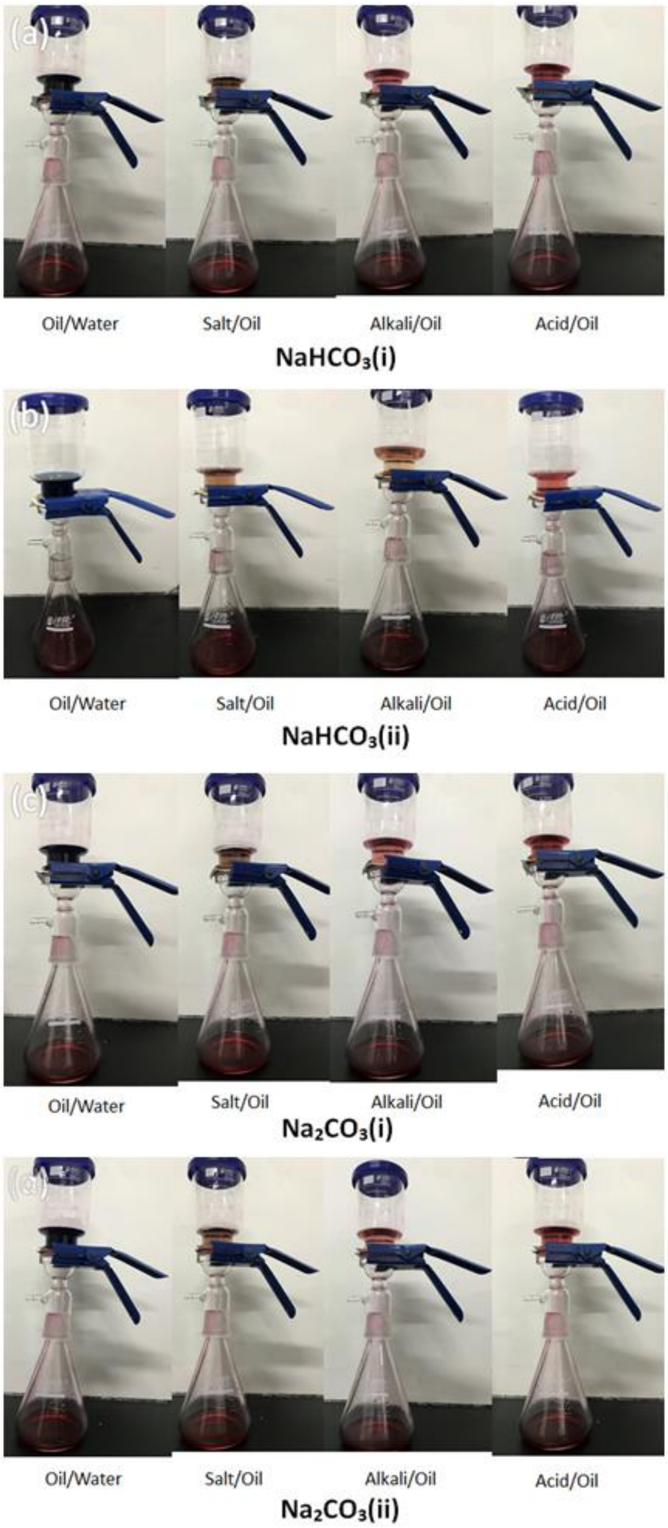

Figure S2: Filtration performance of four sample membranes using mixtures of Dichloromethane (Oil) with D.I Water/ NaCl/ NaOH and HCl.

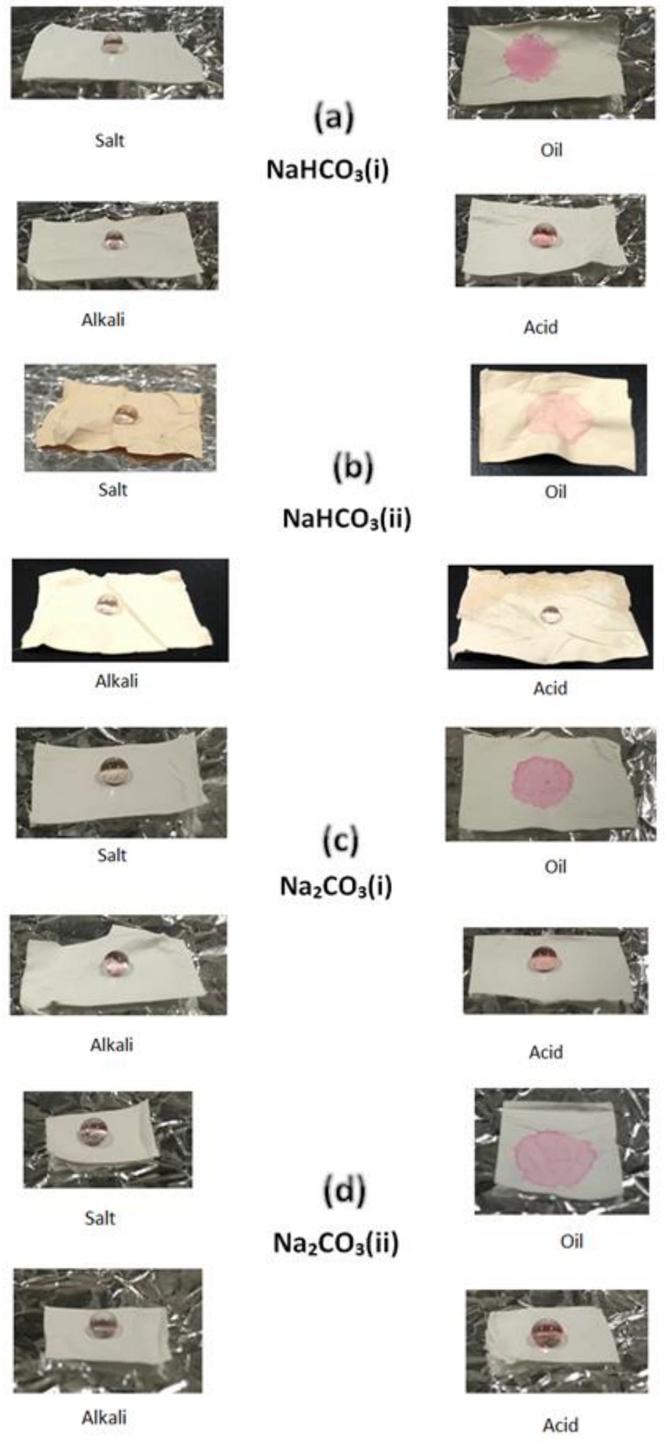

Figure S3: Demonstration of Oil and Different Corrosive Solution droplets on the surface of four sample membranes.

Author Profile

MOHAMMAD FAHIM UDDIN was born in Dhaka, Bangladesh. He is currently pursuing his Post-graduation from Zhejiang Sci-Tech University, one of the best institutes in China specializing in textile studies. With a major in Textile Science & Engineering, his field of research is Nanotechnology and Nonwoven Textiles. He completed his graduation in 2018 from the well-reputed BGMEA University of Fashion and Technology (BUFT) in Bangladesh, majoring in Apparel Manufacturing and Technology. After that, he had work experience as a Merchandising Executive with Multinational Brands like Walmart, PVH, Amazon, etc. Apart from institutional and professional achievements, he also completed training in various certificate courses from the Finance Ministry of Bangladesh and also participated in Inter-university Debate Competitions. Currently, he is living in Hangzhou, the capital of Zhejiang Province of China.

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