A Facile Approach of Thin Film Coating Consisted of Hydrophobic Titanium Dioxide over Polypropylene Membrane for Membrane Distillation

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Abstract

In this work, the hydrophobic modification of TiO₂ nanoparticles (HTiO₂) was carried out by reacting with dodecylphosphonic acid (DDPA) and hexylamine solution. A facile approach of the self-assembly technique was used for the coating of hydrophobic HTiO₂ layer over the microporous polypropylene (PP) membrane. The self-assembled layer was formed between the interface of trimthic chloride (TMC) (in hexane) and trimethylamine (in water) solutions. The high porosity for the coated membranes ascribed to the self-assembled trimesimal acid (TMA) layer and its potential to generate open and loosely packed, two-dimensional hydrogen-bond networks on the membrane surface. The dispersion of HTiO₂ was accomplished in the TMC in hexane leading hydrophobic and porous surfaces than the neat PP membrane. The initial average pore size of the PP membrane was reduced from 0.4 µm to 0.2 µm with a coating of 2.0 wt% of HTiO₂. The new membranes showed high reliability, high rejection, and water flux during the real seawater desalination tested in direct contact membrane distillation (DCMD) configuration. The maximum transmembrane permeate flux of 45.4 kg/m²h with >98% salt rejection was obtained for the coating layer with 2.0 wt% HTiO₂ at 80ºC demonstrating the future potential application towards seawater desalination.

1. Introduction

Membrane Distillation (MD) is a thermally driven membrane-based separation process. One of the promising applications of this process is seawater desalination due to low operating temperatures than conventional process since in the MD method the water isn't essentially heat up to the boiling [1]. The working principle of MD involves vapor transport through the pores of the microporous membrane driven by the vapor pressure gradient across the membrane. The membrane characteristics resemble microfiltration membranes; however, more hydrophobic surfaces and porous structures are desired to overcome wetting and low flux issues respectively. The determination of physical characteristics such as porosity, morphology, pore size, hydrophobicity and liquid entry pressure (LEP) will help to identify the suitable membrane for the practical applications [2]. LEP is the minimum pressure at which the water starts flowing through the membrane pore. For an ideal MD membrane, LEP value should be high to avoid the membrane wetting during the MD operations. The fabrication of potential MD membranes is in a developmental stage and the researchers are focusing on

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the composites of hydrophobic polymers with the nanofillers for the enhanced stability and improved performance [3-5]. Polypropylene (PP) as a membrane material for MD applications offers hydrophobic surfaces owing to its high surface tension, temperature stability, less conductivity of heat, and low surface energy [1].

Despite the essential features of PP membranes towards the MD application, further modification is essential to attain high anti-wetting and antifouling characteristics during the MD application [6]. The hydrophobic modification of PP membrane can be effectively performed by coating method [7,8]. Himma et al. prepared coated PP membrane by dip-coating method to attain the superhydrophobic surface [9]. The polymer solution concentration and drying temperature have influenced the hydrophobicity. The membrane surface roughness and contact angle were increased using methyl ethyl ketone as nonsolvent. Zhou et al. used a converted PP membrane by superhydrophobic PP surfaces by coating with 500-mesh PTFE-coated sieve [10]. Roy et al. used acid functionalized carbon nanotube on the PP substrate to enhance the water vapor transport through the membrane [11]. The functionalized CNT improved the flux, salt rejection and thermal stability of modified PP membrane. Xu et al. coated the PP membrane with the silica dispersed in PP solution followed by treatment with 1H,1H,2H,2H-perfluoroocetytriethoxysilane (POTS) [12] for MD application. Shao et al. achieved the fluorination of SiO nanoparticles on the PP membrane to improve the roughness, hydrophobicity, and durability of the resultant MD membrane [13].

Generally, TiO
2 nanoparticles are hydrophilic and a few studies have been reported on hydrophobic surface tailoring using TiO
2. Sun et al. converted polyethersulphone membrane surface to a superhydrophobic surface by the combined reactions of silane and fluoralkyl silanization [14]. The modified membrane displayed a higher contact angle of 154° compared to 75° for the neat PES membrane. Guosheng et al. converted hydrophilic TiO
2 nanoparticle to hydrophobic by reacting with the silane coupling agent [15]. The wetting studies revealed that the hydrophobicity of silane-modified TiO
2 nanoparticles was increased. Amir et al. used hydrothermal method first to deposit TiO
2 and then to fluorosilanize using 1H,1H,2H,2H-perfluoroocetytriethoxysilane (POTS) membrane [16]. The modified membranes were superhydrophobic with a contact angle 166° and LEP value was increased to 190 kPa from 120 kPa compared to neat PP membrane. The altered PVDF membranes showed superior MD flux and higher antifouling properties. Organic phosphorous acids such as phosphonic acids and their ester derivatives are widely used to functionalize the nano-metal oxide surfaces [17]. The alkyl chains terminating with the phosphonic acid group can easily react with the active -OH groups of TiO
2 through covalent bonding [18,19]. Further, the stability of TiO
2 nanoparticles will be increased by phosphonate groups when paired with an electronegative terminating group [20,21]. The phosphonic acid modification of TiO
2 attached with long-chain alkyl groups imparts hydrophobic modification on the particle surface [22]. In this study, the first time we report the molecular self-assembly approach for the coating of hydrophobic nanoparticles on the PP membrane support. The hydrophobically modified phosphonic acid–TiO
2 nanoparticles (HTiO
2) are used as a nanofiller during coating over the PP membrane. Phosphonic acid groups are ideal to produce self-assembled monolayer due to the low acid dissociation constant [23,24]. The proposed approach of self-assembly is a simple interfacial reaction to generate a charged thin layer on the PP substrate. The seawater desalination of newly fabricated PP nanocomposite membranes was tested in DCMD configuration. The MD performance results are correlated with the physical and chemical characteristics of the modified PP membranes.

2. Experimental

2.1. Materials

n-Dodecylphosphonic acid (DDPA) and hexylamine were procured from Sigma Aldrich Co. Polypropylene was procured from the Petrochemical Industries Company, Kuwait. Methanol and ethyl acetate were purchased from Merck Co. TiO
2 nanoparticles dry powder anatase phase (average size of 4-8 nm) was procured from PlasmaChem GmbH. Trimesoyl chloride (TMC) and Triethylamine were acquired from Alfa Aesar Co. and Sigma Aldrich Co respectively.

2.2. Synthesis of HTiO
2

The chemical functionalization of TiO
2 nanoparticles involved a literature reported protocol with minor modification [25]. TiO
2 nanoparticles (0.5 g) was directly added to an excess amount of DDPA (10 mL) and the slurry was stirred at 25-26 °C for 24 h. The solids were separated from the turbid suspension by centrifugation at 4000 rpm. The excess DDPA was removed by washing with ethyl acetate (25 mL) and then by adding methanol (20 mL). The solid TiO
2 was added to additional methanol (25 mL) and reacted with the hexylamine (10 mL) by stirring for 1 h. The precipitated solids were separated by centrifugation at 4000 rpm and the excess DDPA was removed by washing with ethyl acetate (3 x 25 mL). The washed solids were dried at 60 °C before further characterization.

2.3. Preparation of PP membrane

The preparation of flat sheet PP membranes includes a melt extrusion method due to the insolubility of PP in polar organic solvents. Briefly, PP was dry blended and added into a hopper of the Twin-screw extruder instrument. The extrusion conditions include: barrel; barrel temperature: 230 °C, die temperature: 200 °C and a screw speed: 200 rpm. The precursor films were prepared by compelling molten PP polymer through a shaped film die. Finally, the precursor films were dipped in water coagulation medium. Further improvement in the porosity of the PP membranes, was achieved by the uniaxial stretching at 140 °C. The annealing and stretching of the precursor films for the improved porosity was achieved using a previously reported protocol [26].

2.4. Preparation of coated PP membranes

The membrane fabrication step involved an approach of self-assembly techniques by the coating of the nanocomposite layer over the microporous PP membrane [27]. The coating layer thickness was limited to 100 nm using a composition of 0.1 wt% TEA (in water) with 0.05 wt% of TMC (in n-Hexane). In a typical procedure, the PP membrane surface was treated with 50 mL of TEA solution for 2 min at 25 °C to diffuse TEA molecules into pores of the PP substrate. The excess amine solution was removed by applying a compressed air gun. Now, the solution of TMC (50 mL) dispersed with HTiO
2 was poured on the PP membrane surface consisting of TEA. The TMC could react with TEA molecules for 2 min at 25 °C and the unreacted TMC was removed by applying the compressed air again. The resultant membrane was cured 90 °C for 5 min to convert TMC to trimesic acid. The hot membrane was cooled to ambient temperature and washed with deionized (DI) water and dried at 25-26 °C. The different membranes synthesized with the membrane codes in accordance with the HTiO
2 composition are presented in Table 1.

| Membrane Code | TMC (wt%) | TEA (wt%) | HTiO
2 (wt%) |
<table>
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2.5. Membrane characterization

The Fourier Transform Infrared Spectroscopy (FTIR) spectrum of HTiO
2 was recorded using Attenuated total reflectance- infrared (ATR-IR) instrument from Bruker Co. (ALPHA model). The scanning electron microscope (SEM) from Zeiss was used to determine the membrane surface images and to characterize HTiO
2 particles. The same instrument was used to determine the pore size of the top surface of the membranes assuming that the surface pores were circular [28,29]. The pore size reported in the current study is an average pore size of 50 randomly selected pores from each membrane sample. The contact angle goniometer from USA KINO (model-SL200KB) was used to measure the surface contact angle values of membranes using the sessile drop method. The atomic force measurement (AFM) instrument from Concept Scientific Instrument (Nano-Observer), France, was used to measure the surface roughness values of membranes in resonance mode. A dead-end filtration cell purchased from Sterlitech Corporation, USA was used to evaluate the liquid entry pressure (LEPave) values of membranes. In a typical protocol, the pressure of the feed side was increased at 0.05 MPa at each interval of 10 min and the pressure
corresponding to the first drop of water in the permeate was considered as a LEPs point. The porosity measurement includes a gravimetric method where the membrane sample was soaked in anhydrous isopropanol for 24 h and the initial weight of the wet membrane was recorded as $m_1$ [30]. Then, the wetted sample was dried at 50 °C for 24 h before recording the dry weight of the membrane as $m_2$. The overall porosity was calculated using the following equation (1).

$$\varepsilon = \frac{(m_1 - m_2)/\rho_w}{(m_1 - m_2)/\rho_w + m_2/\rho_p}$$  

(1)

where $m_1$ and $m_2$ are the weights of the wet and dry membranes respectively, $\rho_w$ is the density of isopropanol (786 kg/m$^3$) and $\rho_p$ is the density of the polymer (946 kg/m$^3$).

2.6. Flux and DCMD experimental protocol

For MD performance of the PP and coated PP membranes were tested in a custom-made multiconfiguration MD test unit from Convergence Instruments. The details of the experimental set-up and the experimentation protocol were presented in our previously reported article [31]. Briefly, the flat-sheet membrane 50 mm (diameter) was tightly clamped between the two chambers of the membrane module. The hot feed of real seawater collected from beach well of a desalination plant at Doha, Kuwait. The temperature-dependent flux study was conducted over a feed temperature range of 50-80 °C by maintaining a constant permeate (DI water) temperature at 20 °C. The flux $J$ (kg/m$^2$h) was measured using the following equation 2.

$$J = \frac{\Delta W}{A \Delta t}$$  

(2)

where $\Delta W$ is the weight of distillate (kg), $A$ is the effective membrane area ($m^2$) and $\Delta t$ is the sampling time (h). The details of the analysis of the water parameters using various instrumentation are presented in our previous article [31].

3. Results and Discussion

3.1. FTIR analysis

The FT-IR spectrum of TiO$_2$ nanoparticles showed different characteristic peaks as shown in Figure 1(a). In this spectrum, the broad absorption band from 3200 to 3400 cm$^{-1}$ is attributed to stretching and 1635 cm$^{-1}$ to bending vibration of O-H, corresponding to the moisture [32]. Compared to the TiO$_2$ spectrum, the HTiO$_2$ particles modified by chemically reacting with DDPA and hexylamine showed distinct characteristic peaks as shown in Figure 1(b). The chemical structure of DDPA is presented in Figure 2. In the FTIR spectrum of HTiO$_2$, the peaks corresponding to -CH$_2$ vibrational mode of DDPA molecules appeared at 2917 cm$^{-1}$, 2848 cm$^{-1}$ and 1529 cm$^{-1}$. The peaks at 1458 cm$^{-1}$ and 1003 cm$^{-1}$ corresponds to -P=O and -P-O stretching respectively [33] confirmed the DDPA modification of the TiO$_2$ nanoparticles.

![Fig. 1. FTIR spectra of a) TiO$_2$ nanoparticles and b) HTiO$_2$.](image)

3.2. SEM analysis

The SEM image of HTiO$_2$ particles is presented in Figure 3. It revealed that the size of the DDPA modified particles increased between 60-180 nm attributed to the large size of DDPA chemically attached by the TiO$_2$ particles. The SEM analysis of the neat PP and coated PP membranes in Figure 4 revealed a drop in the pore size for the later membrane surface. The self-assembled layer penetrated in some large pores of PP support membrane by the capillary forces, providing more stability for the self-assembled layer [34]. Also, it is observed that the self-assembled TMC layer has aided the HTiO$_2$ molecules to attach firmly to the membrane surface. The homogeneous dispersion of HTiO$_2$ on the membrane without any agglomeration is attributed to TMC as a suitable solvent selected for nanoparticle dispersion. The aggregation of nanoparticles is a commonly observed phenomenon during the fabrication of nanocomposite membranes as the nanoparticles tend to reduce their high surface energy at higher concentration. The surface SEM images of M-1.0 and M-2.0 membranes quite similar, however, at an elevated loading concentration of 3.0 wt% of HTiO$_2$ the membrane surface witnessed the agglomeration of nanoparticles on the membrane surface as highlighted in Figure 4 (d). The HTiO$_2$ in the coating layer produced porous structures on the membrane surfaces as the self-assembly of TMA on a membrane substrate has a high tendency to produce open and loosely packed, two-dimensional hydrogen-bond networks at the interface [35]. The formation of porous structures is well supported in the literature as Ye et al. observed the self-
assembly of TMA on gold particles with the formation of porous flower type self-assembly structures [36]. The same study revealed the formation of porous structures of trimesic acid stabilized by hydrogen bonding.

3.3. Contact angle, pore size, porosity and liquid entry pressure,

The coated PP membranes showed higher contact angle values than the neat PP membrane as presented in Table 2. This indicated that the coated PP membranes were more hydrophobic than the nascent PP membrane. The surface modification of TiO_2 nanoparticles with DDPA has produced hydrophobic groups terminating with the phosphonic acid. The improved hydrophobicity of the coated membranes attributed to the surface modification with long-chain aliphatic groups ensuring a better dispersion in the self-assembled layer via the interfacial interactions between NPs and the matrix [37]. The introduction of long aliphatic chains on the membrane surface played a positive role in the hydrophobicity enhancement by decreasing the membrane surface free energy [31]. The average pore size of the PP membrane decreased after coating from 0.40 μm to 0.18μm for M-3.0 membrane (Table 2). The decreased pore size values for coated PP membranes complies with the ideal pore size of MD operations, which ranges between 0.1-1.0 μm [38]. The decrease in pore size is due to the partial filling of the surface pores of the PP membrane by the porous structures of the self-assembled TMC layer comprised of HTiO_2. The pore size reduction was proportional to the higher loading of HTiO_2 as revealed in Table 2. This could be the effect of increased concentration of HTiO_2 leading to the formation of compact self-assembled structures with reduced pore size. From Table 2, the overall porosity of coated nanocomposite PP membranes quite improved compared to the pristine PP membrane. The increase in porosity of the membrane attributed to the formation of self-assembled TMA structures on the membrane surface [36]. The nascent PP membranes showed an LEP value of 98 kPa, while for the M-2.0 membrane its value was increased to 215 kPa (Table 2). The higher value of LEPw for coated PP membranes as revealed from Table 2 indicated the increased transmembrane pressure across the membrane due to the reduced pore size after the surface coating [1,39,40].

Fig. 2. Chemical structure of dodecylphosphonic acid.

Fig. 3. SEM image of the HTiO_2 nanoparticles.

Fig. 4. Surface SEM images of a) M-0, b) M-1.0, c) M-2.0 and d) M-3.0 membranes.
3.4. AFM analysis

The 3D AFM images of the pristine PP and coated composite PP membranes at a scan size of 10 μm × 10 μm are presented in Figure 5. The AFM images revealed a slight increase in the surface roughness value for the coated PP membranes. The HTiO$_2$ modified by DDPA containing long-chain hydrophobic organic groups tend to aggregate in aqueous medium via Van der Waals interactions with the membrane surface groups [41]. In contrary, a slight increase in the average surface roughness for modified PP membrane attributed to the least aggregation of HTiO$_2$ up to the loading of 2.0 wt% due to the presence of TMC molecules in the solvent selected for the dispersion. The hydrogen-bonding interactions between the phosphonic acid groups of HTiO$_2$ and acid groups of trimesic acid molecules could be the reason for least aggregation of HTiO$_2$ for attaining control over the increased surface roughness values for the coated membranes. Generally, modifying the nanoparticle surface changes both the nanoparticle-polymer interactions and nanoparticle-nanoparticle interactions. Also, the cross-linking interactions on the membrane surface through bonding or ionic interactions reduces the surface roughness [42, 43]. Thus, the interaction of HTiO$_2$ terminating with the phosphonic acid with the self-assembled trimesic acid groups via intermolecular hydrogen bonding could be the other reason for the small increase in roughness values for the coated PP membranes.

Table 2

<table>
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<tr>
<th>Membrane Code</th>
<th>Contact angle (°)</th>
<th>LEPw (kPa)</th>
<th>Porosity (%)</th>
<th>Pore size (μm)</th>
<th>Average surface roughness (nm)</th>
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<td>55.6±0.8</td>
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<td>56.1±0.63</td>
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<td>M-1.0</td>
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<td>M-2.0</td>
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<td>57.6±0.64</td>
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3.5. The membrane permeation flux and seawater desalination performance

As presented in Figure 6, the permeate flux of all the coated PP membranes was much higher than the nascent PP membrane. Generally, pore size reduction has an adverse effect on the flux, but the increased porosity of the coated PP membranes had dominated effect in determining the membrane flux compared to decreased pore size. The water flux of all the membranes, increased with the increase in temperature due to the increased vapor pressure of seawater as a main driving force of the MD process [1]. In the MD process, the seawater contacted directly to the surface of neat PP membrane causing wetting of the pores. The trapped seawater can reduce the effective liquid evaporation area and causes flux reduction. However, incorporation of HTiO$_2$ into the PP membrane matrix tends to improve both the pore-wetting resistance and the evaporation area eventually increasing the membrane flux [44-46]. The increased loading of HTiO$_2$ increased the water flux as the more hydrophobic surfaces formed for the easy transport of water vapors without wetting. The rejection towards the seawater TDS for both the nascent PP and modified PP membranes was >98%. However, modified PP membranes
attained slightly improved seawater ionic rejection as presented in Table 2. From Table 2, the porosity of M-1 and M-3 were almost similar with significantly higher pore size for the M-1 membrane. However, the permeate flux of M-3 was higher than M-1, particularly at high feed temperature. The increased hydrophobicity due to the higher loading HTiO$_2$ has dominated effect on increasing the permeate flux than the reduced pore size of the membrane. The presence of hydrophobic HTiO$_2$, in the coating layer tends to reduce the membrane pore-wetting. Also, the loading of HTiO$_2$ increased the membrane total vapor evaporation area to increase the mass transfer coefficient, thereby increasing the water flux as reported in other works [47-49]. The coated PP membrane with 2.0 wt% of HTiO$_2$ corresponds to an optimum loading composition with a maximum permeate flux of 45.4 kg/m$^2$h at 80 °C. M-3.0 membrane with higher loading of 3.0 wt % of HTiO$_2$ experienced a drop-in flux value to 43.8 kg/m$^2$h at 80 °C due to the excess loading of HTiO$_2$ resulted in the agglomeration of nanoparticles on the membrane surface as evidenced from SEM study. Additionally, a drop-in porosity value as presented in Table 1 for the M-3.0 membrane compared to the M-2.0 membrane leads to flux depletion. The durability test was conducted over a period of 200 h for the neat PP and M-2.0 membranes at 80 °C. For M-0 membrane, the permeate flux and TDS of the permeate was depleted to 15.6 kg/m$^2$h and 530 ppm, respectively indicating the wetting of the membrane pores by the penetration of water molecules. Whereas, M-2.0 membrane showed an almost stable flux of 43.8 kg/m$^2$h with a permeate TDS of 161 ppm at the end of the experiment representing a superior anti-wetting property.

4. Conclusions

This study demonstrated a facile molecular self-assembly approach for the coating of hydrophobic HTiO$_2$ nanoparticles layer over the microporous PP membrane. The newly synthesized neat PP membrane was porous and a further improvement in porosity with a reduction in pore size was achieved for the coated PP membranes. The high porosity for the coated membranes ascribed to the potentiality of self-assembled TMA layer to generate porous structures on the membrane surface. The PP membranes modified coating were hydrophobic due to the hydrophobic functional groups present on the HTiO$_2$ ensuring a better dispersion in the self-assembled layer. While the decrease in pore size is due to the partial filling of the pores of the PP membrane by the self-assembled TMC network comprised of HTiO$_2$. The LEP gradually increased by the addition of HTiO$_2$ due to the more accumulation of hydrophobic HTiO$_2$ on the membrane surface. The loading of HTiO$_2$ witnessed a slight increase in the surface roughness for the modified PP membrane credited to the least agglomeration of HTiO$_2$. The water flux of modified membranes was increased up to the loading of 2.0wt% HTiO$_2$ with a minor reduction in salt rejection. The less flux for neat PP membranes could be due to the easy trapping of water molecules into the membrane pores, whereas the loading of hydrophobic HTiO$_2$ particles improved pore-wetting resistance with improved flux values. This study confirmed the optimum loading composition of HTiO$_2$ as 2.0 wt%, corresponding to a high flux of 45.4 kg/m$^2$h with the highest durability for the membrane.

<table>
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Fig. 6. The water flux of membranes at different temperatures.
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References