SURFACE MODIFICATION OF THIN FILM NANOCOMPOSITE MEMBRANES USING PLASMA ENHANCED CHEMICAL VAPOR DEPOSITION METHOD FOR DESALINATION PROCESS

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ABSTRACT

Thin film composite reverse osmosis (TFC RO) membrane has been commercially used to desalinate salty water since the 1970s to produce clean water to address water scarcity issue in many countries. However, the commercial TFC RO membranes are still associated with several drawbacks including permeability/selectivity trade-off and scaling problem. Thin film nanocomposite (TFN) membrane incorporating titania nanotube (TNT) as previously reported, was found to offer outstanding features as the incorporation of TNT in polyamide (PA) layer could improve membrane pure water flux (PWF) and salt separation performance. However, TNT tends to have high aggregation ability and low dispersion in the organic solvent, which reduces its practicability for TFN membrane development. Therefore, this study aimed to develop a new type of TFN membrane incorporated with TNT functionalized using an environmentally friendly plasma-enhanced chemical vapour deposition (PECVD) method. The surface of TNT was respectively functionalized with 2-hydroxyethyl methacrylate (HEMA) and methyl methacrylate (MMA) at different plasma deposition times (5 and 10 min). Results showed that the incorporation of 0.05 w/v% MMA-modified TNT (5 min) into the membrane outperformed the HEMA-modified TNT (5 min) with respect to PWF and NaCl rejection, achieving 52.5 L/m2.h (measured at 15 bar) and 97.6% (tested at 2000 ppm NaCl feed solution at 15 bar), respectively. This is due to the even distribution of MMA-modified TNT throughout the PA layer which increased the membrane water affinity. The TFN membrane incorporated with MMA-modified TNT was further coated using hydrophilic acrylic acid (AA) via PECVD method as a strategy to heal the surface defects of the selective layer caused by TNT incorporation. The NaCl passage was observed to reduce from 2.43% to 1.50% (tested at 2000 ppm NaCl feed solution at 15 bar) without significantly altering PWF. This membrane also exhibited extraordinary anti-scaling performance with a higher flux recovery rate (FRR) (>85%) compared to the unmodified TFC membrane (74.8%), which is mainly attributed to its enhanced surface negative charge and improved hydrophilicity. Likewise, the developed AA-modified TFN membrane could effectively mitigate the silica scaling by achieving higher FRR (88.1%) than the unmodified membranes. In conclusion, this work demonstrated the potential of using PECVD method to rapidly modify not only the surface properties of nanomaterials but also the top PA selective layer of TFN membranes, overcoming the drawbacks associated with the TFC membrane and improving the TFN membrane for enhanced salt rejection and anti-scaling performance without trading-off its water permeability.

ABSTRAK

Sejak tahun 1970-an, membran osmosis balikan komposit filem nipis (TFC RO) telah digunakan untuk penyahgaraman air masin supaya menghasilkan air yang bersih dan juga untuk mengatasi masalah kekurangan air yang dialami seluruh dunia. Walau bagaimanapun, membran TFC RO masih mengalami beberapa kelemahan, iaitu masalah tahap tukar ganti antara fluks air dan penolakan garam dan juga masalah penskalaan. Membran nanokomposit filem nipis (TFN) dengan penambahan titania nanotiub (TNT) yang dilaporkan sebelum ini didapati menawarkan ciri-ciri yang baik disebabkan penambahan TNT dalam lapisan poliamida (PA) boleh meningkatkan fluks air tulen (PWF) membran dan kecekapan pemisahan garam. Walau bagaimanapun, TNT mempunyai kecenderungan penggumpalan yang tinggi dan penyerakan rendah dalam pelarut organik boleh menghadkan kegunaannya untuk pembangunan membran TFN. Oleh itu, tujuan kajian ini adalah untuk membangunkan membran TFN baharu dengan penggabungan TNT berfungsi menggunakan kaedah pemendapan wap kimia plasma (PECVD) yang mesra alam. Permukaan TNT masing-masing difungsikan dengan 2-hidroksiletil metakrilat (HEMA) dan metil metakrilat (MMA) monomer pada masa pengendapan plasma yang berbeza (5 dan 10 minit). Keputusan menunjukkan bahawa penggabungan 0.05 w/v% MMA-terubahsuai TNT (5 min) ke dalam membran memberi kesan lebih baik HEMA-terubahsuai TNT (5 min) terhadap PWF dan penolakan NaCl masing-masing mencapai 52.5 $L/m^2.h$ (ukuran pada 15 bar) dan 97.6% (diuji pada larutan NaCl 2000 ppm). Ini adalah disebabkan oleh pengagihan MMA-terubahsuai TNT yang seragam pada seluruh lapisan PA telah meningkatkan kehidrofilikan membran. TFN membran yang gabung dengan MMA-terubahsuai TNT telah dilapisi dengan asid akrilik (AA) yang bersifat hidrofilik melalui kaedah PECVD sebagai strategi untuk menyembuhkan kecacatan pada permukaan lapisan PA yang disebabkan oleh penambahan TNT. Pengeluaran garam NaCl dilaporkan berkurang dari 2.43% kepada 1.50% (diuji pada 2000 ppm NaCl dan 15 bar) tanpa menpengaruhi kebolehtelapan air tulen. Membran ini juga menunjukkan prestasi anti penskalaan yang baik dengan kadar perolehan fluks (FRR) yang lebih tinggi (> 85%) berbanding dengan membran TFC (74.8%). Ini disebabkan peningkatan cas negatif dan sifat hidrofilik. Membran TFN terubahsuai AA dapat mengatasi penskalaan silika dengan mencapai FRR yang lebih tinggi (88.1%) berbanding dengan membran yang tidak terubahsuai. Kesimpulannya, kerja penyelidikan ini menunjukkan potensi kaedah PECVD bukan sahaja mengubah sifat permukaan bahan nano, tetapi juga lapisan PA membran TFN, mengatasi kekurangan membran TFC dan memperbaiki membran TFN untuk meningkatkan penolakan garam dan sifat anti penskalaan tanpa mengorbankan kebolehtelapan air.

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LIST OF ABBREVIATIONS

AA	-	Acrylic acid
AAO	-	Anodic aluminum oxide
AAPTS	-	N-(2-Aminoethyl)-3-aminopropyltrimethoxysilane
AFM	-	Atomic force microscopy
Ag	-	Silver
APTES	-	3-aminopropyltriethoxysilane
ATR-FTIR	-	Attenuated total reflectance-Fourier transform infrared spectroscopy
BIBB	-	Bromoisobutyryl bromide
BSA	-	Bovine serum albumin
$C_2H_5OCS_2K$	-	Potassium ethyl xanthogenate
CA	-	Cellulose acetate
CNT	-	Carbon nanotube
CNT	-	Carbon nanotube
COOH	-	Carboxylic acid
CSA	-	Camphorsulfonic acid
CTA	-	Cellulose triacetate
CuO	-	Copper oxide
EDC·HCl	-	N-(3-dimethylaminopropyl)-N-ethylcarbodiimide hydrochloride
EDX	-	Energy dispersive X-ray
Fe_3O_4	-	Iron oxide
FESEM	-	Field emission scanning electron microscopy
FO	-	Forward osmosis
FRR	-	Flux recovery rate
GA	-	Glutaraldehyde
GHS	-	Globally Harmonized System
GO	-	Graphene oxide
$H_2Ti_3O_7$	-	Trititanate
HCl	-	Hydrochloric acid
HEMA	-	2-hydroxylethyl methacrylate

HFBA	-	Heptafluorobutyric acid
HMO	-	Hydrous manganese oxide
HR-TEM	-	High resolution transmission electron microscopy
IP	-	Interfacial polymerization
KBr	-	Potassium bromide
KCl	-	Potassium chloride
LbL	-	Layer-by-layer
MA	-	Maleic anhydride
MED	-	Multi-effect distillation
MMA	-	Methyl methacrylate
MOF	-	Metal organic framework
MPD	-	M-phenylenediamine
MPDSAH	-	Zwitterionic 3-(methacryloylamino) propyl-dimethyl-(3-sulfopropyl) ammonium hydroxide)
MSF	-	Multi-stage flash
MWCNT	-	Multi-walled carbon nanotube
$Na_2SiO_3.5H_2O$	-	Sodium metasilicate pentahydrate
Na_2SO_4	-	Sodium sulphate
NaCl	-	Sodium chloride
NaOH	-	Sodium hydroxide
NF	-	Nanofiltration
NH_3	-	Ammonia
NPs	-	Nanoparticles
nTNS	-	Negatively charged titania nanosheet
O_2	-	Oxygen
PA	-	Polyamide
PECVD	-	Plasma enhanced chemical vapour deposition
PEI	-	Polyester non-woven fabric
PIP	-	Piperazine
pMEDSAH	-	Poly[(2-methacryloyloxy)ethyl] dimethyl [3-sulfopropyl] ammonium hydroxide
PRO	-	Pressure retarded osmosis
PSf	-	Polysulfone
pTNS	-	positively charged titania nanosheet

PVDF	-	Polyvinylidene fluoride
PWF	-	Pure water flux
RB5	-	Reactive black
RO	-	Reverse osmosis
sccm	-	Standard cubic centimeter per minute
SI	-	Saturated index of silica
SDS	-	Sodium dodecyl sulfate
SEM	-	Scanning electron microscopy
SiO_2	-	Silicon dioxide
SWRO	-	Sea water reverse osmosis
TDS	-	Total dissolved solids
TEA	-	Triethylamine
TEM	-	Transition electron microscopy
TFC	-	Thin film composite
TFN	-	Thin film nanocomposite
TiO_2	-	Titanium dioxide
TMC	-	Trimesoyl chloride
TNT	-	Titania nanotube
UF	-	Ultrafiltration
UV-Vis	-	Ultraviolet-visible spectrophotometry
VIM	-	1-vinylimidazole
WCA	-	Water contact angle
WHO	-	World Health Organization
XLE	-	Extra Low Energy
XPS	-	X-ray photoelectron spectroscope
XRD	-	X-ray diffraction spectroscopy
ZnO	-	Zinc oxide

LIST OF SYMBOLS

A_m	-	Effective membrane surface area
$(-\Delta G_S)$	-	Surface free energy of membrane
K_B	-	Boltzmann constant
C_F	-	Solute concentration in feed solution
C_p	-	Solute concentration in permeate solution
Delta G_{cri}^{het}	-	Critical free energy of heterogeneous nucleation
Delta G_{cri}^{hom}	-	Critical free energy of homogeneous nucleation
Delta t	-	Permeate collection time
Delta V	-	Permeate volume
f(θ)	-	Wetting function
J	-	Final water flux
J_o	-	Initial water flux
R	-	Membrane solute ejection
V	-	Molecular volume of silica
y^-	-	Electron donor components of γ^{AB}
y^+	-	Electron acceptor components of γ^{AB}
y_{ij}^{AB}	-	Lewis acid-base components
y_{ij}^{LW}	-	Lifshitz-van der Waals components
y_L^-	-	Electron donor of probe liquids
y_L^+	-	Electron acceptor of probe liquids
y_L^T	-	Surface tension of water
$y_{mem\text{-}SiO_2}$	-	Interfacial free energy of membrane-silica boundary
$y_{mem\text{-}wat}$	-	Interfacial free energy of membrane-water boundary
y_S^-	-	Electron donor of material
y_S^+	-	Electron acceptor of material
$y_{SiO_2\text{-}wat}$	-	Interfacial free energies of silica-water boundary
y^{TOT}	-	Total surface tension of probe liquids
θ	-	Averaged water contact angle value

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CHAPTER 1

INTRODUCTION

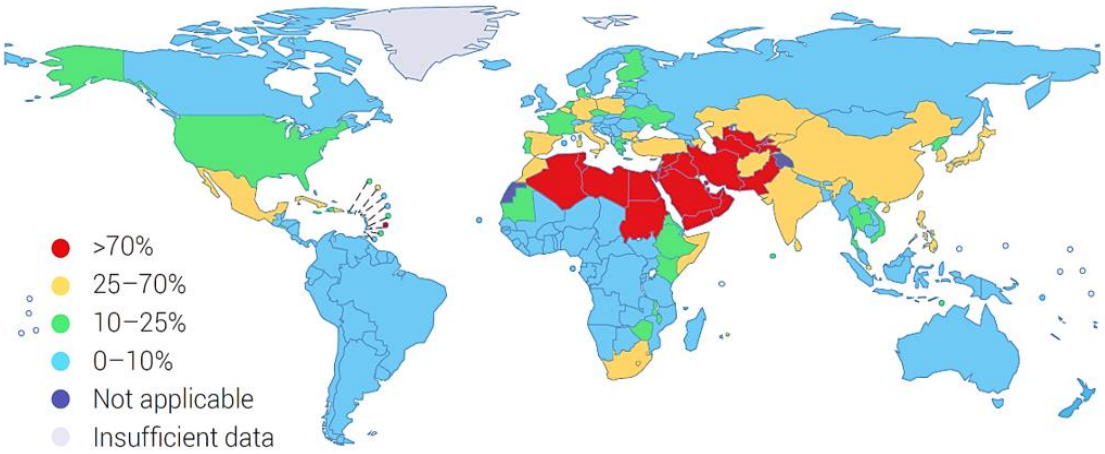
1.1 Background of Research

Population growth, climate change, urbanisation and demographic change in the 21st century had caused numerous challenges for supplying sufficient clean water to people. It is estimated about 4 billion people in the world are now affected by water scarcity, which is higher than the previous estimation of three billion people (Plessis, 2018). Water scarcity has become a severe global issue even though the Earth comprises 75% of water. Nevertheless, it must be noted that close to 97.5% of the water available in the world is covered by seawater and other saline aquifers and thus can't be directly accessed/used by human (Meissner and Mampane, 2005). Furthermore, not all of the fresh water is applicable for humans as some of them are contaminated due to water pollution.

Water demand also increases following the increase in the human population. As economic development and industrialisation grow in the last five decades, colossal population growth worldwide has risen tremendously and this has led to the transformation of massive biodiversity and ecosystem loss. The continuous increase of human population also results in higher demand for shelter, food and other resources. According to the United Nations World Water Development Report 2019, about 22 countries are experiencing severe water stress level (more than 70%) as depicted in Figure 1.1. This situation is significant especially for the United Arab Emirates, Sub-Saharan Africa, Saudi Arabia and Palestine (Azoulay, 2019). In order to solve the water scarcity issue, strategies such as seawater desalination is applied.

Desalination can be applied to waters with different salinity levels, such as estuarine water, brackish groundwater and seawater. There are two major desalination technologies that are available for the treatment, which are membrane-based and

thermal-based desalination. However, due to the high energy consumption of thermal-based technology, membrane desalination is the cost-effective alternative way that is widely used for the water desalination process (World Health Organization, 2011).



Note: Physical water stress is defined here as the ratio of total freshwater withdrawn annually by all major sectors, including environmental water requirements, to the total amount of renewable freshwater resources, expressed as percentage.

Figure 1.1 Global overview of countries experiencing different levels of water stress (Azoulay, 2019)

Among the membrane technologies, reverse osmosis (RO) is the most widely used membrane for desalination in a global scale. This is due to the continuous improvement with concerted research in terms of membrane material, module design, pre-treatment process and energy recovery device. The improvement in mechanical, chemical and biological strength of RO membranes has also reduced its cost per unit volume for treated water by ten folds since 1978 (Lee, Arnot and Mattia, 2011). Such membranes can minimize fouling and concentration polarization to maximize permeate flux.

In 1959, cellulose acetate (CA) membranes were introduced as the first asymmetric RO membrane by Loeb and Sourirajan. However, CA membranes exhibited several limitations, including low pH tolerance and temperature range, susceptible to microbiological attack, low water permeability and inability to withstand high pressure.

To overcome this, thin film composite (TFC) membranes were introduced by adding a polyamide (PA) layer on top of a microporous, polymeric based support membrane. TFC membranes show enhanced stability in acidic and alkaline environments, coupled with excellent salt rejection and adequate flux flow compared to the CA membrane (Lee et al., 2011).

Despite the fact that TFC membranes offer superior salt rejection and water flux, high resistance to pressure compact, wider operating temperature and pH range as well as a higher resistivity towards biological attack, it is still susceptible to trade-off relationship between water permeation and ion rejection (Yin and Deng, 2015) as well as membrane scaling (Bush *et al.*, 2018). Scaling which occurs on membrane surfaces can lead to flux decline which inherently affects the quality of water produced. Although appropriate cleaning methods can be applied to overcome membrane scaling, such approach at the same time increases maintenance cost.

One of the strategies that researchers frequently employ to alter the physicochemical properties of membrane is through the incorporation of inorganic hydrophilic nanofillers within the membrane PA selective layer (Lai *et al.*, 2019a; Chong *et al.*, 2019). However, nanofillers' aggregation could potentially cause surface defects due to void formation within the PA layer (Yin *et al.*, 2012; Dong *et al.*, 2015; Yang *et al.*, 2020). Other than the addition of nanofillers, modifying the membrane surface properties can also be considered as an effective and potentially viable path to improve membrane desalination performances and scaling resistance (Saqib and Aljundi, 2016; Goh *et al.*, 2019b).

Numerous surface modifications such as surface coating, layer-by-layer (LbL) assembly, surface adsorption and chemical grafting have been explored by researchers (Kang and Cao, 2012; Asadollahi, Bastani and Musavi, 2017). However, most of these techniques are time-consuming and require multiple complex steps, which restricts its application. On the other hand, another emerging surface modification technique available is the plasma polymerization method. This is a chemical-free reaction technique which is able to form a thin film layer on any surfaces within a few seconds. They are advantageous compared to other surface grafting and surface coating

techniques as they are chemical free, be able to conduct in low heat, high precision and rapid. Therefore, employing plasma treatment on the surface of RO membrane is considered to be a promising technique to improve the surface physicochemical and anti-scaling properties. Plasma will generate free radicals in the plasma chamber and induce monomer polymerization on the RO membrane surface. Therefore, this study intends to develop a new type of surface-modified TFN membrane, integrated with functionalized nanomaterials via plasma-enhanced chemical vapor deposition (PECVD) to overcome the highlighted limitations.

1.2 Problem Statements

TFC membranes that consist of an ultrathin PA selective layer supported by a microporous substrate have been widely used in the desalination industry due to their stability in a wide range of temperature and pH (compared to cellulose-based membranes), high resistance towards pressure and reasonably good anti-biofouling properties. However, commercial TFC RO membranes still suffer a main drawback which is the trade-off relationship between salt rejection and permeate flux (Ong *et al.*, 2016). One strategy to overcome this limitation is by incorporating hydrophilic nanofillers within the PA layer to improve the resultant membrane hydrophilicity and salt separation efficiency (Yin and Deng, 2015; Yang *et al.*, 2020).

Many varieties of inorganic nanofillers such as graphene oxide (GO), multiwalled carbon nanotubes (MWCNTs), titanium dioxide (TiO_2), titania nanotube (TNT) and metal-organic framework (MOF) have been utilized for TFN membrane fabrication (Bano *et al.*, 2015; Emadzadeh *et al.*, 2015b; Kadhom, Hu and Deng, 2017; Al Mayyahi, 2018). Nevertheless, direct incorporation of hydrophilic nanofillers into the PA layer of TFN membranes is not preferable as it does not disperse uniformly in organic solutions during interfacial polymerization (IP) reaction. Poor nanofiller dispersion can lead to particle agglomeration, which in turn would diminish the membrane performance. In order to enhance its dispersion stability in non-polar solvents, researchers explored ways to modify the surface of titanium-based nanofillers with silane coupling agents such as 3-aminopropyltriethoxysilane (APTES)

and N-(2-Aminoethyl)-3-aminopropyltrimethoxysilane (AAPTS) to improve its dispersion stability (Zhang *et al.*, 2013; Emadzadeh *et al.*, 2015b; Amini, Rahimpour and Jahanshahi, 2016). However, silane coupling agents which consist of amino groups and 3 ethoxy groups are highly hazardous to the environment (Wang *et al.*, 2011). In addition, modification using silane coupling agents is time consuming as it typically requires 24 h to complete the surface modification (including intensive cleaning process) (Zhang *et al.*, 2013; Emadzadeh *et al.*, 2015b). In this study, the surface modification of TNT was carried out by depositing a thin methyl methacrylate (MMA) film on the outer surface of TNT via an environmentally friendly yet rapid PECVD method. This approach in general can complete the modification within seconds. It is expected that the deposition of hydrophobic MMA on the surface of TNT would improve the dispersion stability of TNT in the organic solvent during IP process.

There are numerous membrane surface modification methods found in the literature, including surface coating, surface grafting, and plasma polymerization and the incorporation of nanoparticles. Previous studies have demonstrated the weakness of surface coating (Ni *et al.*, 2014) and grafting that negatively affected membrane water flux (Wang *et al.*, 2015b; Vatanpour, Sheydaei and Esmaeili, 2017; Liu *et al.*, 2019b). Surface coating is not preferable as it can deteriorate over time while surface grafting has poor control on the density of thin film layer formed and has potential to damage the membrane pore structure (Zhao and Yu, 2015). These limitations can adversely affect both membrane permeation rate and membrane stability over time. On the other hand, modification using grafting or physical adsorption methods require additional cross-linking steps, which may be economically undesirable (Kwon *et al.*, 2012a). PECVD method has shown that it can overcome the beforementioned limitations by altering the membrane surface in low reaction time, high versatility and a homogeneous deposition layer (Wang *et al.*, 2018b).

Although better dispersion of nanomaterials in organic solutions can be achieved with surface modification/functionalization, agglomeration could not be completely eliminated as strong van der Waals attractive force between nanoparticles could lead to agglomeration, especially at high loadings used (Al Aani *et al.*, 2018; Lai *et al.*, 2019a). The aggregation of nanomaterials in the PA layer may develop defects

(hole/voids) which inhibit salt removal rate (Emadzadeh *et al.*, 2014; Lau *et al.*, 2015; Liu *et al.*, 2020). For instance, Emadzadeh *et al.* (2015a) reported that NaCl rejection of RO membrane was significantly attenuated from 94.05% to 85.87% upon the incorporation of 0.1 wt% nanofillers into the PA layer, although the flux increased. Ghanbari *et al.* (2015) discovered that the lower selectivity of TFN membranes against NaCl was attributed to nanomaterials' aggregation which led to the development of defects in the PA layer. Furthermore, hydrodynamic shear stress during filtration process is also cited as another reason which causes detachment of nanomaterials and diminishes the performance and stability of TFN membrane (Park *et al.*, 2016; Shao *et al.*, 2017). To overcome nanofillers leaching from PA layer and surface defects due to nanomaterials embedment, in this work, a hydrophilic acrylic acid (AA) monomer was deposited onto the surface of TNT-incorporated TFN membrane via PECVD to produce a defect-free nanocomposite membrane.

On the other hand, silica is an abundant and ubiquitous mineral found in natural waters with a typical concentration between 20 and 60 mg/L (Mi and Elimelech, 2013). However, once the silica concentration in the solution is beyond its solubility limit (generally in the range of 120–150 mg/L near pH 7 at 25^{o}C (Mi and Elimelech, 2013; Milne *et al.*, 2014)), insoluble silica will form and adhere on the membrane surface. This has a significant impact towards membrane performance and its flux recovery rate (FRR). Currently, silica scaling control for desalination membranes relies heavily on scaling inhibitors such as polyacrylate and polyphosphonate-based antiscalants (Tong *et al.*, 2017). However, the utilization of anti-scalants increases its operating cost and can also cause organic and biological fouling (Sweity *et al.*, 2015; Turek *et al.*, 2017). According to Tong *et al.* (2017), the scaling rate is highly dependent on the membrane surface charge. Negatively charged membrane surface can exhibit high selectivity and excellent anti-scaling properties by incorporating hydrophilic nanoparticles or deposition of hydrophilic polymeric based thin film.

In this work, MMA-modified TNT was embedded within the PA selective layer as a solution to improve compatibility of inorganic nanomaterials within polymeric matrix. Even though the MMA-modified TNT is able to reduce the agglomeration (within the PA layer) to certain extent, the membrane surface imperfection

(nanovoids/holes) can still occur and cannot be completely avoided. To address this issue, the AA monomer was deposited on the surface of TFN membrane via PECVD method to heal the surface imperfection. Moreover, the AA-modified TFN membrane with great amount of carboxyl groups is believed to enhance the membrane scaling resistance. It is expected that the hydrophilic AA deposited on the membrane surface would possess higher surface energy barrier against silica nucleation while the increased silica solubility at alkaline condition (higher pH) can reduce the formation of silica aggregation on the membrane surface. The AA-modified TFN RO membrane incorporated with optimized PECVD-modified TNT is believed to improve the anti-scaling properties and overcome the trade-off effect on salt rejection and permeate flux of membrane.

1.3 Research Objectives

Based on the problem mentioned above, this work aims to develop a new generation of TFN RO membrane using the PECVD method for effective desalination process. More specifically, the objectives of this work are:

1. To investigate the impacts of different types of PECVD-modified TNT and the plasma modification time on the dispersion stability of nanomaterials in the organic solvent and thus its influence on the TFN membrane properties in terms of water flux and salt separation.

2. To assess the efficiency of the PECVD method by depositing a hydrophilic polymer atop the PA layer in order to minimize surface imperfections of TFN membrane upon the modified TNT incorporation.

3. To evaluate the effect of feed solution pH and concentration of the silica in the feed solution on the anti-scaling properties of TFC and selected PECVD-modified TFN membranes.

1.4 Scope of Study

In order to achieve the objective of the research, the following scopes are listed:

For Objective 1:

(a) Synthesizing TNT (with diameter ranging between 20 and 48 nm) from TiO_2 nanoparticles via hydrothermal method.

(b) Modifying the outer surface of TNT with hydrophilic HEMA monomer and hydrophobic MMA monomer via PECVD method with different plasma duration time (5 min and 10 min). The plasma duration should be controlled at maximum 10 min to prevent the formation of very thick coating layer.

(c) Studying the morphology of pristine and PECVD-modified TNT using high resolution transmission electron microscopy (HRTEM).

(d) Characterizing the chemical properties of pristine and PECVD-modified TNT via Fourier transform infrared spectroscopy (FTIR).

(e) Characterizing the crystallinity of TNT via X-ray diffraction spectroscopy (XRD).

(f) Fabricating TFC membrane using m-phenylenediamine (MPD) (2% (wt/v)) and trimesoyl chloride (TMC) (0.1% (wt/v)) via IP method.

(g) Fabricating TFN membranes by incorporating pristine and PECVD-modified TNT (at a fixed loading of 0.05% (wt/v)) within the PA selective layer via IP method.

(h) Studying the impacts of plasma duration of PECVD-modified TNT (5 min and 10 min) and different coating materials (HEMA and MMA monomers) on the properties of TFN membranes.

(i) Characterizing surface properties of TFC and TFN membranes using different analytical instruments, including field emission scanning electron microscope (FESEM), FTIR, atomic force microscopy (AFM), X-ray photoelectron spectroscope (XPS), energy dispersive X-ray (EDX), and contact angle measurements.

(j) Comparing the pure water flux (PWF) and NaCl rejection of TFC and resultant TFN membrane incorporated with pristine and PECVD-modified TNT at 15 bar using 2000 ppm of NaCl aqueous solution.

For Objective 2:

(a) Studying the effects of two different types of hydrophilic monomers, i.e., AA and HEMA, on the surface properties and filtration performance of XLE commercial membrane (Dow FilmTec) based on the PECVD technique.

(b) Investigating the impact of plasma deposition time (15 s, 1 min and 5 min) on the surface properties of XLE commercial membrane.

(c) Characterizing surface properties of XLE commercial membrane with and without PECVD modification using FTIR, FESEM, contact angle measurements and zeta potential analysis.

(d) Selecting the optimum plasma duration and hydrophilic monomer from the surface modification of the XLE commercial membrane and apply it for self-synthesized TFC and TFN RO membranes (from objective one).

(e) Characterizing surface properties of PECVD-modified TFC and TFN membrane using different analytical instruments including FESEM, TEM, FTIR, AFM, zeta potential analysis, and contact angle measurements.

(f) Evaluating PECVD-modified TFC and TFN membrane performance with respect to PWF and NaCl rejection (2000 ppm NaCl aqueous solution) at operating pressure of 15 bar.

For Objective 3:

(a) Evaluating the effect of silica feed concentration (100 ppm, 168 ppm and 300 ppm) solution on anti-scaling properties of TFC, TFN, and PECVD-modified TFN membranes for 720 min at 15 bar. It is important to evaluate the silica resistance of membranes at different silica saturation level.

(b) Investigating the effect of silica feed pH value (pH 4, pH 6.7 and pH 10) on anti-scaling properties of TFC, TFN and PECVD-modified TFN membranes for 720 min at 15 bar.

(c) Evaluating the prolonged stability of unmodified and PECVD-modified TFN membranes in terms of anti-scaling properties for 4-cycle silica filtration process with each cycle lasted for 6 h at 15 bar.

(d) Characterizing the surface properties of scaled TFC, TFN, and PECVD-modified TFN membranes using different analytical instruments, including FTIR, FESEM, EDX and UV-vis spectrophotometer analysis.

(e) Studying the TNT leaching stability of TFN and PECVD-modified TFN membrane using pure water prolonged filtration test (up to 480 min).

(f) Comparing the TNT leaching tendency of TFN and PECVD-modified TFN membrane using inductively coupled plasma mass spectrometer (ICP-MS) to determine the Ti element in feed and permeate solution.

1.5 Significance of Study

The present RO technology has been in operation for over half a century in various industrial sectors. It has exhibited exemplary performance in removing wide range of dissolved ions including monovalent salts. However, despite its many advantages, one of the limitations of PA TFC RO membrane is the trade-off effect between water flux and selectivity, together with membrane scaling. The incorporation of TNT into membrane is believed to be able to overcome such trade-off effect. Although the incorporation of TNT can enhance membrane water flux and salt rejection, weak dispersion of hydrophilic TNT in organic solvent can result in severe TNT agglomeration. As a result, the membrane performance would deteriorate due to

the formation of surface defects/holes. Therefore, in this work, the outer surface of TNT is functionalized with hydrophobic monomer via PECVD method to improve the dispersion stability within the organic solvent during IP process. PECVD method offers progressive platforms for rapid surface functionalization of materials while allowing simultaneous tuning of surface morphology. Moreover, the highly versatile and environmentally friendly PECVD method only utilizes plasma discharge energy to activate and polymerize the precursors. It eliminates the use of any hazardous solvents/chemicals during the modification process. On the other hand, desalination industries rely heavily on chemical inhibitor to minimize silica scaling of RO membrane and this study introduces an eco-friendly PECVD surface modification technique to mitigate silica scaling onto TFN membranes by improving its surface chemistry. The deposition of hydrophilic AA containing carboxyl functional group as shown in this work is found to increase the membrane surface negativity, leading to improved antiscaling performance by repelling similar charge of from the membrane surface.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Khoo, Y. S.**, Lau, W. J., Liang, Y. Y., Karaman, M., Gürsoy, M. and Ismail, A. F. (2021) Eco-Friendly Surface Modification Approach to Develop Thin Film Nanocomposite Membrane with Improved Desalination and Antifouling Properties, *J. Adv. Res.* In Press. **(Q1, IF:10.479)**

2. **Khoo, Y. S.**, Seah, M. Q., Lau, W. J., Liang, Y. Y., Karaman, M., Gürsoy, M., Meng, J., Gao, H. and Ismail, A. F. (2021) Environmentally friendly approach for the fabrication of polyamide thin film nanocomposite membrane with enhanced antifouling and antibacterial properties, *Sep. Purif. Technol.*, 260, 118249. **(Q1, IF:7.312)**

3. **Khoo, Y. S.**, Lau, W. J., Liang, Y. Y., Yusof, N. and Ismail, A. F. (2021) Surface Modification of PA Layer of TFC Membranes: Does it Effective for Performance Improvement?, *J. Ind. Eng. Chem.*, 102, 271–292. **(Q1, IF: 6.064)**

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9 **Khoo, Y.S.**, Lau, W. J., Lai, G. S., Gray. S., and and Ismail, A. F. (2021) Deposition of Inorganic Particles on the Surface of RO Membrane via a Simple and Environmentally Friendly Approach based on Surface Mineralization, *Chem. Eng. J.*,Submitted. **(Q1, 13.273)**

10. Said, N., **Khoo, Y. S.**, Lau, W. J., Gürsoy, M., Karaman, M., Ting, T. M., Abouzari-Lotf, E. and Ismail, A. F. (2020) Rapid surface modification of ultrafiltration membranes for enhanced antifouling properties, *Membranes (Basel).*, 10(12), 1–15. **(Q2, IF:3.094)**

11. Seah, M. Q., **Khoo, Y. S.**, Lau, W. J., Goh, P. S. and Ismail, A. F. (2021) New Concept of Thin-Film Composite Nanofiltration Membrane Fabrication Using a Mist-Based Interfacial Polymerization Technique, *Ind. Eng. Chem. Res.*, 60(25), 9167–9178. **(Q2, IF:3.72)**

Non-indexed Journal

1. Zoka, L., **Khoo, Y. S.**, Lau, W. J., Ismail, A. F., Matsuura, T., Narbaitz, R. (2021) Flux Increase that Occurs when Ultrafiltration Membrane is Flipped from Normal to Inverted Position - Experiments and Theory, ACS Engineering Au. Under review.

Book Chapters

1. **Khoo, Y. S.**, Lau, W. J., Liang, Y. Y. and Ismail, A. F. (2021) Recent progress of polyamide thin film nanocomposite membranes for water applications. Handbook of Nanotechnology Applications. *Elsevier*, 125–145.
2. Origomisan, J. O., **Khoo, Y. S.**, Lau, W. J., Ismail, A. F., Adewuyi, A. (2022) A 15-year Review of Novel Monomers for Thin Film Composite (TFC) Membrane Fabrication for Water Applications. *Elsevier.* Submitted.

Patent

1. Lau, W. J., **Khoo, Y. S.**, Seah, M. Q., Goh, P. S., Ismail, A. F., Karaman, M., Gürsoy, M. and Ting, T. M. (2020). Method of Preparing Modified Thin Film Composite Membrane having Improved Surface Hydrophilicity and Antifouling Properties for Use in Water Treatment and Assembly Thereof. Application No.: PI2020007224. MyIPO