POLYAMIDE THIN FILM NANOCOMPOSITE MEMBRANE INCORPORATED WITH CARBON NITRIDE FOR FORWARD OSMOSIS DESALINATION

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DEDICATION

This thesis is dedicated to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

Forward osmosis (FO) is an emerging desalination process. It has been extensively studied to enhance the production of fresh water owing to its lower energy consumption and fouling tendency compared to the conventionally used reverse osmosis (RO). The design of a desired membrane structure has been recognized as one of the most crucial factors to counter some drawbacks of FO processes, such as internal concentration polarization effect and reverse passage of the draw solute. Hence, the main objective of this study is to develop a thin film nanocomposite (TFN) FO membrane for desalination application. The polyamide (PA) TFN FO membranes incorporated with protonated and unprotonated carbon nitride (CN) were prepared through interfacial polymerization of m-phenylenediamine and trimesoyl chloride. CN was synthesized through a thermal condensation method using melamine as the precursor. The protonated carbon nitride (pCN) was obtained by treating the as-synthesized CN with inorganic acid. pCN morphology observed less agglomeration nanosheet compared to CN and the size was shown to be approximately 28.95 nm based on the transmission electron microscopy images. Besides that, the acid treatment towards CN had changed the surface charge from -34.6 to 8.3 mV due to positive charged hydrogen absorption on the CN structure. Also, pCN peak on x-ray diffraction analysis pattern representing planar graphitic interlayer was shifted from 28° to 27.2° that makes the distance to become 0.325 from 0.318 nm. Meanwhile, on attenuated total reflectance Fourier transform infrared spectra, broader peak was observed on N-H stretching of CN instead of pCN. Performance evaluation of the TFN membrane was conducted in RO and FO modes. In RO mode, the water permeability and salt rejection were determined, while in FO system, the structural parameter and the reverse salt flux were determined in both active layers facing feed solution (AL-FS) and active layer facing draw solution (AL-DS). With the addition of pCN within the substrate, the pore and leaf-like structure became larger, as observed in the field emission scanning electron microscopy cross-sectional images. The presence of pCN had also increased the average surface roughness of the substrate. The formation of PA through IP was performed with 0.05, 0.1, and 0.15 w/v% loadings of pCN and CN dispersed in TMC monomer solution. Based on atomic force microscope images, the increasing loading of pCN and CN within the PA layer increased the surface roughness of the resultant TFN membrane as compared to that of TFC membrane. The decrease in water contact angle observed through goniometry analysis suggested the increase in the surface hydrophilicity of the TFN membrane. Other than that, the membrane surface charge was also changed. TFC membrane showed high negativity of -47.3 mV. However, the presence of pCN decreased the surface negativity to -5.76 mV and with the increasing loadings of CN, the negativity was further reduced to -10.2 mV compared to TFC membrane. The effect of the loading of nanomaterials in the range of 0.05 to 0.15 % on the performance of the membranes was also studied. Among the membranes prepared, 0.05 CN-pCN-TFN membranes which contained 0.05 w/v% CN in PA layer and 0.5 w/v% pCN within the support membrane was identified as the best performing membrane. The water flux achieved was 6.20 and 9.23 $Lm^{-2}h^{-1}$ in AL-FS and AL-DS mode, respectively. The reverse salt flux was recorded as 0.08 and 0.03 $gm^{-2}h^{-1}$ for AL-FS mode and AL-DS mode, respectively. With this optimal membrane, fouling behaviour was studied and compared with TFC membrane by using sodium alginate and bovine serum albumin (BSA) as model foulants. 0.05 CN-pCN-TFN membrane outperformed the TFC membrane in both tests with water flux reduced to 96 % after 9 h operation compared to TFC membrane which had reduced to 91.5 % for sodium alginate test and maintained at 100 % of water flux after 9 h operation for BSA compared to 97.5 % water flux for TFC membrane. This work evidenced the potential of using both CN and pCN in the design and fabrication of TFN to simultaneously achieve improved water flux, salt rejection and antifouling properties.

ABSTRAK

Osmosis hadapan (FO) adalah proses penyahgaraman yang sedang berkembang. Pelbagai penyelidikan dilakukan bagi meningkatkan penghasilan bekalan air bersih dengan penggunaan tenaga dan tahap pencemaran yang rendah berbanding osmosis balikan (RO) yang lazim digunakan. Rekabentuk struktur membran yang terbaik telah dikenalpasti sebagai faktor yang penting bagi mengatasi kekurangan proses FO seperti kesan pengutuban kepekatan dalaman dan pengaliran garam berbalik. Dengan sebab itu, tujuan utama kajian ini adalah untuk membangunkan membran osmosis hadapan nanokomposit filem tipis (TFN) untuk aplikasi penyahgaraman. Membran poliamida (PA) TFN FO campuran bersama karbon nitrida (CN) dan protonasi karbon nitrida (pCN) difabrikasi dengan cara pempolimeran antara permukaan antara monomer m-fenilenadiamina dan trimesoyl klorida. CN disintesis dengan menggunakan kaedah pemeluwapan haba terhadap melamin yang bertindak sebagai prapenanda. Proton CN (pCN) dihasilkan dengan merawat CN yang disintesis dengan campuran asid tak organik. Morfologi pCN menunjukkan kurang penggumpalan nanokepingan berbanding CN dan menunjukkan saiz hampir 28.95 nm berdasarkan imej mikroskop elektron penghantaran. Selain itu, rawatan asid terhadap CN telah mengubah cas permukaan daripada -34.6 kepada 8.3 mV disebabkan oleh penyerapan hidrogen bercas positif ke dalam struktur CN. Begitu juga, puncak pCN pada corak belauan sinar-X mewakili satah grafitik antara lapisan teranjak dari 28° ke 27.2° menjadikan jarak berubah kepada 0.325 dari 0.318 nm. Sementara itu, pada spektrum jumlah pantulan terkecil inframerah jelmaan Fourier, puncak yang lebih lebar dicerap pada regangan N-H CN berbanding pCN. Prestasi membran TFN dinilai melalui mod RO dan FO. Di dalam proses RO, kebolehtelapan air dan penyingkiran garam ditentukan, manakala di dalam sistem FO, parameter struktur dan fluks garam balikan ditentukan dalam lapisan aktif menghadap larutan suapan (AL-FS) dan lapisan aktif menghadap larutan larut (AL-DS). Dengan penambahan pCN di dalam substratum, struktur liang dan struktur berbentuk daun menjadi lebih besar berdasarkan pemerhatian pada imej keratan rentas mikroskop elektron imbasan pancaran medan. Kehadiran pCN juga telah meningkatkan purata kekasaran permukaan substratum. Pembentukan PA melalui IP dilakukan melalui penambahan pCN dan CN sebanyak 0.05, 0.1, dan 0.15 % yang diuraikan ke dalam larutan TMC. Berdasarkan imej daya atom mikroskop, penambahan nanopartikel pCN dan CN di dalam lapisan PA telah menyebabkan permukaan membran TFN lebih kasar berbanding membran TFC. Pemerhatian terhadap penurunan sudut sentuhan air melalui analisis goniometri menunjukkan permukaan hidrofilik membran TFN semakin meningkat. Selain itu, cas permukaan membran turut berubah. Membran TFC menunjukkan permukaan negatif yang tinggi (-47.3 mV). Manakala, peningkatan kandungan pCN mengurangkan kenegatifan sehingga mencecah -5.76 mV dan peningkatan jisim CN telah menurunkan kenegatifan sehingga -10.2 mV berbanding membran TFC. Kesan daripada penambahan nanopartikel adalah dalam julat 0.05 sehingga 0.15 % terhadap prestasi membran juga dikaji. Antara membran yang difabrikasi, membran 0.05 CN-pCN-TFN yang mengandungi 0.05 % CN di dalam lapisan PA dan 0.5 % pCN di dalam membran penyokong dikenalpasti sebagai membran yang terbaik. Ketelapan air mencapai 6.20 dan 9.23 $Lm^{-2}h^{-1}$ masing-masing dalam AL-FS dan AL-DS. Manakala, pengaliran garam berbalik direkodkan sebanyak 0.08 dan 0.03 $gm^{-2}h^{-1}$ masing-masing untuk AL-FS dan AL-DS. Dengan membran optimum ini, ketahanan membran ini dikaji dan dibandingkan dengan membran TFC dengan menggunakan natrium alginat dan albumin serum lembu (BSA) sebagai rujukan bahan cemar. Membran 0.05 CN-pCN-TFN mengatasi membran TFC dalam kedua-dua ujian dengan ketelapan air menurun kepada 96% selepas 9 jam operasi berbanding membran TFC yang menurun kepada 91.5 % di dalam ujian natrium alginat dan ketelapan air kekal sebanyak 100 % selepas proses selama 9 jam di dalam ujian dengan BSA berbanding 97.5 % ketelapan air bagi membran TFC. Kajian ini membuktikan potensi penggunaan kedua-dua CN dan pCN dalam rekabentuk dan fabrikasi membran TFN untuk sentiasa mencapai peningkatan di dalam ketelapan air, penyingkiran garam dan sifat anti cemar.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
AGQDs	-	Acid functionalized graphene quantum dots
AL-DS	-	Active layer facing draw solution
AL-FS	-	Active layer facing feed solution
AnOMBRs	-	Anaerobic osmotic membrane bioreactors
ATR-FTIR	-	Attenuated total reflectance-fourier transmission infrared spectroscopy
BSA	-	Bovine serum albumin
CN	-	Carbon nitride
CNF	-	Carbon nanofibers
CP	-	Concentration polarization
DMF	-	Dimethylformamide
DS	-	Draw solution
ECP	-	External concentration polarization
ED	-	Electrodialysis
EHT	-	Electron high tension
ePAN	-	Electrospun acrylonitrile
FESEM	-	Field emission scanning electron microscopy
FO	-	Forward osmosis
FS	-	Feed solution
FTIR	-	Fourier transform infrared spectroscopy
GO	-	Graphene oxide
ICP	-	Internal concentration polarization
IP	-	Interfacial polymerization
MED	-	Multi effect distillation
$MgCl_2$	-	Magnesium chloride
$MgSO_4$	-	Magnesium sulphate
MMM	-	Mixed matrix membrane
MOF	-	Metal organic framework
MPD	-	m-phenylenediamine

MSF	-	Multi stage flash
NaCl	-	Sodium chloride
NMP	-	N-methyl-2-pyrrolidone
NPs	-	Nanoparticles
PA	-	Polyamide
pCN	-	Protonated carbon nitride
PES	-	Polyethersulfone
PSf	-	Polysulfone
PVP	-	Polyvinylpyrrolidones
RO	-	Reverse osmosis
SEM	-	Scanning electron microscopy
TEM	-	Transmission electron microscopy
TFC	-	Thin film composite
TFN	-	Thin film nanocomposite
TMC	-	Trimesoyl chloride
TNT	-	Titanium dioxide nanotube
VIPS	-	Vapor-induced phase separation
XRD	-	X-ray diffractometer

LIST OF SYMBOLS

A	-	Water permeability coefficient ($m^3/m^2.s.Pa$)
B	-	Solute permeability coefficient (m/s)
J_s	-	Reverse solute flux ($g.m^{-2}.h^{-1}$)
J_w	-	Water flux ($m^3.m^{-2}.s^{-1}$)
M	-	Molarity
P	-	Pressure
R	-	Salt rejection
S	-	Membrane structural parameter
Π	-	Osmotic pressure
ΔP	-	Hydraulic pressure difference
w/v	-	Weight over volume

CHAPTER 1

INTRODUCTION

1.1 Research Background

About 71 % of the surface of the Earth is covered by water. Seawater holds about 97 % of total water in the Earth and only about 3.0 % of the freshwater sources is available as drinkable water (1). Unfortunately, 2.5 % of this drinkable water is frozen and locked up in Antarctica and Arctic as glaciers, hence hardly be reached by human. With this limited availability of drinkable water, it is a great challenge to fulfill the fresh water demand by global population that expected to rise up to 6900 billion m^3 in 2030 (2). The scenario has been further exacerbated with the population growth. Based on the figure revealed by the United Nations, the total population has reached about 7 billion in 2015 (3). By 2030, total population in the world are expected to increase until approximately 8 billion peoples. As water is the most essential component to survive, the demand of fresh water is expected to drastically increase with the increasing of the total global population (4–6).

Due to the increasing demand for the fresh water supply, various resources are being considered to fulfill the needs. These include the construction of a new reservoir in developed areas (2). Additionally, approaches have also been attempted to obtain fresh water from seawater, low-quality water, brackish water, storm water and wastewater (7). Among the strategies, the most promising method to fulfill the demand of freshwater supply is probably by desalination (5). Desalination has been regarded as a sustainable climate-independent solution for water shortage and most promising methods to recover fresh water supply considering the amount of seawater exists. Seawater is available in most countries, highly reliable and open access especially for countries that located at the coastal regions.

There are two types of desalination namely thermal-based and membrane-based desalination. In thermal desalination, energy or heat is used to evaporate the seawater. Then, the evaporated steam is condensed to produce fresh water (6). While in membrane desalination, membrane acts as a medium to separate water from the solute solution. The most promising technologies being used in membrane-based desalination industry is reverse osmosis (RO). A large number of RO desalination plants have been installed worldwide to address the water shortage issues (8). In RO operation, external hydraulic pressure is required as a driving force to flow water pass through the semi permeable membrane and produce fresh water (9). RO is a matured and well accepted desalination technology worldwide. Compared to thermal desalination, RO only needs relatively low energy consumption to produce fresh water and the product water is of high quality. Despite the attractive features, RO still faces some shortages. The energy requirement is still higher than many conventional water reclamation technologies which makes RO less affordable for many countries (10). Furthermore, the high hydraulic pressure operation has also accelerated membrane fouling tendency (6). As a result, additional cost is incurred for membrane cleaning and membrane replacement.

Forward osmosis (FO), an emerging desalination technology with low fouling tendency and low energy consumption has attracted attentions as promising alternative for RO desalination. FO system relies on osmotic pressure difference as a driving force for mass transport to purify seawater. The main advantage of using FO is that the osmotic pressure produced by the high concentration of solute solution is used as driving force for water to pass through the semi-permeable membrane. This favorable condition has also reduced the tendency of membrane fouling. The internal concentration polarization (ICP) issue that is related to the loss of draw solute into the feed solution and concentration polarization are the major problems of FO. Currently, FO membranes and system have been widely investigated and optimized to improve its capability to improve the water flux and fouling resistance as well as reducing the effect of ICP (6,11,12).

For FO membranes, the hydrophilicity of the membrane surface rather than reduce the thickness of the membrane is important to increase its performance (13). Recently, mixed matrix membrane and thin film nanocomposite (TFN) have been acknowledged as emerging nano-enabled membranes that hold good potential to solve the underlying issues of polymeric and inorganic membranes. Nanoparticles (NPs) such as silver, alumina, silica, zinc oxide, titanium dioxide (TNT) and graphene oxide (GO) are introduced as additive or nanofiller to enhance the performance of polymeric membranes (14,15). Up to present, many more NPs that being introduced and developed into the desalination research field. Among them is carbon nitride (CN) that structural properties identically to that of GO. With their chemical and thermal resistance, CN has been widely used as photocatalyst and semiconductor. The modification of FO membrane substrate layer and selective layer with nanoparticles is known to be one of the most straightforward approaches to increase the FO performance and counter the drawbacks of FO membranes. The usage of NPs in the polymer substrate and polyamide (PA) layer significantly improves the physico-chemical properties such as mechanical strength, hydrophilicity, surface charges, porosity and anti-fouling properties.

1.2 Problem Statements

Currently, membrane desalination has been acknowledged as one the most efficient and low-cost method to produce fresh water from seawater or brackish water. Up to date, RO is undeniably the most promising technology for desalination. However, one of the most significant drawbacks of RO desalination is the high energy consumption and severe membrane fouling, which eventually associated with the desalination production cost. FO comes with a great benefits and solution to overcome the problems in RO. As an osmotically pressure driven process, FO possesses some advantages such as low energy consumption and low fouling propensity compared to its RO counterpart. FO was favourable since it is driven by the osmotic pressure difference between two separate solution.

Despite its advantages, FO still suffers several limitations. ICP is one of the issues that responsible for the water flux decline in FO (16). ICP takes place at the substrate layer of the typically used thin film composite (TFC) FO membranes. Since ICP cannot be addressed by altering the hydrodynamic conditions, membrane modification has become a prominent approach to mitigate ICP effect. The increase of membrane substrate hydrophilicity has known to solve ICP problem that exists in TFC membrane (17,18). To achieve this purpose, TFN membrane that is embedded with nanomaterials serves as an attractive option. Hydrophilic nanomaterials that are embedded in the substrate layer or PA layer can significantly increase the water permeability of the FO membrane.

Like ICP, fouling is an inevitable issue of FO membranes. Although the fouling tendency of FO is generally lower than that of RO, fouling still happens on the membrane surface after long term operation. As a result, the overall permeability performance of the membrane decreases. Currently, many researchers focusing on altering the PA layer to improve their FO membrane performances either in water permeability, salt separation or anti-fouling properties and neglected the importance of substrate modification that also playing roles in inquiring the optimum membrane performance (19,20). In fact, both layers can be simultaneously approach and modified since the water transport not solely depends on the PA layer (21). Nanomaterials are incorporated onto or into the substrate or PA layer to reduce the adhesion of foulant thus ensure the performance of the membrane to be optimum as possible. Also, most of the studies only focused on the effect of hydrophilicity of NPs on the membrane performance (22,23). Effect of membrane surface charge has been scarcely reported Surface charge of the membrane also has an important role in determining the membrane performance since its characteristics is important in salt rejection. In addition, charged surface capable to reduce the fouling factor against the same charge molecule or solution (24).

In this research, PA TFN consists of polysulfone (PSf) substrate and PA selective layer was fabricated. Carbon nitride (CN) and protonated CN (pCN) was embedded in both PSf substrate and PA layer to simultaneously address ICP and fouling issues. The surface protonation of CN was aimed to alter the surface charge of the NPs, in order to facilitate the formation PA layer while maintaining the membrane surface hydrophilicity. Although many types of nanomaterials have been attempted to enhance the performance of TFN FO membranes, no studies have been reported on the effects of positively and negatively charged CN on the formation of substrate and PA layer of the TFN. Thus, it was expected that this study would provide insights into the physio-chemical properties and separation performance of the CN and pCN incorporated TFN for FO desalination.

1.3 Objective of the Study

The aim of this study is to fabricate TFN FO membrane incorporated with CN and pCN for desalination application. Based on the aim of this study, the specific objectives were listed below:

i) To fabricate and characterize PA TFN membranes that are incorporated with pCN in the substrate and CN or pCN in the PA layer.
ii) To evaluate the desalination performance of the TFN membranes with different loading of CN and pCN in terms of the pure water flux, reverse salt flux, salt rejection and antifouling properties in RO and FO processes.

1.4 Scope of the Study

In order to achieve the objectives, the following scopes have been identified.

Objective i:

1. Preparation of CN through condensation method using melamine as precursor.
2. Protonation of CN to form pCN through acid treatment using 5.0 M hydrochloric acid.

3. Characterization of CN and pCN in terms of morphology, crystallinity, functional group, and surface charge using transmission electron microscopy (TEM), X-ray diffractometry (XRD), attenuated total reflectance fourier transmission infrared spectroscopy (ATR-FTIR), and zeta potential analyzer, respectively.
4. Preparation of PSf substrate using phase inversion method. Substrate formulation was 17.5 wt% of PSf, 0.5 wt% of PVP K29-32 and 82 % of NMP. 0.5 wt% of pCN was added to produce PSf/pCN substrate.
5. Formation of PA layer on the neat and PSf/pCN substrate via interfacial polymerization (IP) of 2.0 w/v% amine monomer (MPD) in aqueous solution and 0.1 w/v% of acyl chloride monomer (TMC) in n-hexane solution.
6. Incorporation of CN and pCN with loadings of 0.05 w/v%, 0.1 w/v% and 0.15 w/v% respectively in the TMC phase prior to interfacial polymerization with MPD to form PA layer.
7. Characterization of the fabricated TFN membranes using field emission scanning electronic microscope (FESEM), attenuated total reflectance fourier transmission infrared spectroscopy (ATR-FTIR), atomic force microscopy (AFM), zeta potential analyzer and contact angle goniometer.

Objective ii:

8. Evaluation of TFN membranes performance in RO system and determination of S parameter for water permeability and NaCl rejection.
9. Performance evaluation of synthesized TFN membranes in terms of water flux and reverse draw solute using RO dead end permeation system (feed solution used distilled water and 2000 ppm of NaCl solution, pressure: 15 bar) and FO system (feed solution: distilled water, draw solution: 2 M NaCl solution, flow rates: 257.1 $mLmin^{-1}$).
10. Antifouling performance evaluation based on the optimum membrane using 500 ppm of sodium alginate and bovine serum albumin (BSA). The testing was performed for 9 h in active layer facing feed solution (AL-FS) mode.

1.5 Significance of the Study

This study was conducted to improve the membrane properties embedded with nanofillers thus enhance the performances of the membrane proved via salt rejection and water flux. Relation between water flux and salt rejection have attracted researcher to improve the membrane performances by adding the nanofillers in their TFN membrane. It was expected the water flux of embedded CN on TFN membrane improved without sacrificing the salt rejection due to the characteristic of CN that increased the hydrophilicity of the membrane. Other than that, acid treated CN reduced agglomeration of NPs in the PA layer, thus improving the water flux and salt rejection. It is also solved the reverse solute flux problem in FO. Besides that, the incorporation of CN and pCN within the TFN membrane has been proven to improve the membrane anti-fouling properties. This work is the first attempt to incorporate oppositely charged NPs in TFN membrane to improve the interaction between PA layer and substrate. Thus, the effects of opposite charges on membrane surface are investigated in depth to contribute to the advancement of knowledge in this aspect.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Abdul Aziz, A.**, Wong, K. C., Goh, P. S., Ismail, A. F., & Wan Azelee, I. (2020). Tailoring the surface properties of carbon nitride incorporated thin film nanocomposite membrane for forward osmosis desalination. Water Process Engineering, 33, 101005. https://doi.org/10.1016/j.jwpe.2019.101005. (**Q1, IF: 3.173**)

Non-indexed Journal

1. **Abdul Aziz, A.**, Goh, P. S., Azali, M. A., Zainal Abidin, M. N., & Abu Ba'dah, M. H. (2019). Protonated Carbon Nitride Incorporated Polyamide Thin Film Nanocomposite for Reverse Osmosis Desalination. Applied Membrane Science & Technology. 23, 29-43. https://doi.org/10.11113/amst.v23n2.153.