PERFORMANCE OF THIN FILM NANOCOMPOSITE MEMBRANES INCORPORATED WITH ZWITTERION AND TITANIA NANOTUBE FOR PRESSURE RETARDED OSMOSIS

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DEDICATION

To the most beloved mother and father
Saffiah Binti Sulaiman & Sharudin Bin Lot

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ABSTRACT

Osmotic power generation through pressure retarded osmosis (PRO) has been recognized as an alternative source of energy. Membrane is one of the major elements to guarantee the successful application of PRO for power generation. However, the major current limitation in PRO lies in the design of a high-performance membrane which is endowed with desired properties in terms of flux and anti-fouling properties. Hence, the main objective of this study was to fabricate a hydrophilic and high flux PRO thin film nanocomposite (TFN) membrane with high flux and anti-fouling properties through the incorporation of zwitterionic polymers, poly (3-methacryloylethoxy carbonyl pyridinium sulfopropyl betaine) (PMAPS) in the substrate and titanium dioxide nanotube (TNT) into the polyamide (PA) layer. Different loadings of PMAPS were physically mixed with polysulfone (PSF) dope prior to the formation of the TFC substrate. Further optimization via etching treatment was performed to increase substrate porosity and the PA selective layer incorporated with TNT was formed on top of the substrate through interfacial polymerization technique. Membrane characterizations were carried out using scanning electron microscope, transmission electron microscopy, Fourier-transform infrared spectroscopy, x-ray diffractometer, energy dispersive x-ray, and contact angle goniometer. The water flux and power density performance of the zwitterion incorporated TFN membranes were evaluated using a custom-made PRO system. The power density exhibited by etched TFN membrane incorporated with 2.0% PMAPS (PSF/PMAPS-2.0 Etched TFN) was 2.12 W/m^2 at 5 bar while unetched TFN membrane exhibited power density of 0.96 W/m^2 at 7 bar. Addition of TNT resulted in the highest power density of 2.22 W/m^2 at 5 bar. In terms of anti-fouling properties, PSF/PMAPS-2.0 Etched TFN achieved higher normalized water flux with 97% flux recovery compared to control substrate with 90% flux recovery. In conclusion, membrane modification using PMAPS zwitterions and TNT nanoparticles improved water flux, anti-fouling properties and power density.

ABSTRAK

Penjanaan kuasa osmosis melalui tekanan osmosis terencat (PRO) telah dikenal pasti sebagai sumber alternatif tenaga. Membran merupakan satu elemen utama untuk menjamin kebolehlaksanaan aplikasi PRO dalam penjanaan kuasa. Walau bagaimanapun, antara halangan utama dalam aplikasi PRO ini ialah reka bentuk membran berkeupayaan tinggi yang dapat memenuhi kriteria yang ditetapkan dari segi sifat fluks dan anti kotoran membran. Oleh itu, objektif utama dalam kajian ini ialah menghasilkan membran lapisan saput nipis poliamida komposit nano (TFN) yang mempunyai sifat hidrofilik serta kadar fluks dan tahap anti kotoran yang tinggi dengan melalui penggabungan polimer zwitterion poli (3-metakriloiletoksi karbonil piridinium sulfopropil betaina) (PMAPS) di dalam substrat dan tiub nano titanium dioksida (TNT) ke dalam lapisan poliamida (PA). Muatan berbeza PMAPS dicampurkan secara fizikal dengan dop polisulfona (PSF) sebelum pembentukan substrat TFC. Pengoptimuman seterusnya melalui rawatan punar dilakukan untuk meningkatkan keliangan substrat dan lapisan memilih PA digabungkan dengan TNT telah dibentuk di atas substrat melalui teknik pempolimeran antara muka. Pencirian membran telah dilakukan dengan menggunakan mikroskop electron imbasan, mikroskop elektron transmisi, spektroskopi inframerah jelmaan Fourier, difraktometer sinar-x, sinar-x pelepasan tenaga, dan goiniometer sudut sentuhan. Prestasi fluks air dan ketumpatan kuasa TFN membran yang digabungkan dengan zwitterion dinilai menggunakan sistem PRO buatan sendiri. Ketumpatan kuasa yang dipamerkan oleh membran TFN punar yang digabungkan dengan 2.0% PMAPS (TFN PSF/PMAPS-2.0 TFN punar) ialah 2.12 W/m^2 pada 5 bar, manakala bagi membran TFN tanpa punar mempamerkan ketumpatan kuasa 0.96 W/m^2 pada 7 bar. Penambahan TNT menghasilkan ketumpatan kuasa tertinggi dengan nilai 2.22 W/m^2 pada 5 bar. Dari segi sifat anti kotoran, PSF/PMAPS-2.0 TFN punar telah mencapai fluks air ternormal tertinggi dengan 97% perolehan fluks berbanding dengan substrat kawalan dengan 90% perolehan fluks. Kesimpulannya, pengubahsuaian membran dengan zwitterion PMAPS dan nanopartikel TNT telah meningkatkan fluks air, sifat anti kotor, dan ketumpatan kuasa.

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LIST OF ABBREVIATIONS

AEM	-	Anion exchange membrane
AFM	-	Atomic force microscopy
AgNPs	-	Silver nanoparticles
AL-DS	-	Active layer facing draw solution
AL-FS	-	Active layer facing feed solution
CAPMIX	-	Capacitive mixing
CEM	-	Cation exchange membrane
CNT	-	Carbon nanotubes
ECP	-	External concentration polarization
EDX	-	Energy dispersive x-ray
FO	-	Forward osmosis
FTIR	-	Fourier transform infrared spectroscopy
ICP	-	Internal concentration polarization
IEM	-	Ion exchange membrane
IP	-	Interfacial polymerization
MPD	-	1,3-phenylendiamine
MWCNTs	-	Multi-walled carbon nanotubes
NF	-	Nanofiltration
NMP	-	N-Methyl-2-pyrrolidone
PA	-	Polyamide
PAN	-	Polyacrylonitrile
PEI	-	Polyetherimide
PMAPS	-	Poly (3-methacryloylethoxy carbonyl pyridinium sulfopropyl betaine)
PRO	-	Pressure retarded osmosis
PSF	-	Polysulfone
PVDF	-	Poly(vinylidene fluoride)
PVP	-	Polyvinylpyrolidone
RED	-	Reverse electrodialysis
RO	-	Reverse osmosis

SEM	-	Scanning electron microscopic
SGE	-	Salinity gradient energy
SWRO	-	Seawater reverse osmosis
TEM	-	Transmission electron microscopy
TFC	-	Thin film composite
TFN	-	Thin film nanocomposite
TMC	-	Trimesoyl chloride
TNT	-	Titania nanotubes
UF	-	Ultrafiltration
XRD	-	X-ray diffractometer

LIST OF SYMBOLS

A	-	Water permeability coefficient ($L/m^2.h.bar$)
B	-	Solute permeability coefficient (m/s)
Js	-	Reverse salt flux ($g/m^2.h$)
Jv	-	Water flux ($L/m^2.h$)
M	-	Molality (M)
P	-	Pressure (bar)
R	-	Salt rejection (%)
S	-	Membrane structural parameter (mm)
π	-	Osmotic pressure (Pa)
ΔP	-	hydraulic pressure difference (bar)
W	-	Power density (Wm^{-2})
w/v	-	weight over volume

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CHAPTER 1

INTRODUCTION

1.1 Problem Background

Energy is a crucial aspect of every life on the earth including economic and technological development which is derived mostly from fossil fuel such as petroleum and coals (Chow *et al.*, 2009). From simulation studies conducted on global energy consumption, the energy consumption will face large increase in the tropic region countries by 7% and 17% under moderate and warming weather temperature respectively by 2050 (De Cian and Sue Wing, 2019). It is expected that the global energy demand will increase by 56% in 2040, and the global energy consumption will spike up to 240 TWh. However, the current stock of oil and gas can only sustain until 2042, while coals as another alternative of fossil fuel is expected to sustain until 2112 (Han *et al.*, 2014; Shafiee and Topal, 2009). Although the reserve of fossil fuel is not alarming in foreseeable future, the combustion of these fuel will lead to the increase of the greenhouse gasses such as carbon dioxide in the atmosphere. The carbon dioxide emission from fossil fuel and industry comprise 90 % of all CO_2 emission from human activities and it is the main factor that contributes to global climate change (Jackson *et al.*, 2017).

Reliability on natural resources energy surely one day will come to an end. New technology of energy generation must be developed as energy plays important role in the world evolution and advancement. The major challenge of the energy system is to ensure adequate supply of energy services at low cost, while not giving any bad impact to the environment locally and globally (Sagar and Holdren, 2002). Considering the current depletion of natural resources such as crude oil and coal, renewable energy is still relevant and needed to ensure the global energy supply is on its track. New type of renewable energy needs to be developed to eliminate or at least decrease our dependency on natural resources.

Green and renewable energy refers to the energy that is harvested from renewable resources in which they are replenished naturally through time, such as sunlight, rain, wind, tides, geothermal heat, and waves (Ellabban, Abu-rub and Blaabjerg, 2014). There are many advantages in renewable energy as its source will not run out, numerous benefits to health and environment with less carbon footprint, lower maintenance compared to natural energy resource, and many more. However, each type of them has their own limitations and disadvantages. Currently, solar energy has been utilized in many ways such as in the forms of photovoltaic system, solar hot water, solar electricity, passive solar heating and day lighting, and space heating & cooling. However, in order to commercialize solar energy, enormous space is needed to build the plant. Topaz Solar Farm located in California, United States takes almost 25 km^2 land space for their plant and this size is equivalent to 3500 standard football fields (Journal and Technologies, 2017). Such large area is not suitable for small country like Malaysia, Singapore, and many European countries and also there has no constant sun light throughout the year. As for wind energy, despite the zero-cost fuel and no production of harmful polluting gases, wind farms are noisy and may spoil the view for people living near them. Furthermore, the amount of electricity generated depends on the strength of the wind where if there is no wind, there is no electricity. On the other hand, geothermal energy is known to be the most expensive in terms of capital expenditure due to the cost of drilling wells to the geothermal reservoir as well as the cost of heating, and cooling system installation.

As an alternative of currently available renewable energy sources, salinity gradient energy (SGE) is one of the promising areas in which need to be developed as new energy source. Roughly, about 2 TW of SGE is available globally of which possibly 980 GW of energy can be harnessed if all river water discharging into the sea are systematically utilized and this equivalent to the supply of 80% of global electricity demand for 2018 (Güler and Nijmeijer, 2018)(Logan and Elimelech, 2012). By continuing research and development on SGE as an alternative for energy generation, it is believed that our dependency and reliability towards fossil fuel can be reduced. One of the technologies introduced in SGE is pressure retarded osmosis (PRO). PRO is a new form of renewable energy that converts the pressure difference between two water flows with different salinity gradient which are waters with high salinity to water with low salinity or no salinity into hydraulic pressure. The hydraulic pressure can be used to drive a turbine in order to produce electrical energy. PRO holds the potential to produce renewable energy from natural and anthropogenic salinity gradients (Yip *et al.*, 2011) PRO has many advantages such as it can be operated 24 hours daily, not affected by wind and solar radiation, small foot-print, and easy to scale-up. A PRO prototype plant system was developed in Norway in the late 2009 (Achilli and Childress, 2010) by using combination of river water as feed solution and sea water as draw solution. In terms of energy production, the power density of PRO is known to directly proportional to the water flux. Hence, desired range of power density cannot be achieved because of low water flux through the membrane.

Normally, ideal PRO requires high water flux, high salt rejection, high mechanical strength, and low fouling tendency (Cai *et al.*, 2016). Two major factors that affect PRO productivity are PRO membrane and the feed pair which are feed and draw solutions. Feed and draw solutions must be two different solutions with different salinity. The greater the salinity difference, the higher the osmotic pressure, and this leads to high water flux and results in higher power density. The foulant molecules from feed and draw solutions during separation process tends to clog the membranes by depositing the retained inorganic materials, or organic compound and microorganism on the membrane pores. This problem is known as membrane fouling and it affects the productivity of the membrane over the operation time. This problem not only causes inconvenience for practical operation, but also increase the operating cost due to membrane cleaning and replacement process and also increase the energy input for PRO operation (Shahkaramipour *et al.*, 2017). This fouling problem leads to the significant decrease in power density of PRO system, and hence the energy is produced below the expectation for practical application. Concerns on anti-fouling performance have raised among membrane researchers as fouling effect can reduce water flux with time. To address this problem, various methods can be adopted such as pretreatment of feed solution, periodic cleaning, or surface modification of membrane. In terms of feasibility, surface modification of PRO membrane via molecular design is a preferred method to deal with fouling problem without affecting the membrane bulk properties (Cai *et al.*, 2016).

In recent years, researchers have shown interest in developing polymeric membrane with the incorporation of zwitterionic polymer (Bengani-Lutz *et al.*, 2017; Wang, Y. lei Su, *et al.*, 2009; Zhu *et al.*, 2017). The key components that make zwitterionic polymer as a new material to be incorporated in separation membranes are the ability to improve hydrophilicity of the membrane itself and to exhibit excellent anti-fouling properties. It has been reported that zwitterionic poly(arylene ether sulfone) incorporated with poly(vinylidene fluoride) (PVDF) resulted in excellent anti-fouling properties and good thermostability (Rong *et al.*, 2018). Zwitterion, which also known as dipolar ion, is a molecule that has two or more functional groups, in which one has a positive electrical charge and the another one has the negative electrical charge, ang hence the net electrical charge of the entire molecule is zero (Mi *et al.*, 2017). The incorporation of zwitterion and membrane can be achieved via phase inversion, interfacial polymerization (IP) or spray grafting method. Zhu et, al. blended zwitterion with PVDF to create a membrane via phase inversion method for oil in water emulsion separation (Zhu *et al.*, 2017). It was reported that the zwitterion cross-linked membrane reached nearly 91% of permeate recovery. On the other hand, TFC was formed via IP of 3, 3′-diamino-N-methyldipropylamine (DNMA) zwitterion and trimesoyl chloride (TMC) (Mi *et al.*, 2017).

Apart from zwitterion polymer, titanium dioxide nanotubes (TNT) has also been widely used in various applications such as in fuel cell technology, photocatalytic system, sensors, energy storage, and environmental analysis (Abdullah and Kamarudin, 2017). Researchers incorporated TNT into the membrane substrate and polyamide (PA) active layer as nanofiller to enhance the properties of the membranes. This inorganic photocatalytic nanomaterial attract the interest of researchers due to their unique properties such as self-cleaning, and anti-fouling, anti-microbial (Geng *et al.*, 2019). Subramaniam et al., (2016) incorporated TNT with polyether sulfone (PES) via physical blending method. The incorporation of TNT into PES showed improvement of 20% membrane flux and the rejection was improved from 79% to 96%.

1.2 Problem Statement

Salinity gradient energy created by PRO commonly falls between 0.70–0.75 kWh (2.5–2.7 MJ) when 1 m^3 of river water mixes with 1 m^3 of sea water (Yip and Elimelech, 2012). But in current situation, the practicability of PRO is low due to the poor performance of PRO membranes. The two major limitations related to PRO membranes are their low flux and fouling issues. Currently, no commercial PRO membrane is available, and the early studies of PRO were based on the use of commercial RO membranes. The thick layer support has resulted in unfavorable low permeate flux. While sufficiently thick membrane support is required to render high mechanical strength in order to withstand the hydraulic pressure of PRO system, the water flux can be improved by increasing the membrane hydrophilicity. Currently, one of the most feasibly used methods to achieve this purpose is through the incorporation of hydrophilic nanoparticles into the TFC membranes. However, one concern with this strategy is the incompatibility of the inorganic nanofiller and polymeric phases where the dispersion of the nanofillers is difficult to be achieved. This has in turn caused the formation of defective membranes.

Like other membrane processes, PRO suffers from membrane fouling which is caused by the contaminants present in feed water. Zwitterionic materials have been extensively studied as hydrophilic and fouling-resistant modifiers for membrane surface due to their absorption resistance towards organic compounds. One of the most common approaches to introduce zwitterion is through membrane surface coating. However, this method has several limitations such as lack of surface functionalities for surface coating, leaching of coating layer and increased surface roughness which may result in more severe fouling. In this study, the blending of PMAPS zwitterion polymer into the substrate layer was proposed to overcome the abovementioned issues. Meanwhile, TNT has been used to address the fouling issue. The incorporation of TNT in membranes can be achieved by various method such as physical blending (Subramaniam *et al.*, 2016), physical deposition (Veréb *et al.*, 2019), and PA thin film nanocomposite modification (Azelee *et al.*, 2017).

The aim of this research was to develop highly hydrophilic and high-flux PRO membranes that also have an excellent anti-fouling performance to offer high power density. In this study, zwitterionic polymer, a highly hydrophilic material, was used in modifying the substrate of the PRO membrane to increase the water flux and render good anti-fouling properties. TNTs were also incorporated within the PA (PA) layer for better water flux in order to increase the power density of PRO system. Physical blending approach between zwitterionic polymer and common polymer was used to create the substrate dope solution. Phase inversion casting was performed to form the substrate of the membrane and followed by IP to form the thin film selective layer. Characterizations tests were performed to investigate the effects of zwitterionic polymer modification on the properties of the resultant modified TFC membrane. Lab scale PRO system was used to determine the flux, power density and anti-fouling properties of the membranes.

1.3 Objectives of Study

In order to address the abovementioned problems, the objectives of this study are set as below:-

1. To characterize PRO TFC membrane with substrate incorporated with PMAPS zwitterionic polymer.

2. To optimize and characterize TFC membrane incorporated with TNT in PA active layer.

3. To evaluate the performance of the PRO membranes in terms of flux, power density and anti-fouling performance through PRO system.

1.4 Scope of Study

To achieve the objectives of this study, the following scopes have been derived:

1. Characterization of PMAPS using Fourier-transform infrared spectroscopy (FTIR) for chemical functional group studies.

2. Preparation of membrane substrate dope based on 15% of polysulfone (PSF), 84% of n-methyl-2-pyrrolidone (NMP) solvent and 1% of polyvinylpyrrolidone (PVP) pore former.

3. Mixing of 0.5-2.0 wt% of zwitterionic polymer into the PSF dope through physical blending method.

4. Fabrication of membrane substrate through phase inversion technique. Substrate etched using 500 ppm of sodium hypochlorite (NaOCl) (4-4.99%) aqueous solution for 1 hour.

5. Fabrication of TNTs nanoparticles using hydrothermal synthesis method and titanium dioxide (TiO_2) powder and 10 M of sodium hydroxide were used during the process.

6. Characterization of TNTs nanoparticles using transmission electron microscopy (TEM), x-ray diffraction (XRD) and FTIR for morphology and chemical functional group studies.

7. Fabrication of PA thin film using 2% (w/v) m-Phenylenediamine MPD aqueous solution, 0.1% (w/v) trimesoyl chloride (TMC) solution in n-hexane, and 0.5% (w/v) of TNTs via IP

8. Characterizations of fabricated TFC membrane using scanning electronic microscope (SEM), Fourier-transform infrared spectroscopy (FTIR) and

contact angle meter goniometer for morphology, chemical functional group, and hydrophilicity, respectively.

9. Evaluation of neat TFC, zwitterion incorporated TFC, and TFN membrane using lab scale PRO module for water flux and power density. 3, 5, 7, and 10 bars of operating pressure used. RO water used as feed solution while 2 M of NaCl used as draw solution.

10. Calculation of water flux, reverse salt flux, power density, and normalized water flux to compare the performance of neat TFC and zwitterion incorporated TFC membrane with and without TNT.

1.5 Significance of Study

High water flux and salt rejection are great concerns for membrane properties and have attracted many researcher attentions' in their studies. The application of zwitterionic polymer has been proven to render more hydrophilic properties to the membrane and improve the anti-fouling performance of the membranes. This study was the first attempt of applying novel PMAPS zwitterionic polymer in flat sheet membrane substrate and addition of TNTs nanoparticles for PRO application. It did give higher power density than current PRO membrane available in industry.

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LIST OF PUBLICATIONS

Indexed Journal

1. **Sharudin, S.**, Goh, P., Ismail, A. (2019). 'Modification of Polymeric Membrane for Energy Generation through Salinity Gradient: A Short Review', Journal of Membrane Science and Research, (), pp. 0-0. https://doi.org/10.22079/jmsr.2019.115128.1294

Conferences

1. **National Congress on Membrane Technology (NATCOM 2018)**
   30th – 31st of October 2018, Pulai Springs Resort, Johor Bahru, Malaysia.
   Oral presentation on *"ZWITTERION INCORPORATED THIN FILM COMPOSITE MEMBRANE FOR POWER GENERATION THROUGH PRESSURE RETARDED OSMOSIS"*

2. **International Conference of Sustainable Environment Technology 2019 (ISET)**
   20th – 22nd of August 2019, Double Tree by Hilton, Johor Bahru, Malaysia.
   Oral presentation on *"ZWITTERION INCORPORATED THIN FILM COMPOSITE MEMBRANE FOR POWER GENERATION THROUGH PRESSURE RETARDED OSMOSIS*