THIN FILM NANOCOMPOSITE REVERSE OSMOSIS MEMBRANE WITH LAYER BY LAYER ASSEMBLED TITANIA NANOSHEETS FOR PRODUCED WATER DESALINATION

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DEDICATION

This thesis is dedicated to my beloved mother, Sobberiah Binti Shuib and father, Ahmad Bin Hasan who have always been a source of inspiration, encouragement and stamina to undertake my study and to face the eventualities of life with zeal, enthusiasm and fear of Allah

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ABSTRACT

Reverse osmosis (RO) is an emerging desalination technology that holds great potential to provide an effective approach for solving global water scarcity issues. In the blooming oil and gas industries, produced water (PW) desalination is a viable option to resolve the oily water disposal issue. However, one great challenge of PW desalination is the membrane fouling caused by the presence of hydrocarbon contents in the PW. In this study, thin film nanocomposite (TFN) membrane for RO desalination was fabricated by depositing positively charged titania nanosheet (pTNS) and negatively charged titania nanosheet (nTNS) on the surface of polyamide (PA) layer via layer by layer (LbL) assembly. The pTNS was synthesized through solid-state calcination and acid ion-exchange. Through the additional step of exfoliation, single nTNS with improved hydrophilicity property was obtained. The formation of pTNS/nTNS assembly that created as TNS was formed atop PA layer with the number of bilayer ranged from 0 to 4. The hydration layer created on the surface of TNS-PA could hinder direct contact of foulants with the membrane surface, hence significantly enhanced the water permeability and salt rejection as well as foulant resistance. The characterization findings revealed that the membrane surface hydrophilicity was improved while surface roughness was decreased by increasing number of bilayer. However, the excessive TNS bilayer coating has imposed additional hydraulic resistance, resulting in the reduction of water permeability. The highest water permeability of 0.97 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$ (33% improvement) was achieved with the 2-bilayer of TNS-PA TFN membrane compared to the pristine PA membrane. The sodium chloride (NaCl) rejection was >98% which was also higher than pristine thin film composite (TFC) membrane of 96%. Furthermore, the 2-bilayer of TNS-PA TFN membrane achieved >99% for oil rejection. On the other hand, manipulating nanomaterials loading (0-0.10 wt.%) of 2-bilayer could lead to more positive features on the resultant TFN membrane. The effect of 0.05 wt.% TNS loading in tandem with controlled 2 bilayer achieved the highest permeability and solute rejection, recording 0.98 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$ and >98%, respectively. This study also found that the 2TNS-PA TFN membrane outperformed the pristine TFC membrane by exhibiting higher permeability and much lower fouling propensity for low to high concentration of saline oily water (2000 ppm, 5000 ppm and 10,000 ppm) and oily saline water (1000 ppm, 5000 ppm and 10,000 ppm) over a 960 min operation. With saline oily feedwater, the permeability relative rate became low with 84.48%, 78.82% and 78.82%, respectively. However, for oily saline feedwater, the 2TNS-PA TFN membrane achieved almost 100% flux recovery for three cycles by hydraulic washing. While the average permeability of uncoated TFC membrane could only be recovered by 95.7%, 89.1% and 82.9% for 1000 ppm, 5000 ppm and 10,000 ppm of the oily saline feedwater, respectively. The 2TNS-PA TFN membrane suffered a less significant decline and exhibited a relatively higher average permeability recovery rate compared to the TFC membrane due to the better resistance to the oil adsorption on the skin layer. Overall, the surface modification of TNS on the TFC membrane can provide hydrophilic, stable and effective thin water layer to tackle the fouling problem encountered by the TFC membrane.

ABSTRAK

Osmosis terbalik (RO) adalah teknologi penyahgaraman yang berpotensi besar dalam memberikan pendekatan yang berkesan bagi menyelesaikan masalah kekurangan air global. Dalam industri minyak dan gas yang kian membangun, penyahgaraman bagi air yang dihasilkan semasa pengekstrakan minyak (PW) adalah pilihan yang tepat untuk menyelesaikan masalah pelupusan air berminyak. Namun, satu cabaran besar penyahgaraman PW adalah pengotoran membran yang disebabkan oleh adanya kandungan hidrokarbon dalam PW. Dalam kajian ini, membran komposit nano filem tipis (TFN) untuk penyahgaraman RO dihasilkan dengan mendepositkan lembaran nano titania bercas positif (pTNS) dan lembaran nano titania bercas negatif (nTNS) pada permukaan lapisan poliamida (PA) melalui kaedah lapisan demi lapisan (LbL). pTNS disintesis melalui pengkalsinasi keadaan pepejal dan pertukaran ion asid. Melalui langkah pengelupasan, nTNS lembaran tunggal dengan sifat hidrofilik dapat diperoleh. Gabungan pTNS/nTNS sebagai dual lapisan TNS terbentuk pada lapisan PA dengan bilangan dual lapisan TNS yang berbeza dari 0-4. Lapisan hidrasi yang terbentuk di permukaan TNS-PA dapat menghalangi kontak langsung kotoran dengan permukaan membran TFN, akibatnya dapat meningkatkan kebolehtelapan air dan penolakan garam serta ketahanan kotoran. Penemuan pencirian menunjukkan bahawa hidrofilik permukaan membran diperbaiki dan kekasaran permukaan menurun dengan bertambahnya bilangan dual lapisan TNS. Walau bagaimanapun, dual lapisan TNS yang berlebihan telah memberikan ketahanan hidraulik tambahan sehingga mengakibatkan penurunan kebolehtelapan air. Kebolehtelapan air tertinggi 0.97 $\mathrm{L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}}$ (peningkatan 33%) dicapai dengan 2-dual lapisan TNS-PA TFN berbanding dengan membran PA. Penolakan natrium klorida (NaCl) adalah > 98% yang juga lebih tinggi daripada membran TFC dengan penolakan 96%. Membran TFC yang dilapisi 2-dual lapisan TNS mencapai > 99% untuk penolakan minyak. Sebaliknya, manipulasi pemuatan 2-dual lapisan TNS pada membran secara signifikan dapat menunjukkan potensi yang lebih positif. Antara 0-0.10 wt.%, kesan pemuatan TNS 0.05 wt.% terhadap 2-dual lapisan, menentukan kebolehtelapan tinggi dan penolakan zat terlarut masing-masing dengan 0.98 $\mathrm{L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}}$ dan > 98%. Semasa kajian, membran TFN 2TNS-PA mengungguli membran TFC dengan menunjukkan kebolehtelapan tinggi dan kecenderungan pengotoran yang jauh lebih rendah bagi kepekatan rendah ke tinggi untuk air masin berminyak (2000 ppm, 5000 ppm dan 10,000 ppm) dan air minyak bergaram (1000 ppm, 5000 ppm dan 10,000 ppm) selama 960 min operasi. Dengan air masin berminyak, kadar relatif kebolehtelapan menjadi rendah masing-masing dengan 84.48%, 78.82% dan 78.82%. Namun, bagi air minyak bergaram, membran TFN 2TNS-PA mencapai pemulihan fluks hampir 100% selama tiga kitaran dengan cucian hidraulik. Sementara itu, kebolehtelapan rata-rata membran TFC yang tidak dilapisi hanya dapat dipulihkan sebanyak 95.7%, 89.1% dan 82.9% untuk 1000 ppm, 5000 ppm dan 10,000 ppm air minyak bergaram. Membran TFT 2TNS-PA mengalami penurunan yang kurang ketara dan menunjukkan kadar pemulihan kebolehtelapan purata yang lebih tinggi berbanding dengan membran TFC kerana ketahanan yang lebih baik terhadap penjerapan minyak pada lapisan kulit. Secara keseluruhan, modifikasi permukaan TNS pada membran TFC dapat memberikan lapisan air nipis yang hidrofilik, stabil dan berkesan untuk mengatasi masalah pengotoran pada membran TFC.

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LIST OF ABBREVIATIONS

0D	-	0 dimensional
1D	-	1 dimensional
2D	-	2 dimensional
AFM	-	atomic force microscopy
AGO	-	aminated graphene oxide
BOD	-	biological oxygen demand
BSA	-	bovine serum albumin
CA	-	cellulose acetate
CNT	-	carbon nanotube
COD	-	chemical oxygen demand
CP	-	concentration polarization
DTAB	-	dodecyltrimethylammonium bromide
EDX	-	energy dispersed x-ray
FESEM	-	field emission scanning electronic microscopy
FO	-	forward osmosis
FTE	-	freeze-taw/evaporation
FTIR	-	fourier transform infrared spectroscopy
GO	-	graphene oxide
HBD	-	hydrate-based desalination
HCl	-	hydrochloric acid
HRTEM	-	high resolution transmission electron microscopy
ICP-MS	-	inductively coupled plasma mass spectrometry
IP	-	interfacial polymerization
LbL	-	layer by layer
MD	-	membrane distillation
MF	-	microfiltration
mLbL	-	molecular layer by layer

MPD	-	1,3-phenylenediamine
MVR	-	mechanical vapor recompression
MW	-	molecular weight
MWCO	-	molecular weight cut-off
NaOH	-	sodium hydroxide
NF	-	nanofiltration
NMP	-	n-methyl-2-pyrrolidone
NOM	-	natural organic matter
nTNS	-	negative charged of titania nanosheets
PA	-	polyamide
PAA	-	polyacrylic acid
PAMAM	-	polyamidoamine
PDA	-	polydopamine
PEG	-	polyethylene glycol
PEGMA	-	poly(ethylene glycol) methyl ether methacrylate
PES	-	polyethersulfone
PhA	-	phytic acid
PSf	-	polysulfone
PSM-AgNP	-	polyelectrolyte-silver nanoparticle
pTNS	-	positive charged of titania nanosheets
PVDF)-g-PTA	-	polyvinylidene fluoride-terephthalic acid
PVP	-	polyvinylpyrolidone
PW		produced water
RO	-	reverse osmosis
SA	-	sodium alginate
SDS	-	sodium dodecylbenzenesulfonate
SEM	-	scanning electronic microscopy
TBAOH	-	tetrabutylammonium hydroxide
TDS	-	total dissolved solids

TEM	-	transmission electron microscopy
TFC	-	thin film composite
TFN	-	thin film nanocomposite
Ti	-	titanium
TMC	-	trimesoyl chloride
TNP	-	titania nanoparticle
TNR	-	titania nanoribbon
TNS	-	titania nanosheet
TNS-PA TFN	-	titania nanosheets polyamide thin film nanocomposite
TNT	-	titania nanotube
TOC	-	total organic carbon
UF	-	ultrafiltration
UV-vis	-	ultraviolet–visible
XRD	-	x-ray diffractometer
M3-PALS	-	mixed-mode measurement phase analysis light scattering

LIST OF SYMBOLS

π - osmotic pressure

λ - wavelength

θ - diffraction angle

A - cross-sectional area (m^2)

A_f - absorbance of UV light by the feed solutions

A_p - absorbance of UV light by the permeate solution

$A_{w,o}$ - water permeability of oily saline

$A_{w,s}$ - water permeability of salt

b - TNS bilayer

$C_{f,o}$ - concentration of oil feed (abs)

$C_{f,s}$ - concentration of salt feed

$C_{p,o}$ - concentration of oil permeate oil (abs)

$C_{p,s}$ - concentration of salt permeate

d-spacing - interlayer-spacing

E_S - tangential streaming potential

ε_0 - vacuum permittivity

ε_r - relative dielectric constant

F - water permeability

J_0 - initial permeability

Jr - relative permeability recovery

J_t - steady permeability

J_{wv} - permeability of fresh membrane

J_{wp} - permeability of washed membrane

m - cross-link of the PA layer

n - linear parts of the PA layer

P - pressure gradient

PRR - permeability recovery rate

R - rejection

$\mathrm{R_a}$ - average plane roughness

$\mathrm{R_{ir}}$ - irreversible fouling ratio

$\mathrm{r_m}$ - average pore radii

$\mathrm{R_o}$ - oil rejection

$\mathrm{R_s}$ - solute separation efficiency

t - fixed time

t - time treatment (h)

V - total amounts of the collected permeate (L)

$\ddot{\mathrm{y}}$ - pTNS loading

$\check{\mathrm{z}}$ - nTNS loading

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CHAPTER 1

INTRODUCTION

1.1 Research Background

‘Thousands have lived without love, not one without water’.

Wystan Hugh Auden

Water is essential to life. Demand for fresh water has been accelerating exponentially with the overgrown population and rapid industrialization. Water reclamation from various potential sources has become an attractive solution to fulfil the increasing demand for freshwater (Alsalhy, Albyati and Zablouk, 2013). Oily saline wastewater, also known as produced water (PW), is a potential water source as it is one of the largest waste streams produced in the oil and gas industry (Azetsu-Scott *et al.*, 2007). By eliminating the dissolved and suspended components present in PW, the treated water can be used to serve multiple purposes, including potable and non-potable uses. Water reclamation from PW also offers another advantage related to handling and disposal of hazardous PW. Oily saline wastewater containing a high amount of dissolved and dispersed hydrocarbons, surfactants, clay particles and salts is considered as one of the main water pollutants. The allowable limit for the total oil and grease discharged is normally set in the range of 10–15 mg/L (Gohari *et al.*, 2015; Ge, Amy and Chung, 2017). Oily saline wastewater needs to be treated in order to effectively alleviate its adverse environmental impact.

Thus, many technologies such as coagulation, skimming, floatation and hydrocyclones have been attempted for the treatment of PW (Maltos *et al.*, 2018; Ostarcevic *et al.*, 2018; Elhady *et al.*, 2020). Most of these techniques are solely suitable for pre-treatment of wastewater or as for oil/water separation and limited by low separation efficiency with a decrease in stabilized oil droplet size (below ~10μm) (Tummons *et al.*, 2017). Despite the long history of thermal-based and hydrate-based desalination, these techniques still suffer from several drawbacks, such as high cost, toxic compounds utilization, large space for installation and generation of secondary pollutants which do not meet the water effluent standard. In most cases, the PW treatment achieved not more than 90% rejection of salt. Membrane-based process is a promising desalination technology with the prominent advantages including removal efficiency, low energy requirement, smaller footprint, simple installation, operation, and scaling up (Dickhout *et al.*, 2017). Membrane-based PW desalination is commonly achieved by membrane distillation (MD), forward osmosis (FO) and reverse osmosis (RO) (Samaei, Gato-Trinidad and Altaee, 2018). MD, FO and RO are driven by thermal, osmotic and pressure, respectively. The main driving factor for implementing FO and MD system is associated to the energy consumption and capital expenses that are lower in relative to the conventional pressure-driven system. Nevertheless, the large-scale application is impeded by the challenge in draw solution recovery for FO and membrane wetting for MD.

Among membrane processes, RO has witnessed a huge commercial success by representing about 65% of the worldwide installed desalination capacity (Liu and Xu, 2016; Asadollahi, Bastani and Musavi, 2017; Maltos *et al.*, 2018; Goh and Ismail, 2020). It has been touted as an effective process to treat oily saline wastewater generated from oil and gas industry (Al-Jeshi and Neville, 2008; Baransi-Karkaby, Bass and Freger, 2019). This mature technology is promising in removing many types of molecules, including oil particles and ions, to yield fresh water that is suitable for potable and industrial uses (Li *et al.*, 2012; Maltos *et al.*, 2018; Pei *et al.*, 2018; Yang *et al.*, 2019). At present, thin film composite (TFC) membrane remains the dominant membrane used for RO desalination. The asymmetric structure consisting of a thin dense top layer and porous substrate bottom layer. Regardless of the types of driving forces, fouling is the huge barrier to the widespread implementation of membrane-based filtration, particularly when treating saline water with high oil concentration. In PW desalination, the coalescence of emulsified oil found in oily saline wastewater can easily foul the RO membranes (McCloskey *et al.*, 2012). The stabilized emulsion PW with a size smaller than 10 μm is the most stable oil droplet and tends to attach to the membrane surface. Fouling by these oil droplets takes place within a short period and significantly shortens the operation lifetime of the RO membrane (Jhaveri and Murthy, 2016).

When dealing with hydrophobic foulant, such as emulsified oil, it is generally agreed that smooth and hydrophilic membrane surfaces can limit the surface–foulant hydrophobic interaction, hence suppressing membrane fouling (Huang, Ras and Tian, 2018). The incorporation of highly functional nanomaterials into or onto the substrate and/or PA layer improves the membrane surface and intrinsic properties, hence enhancing the efficiency of RO desalination (Ji *et al.*, 2012; Pei *et al.*, 2018). Titanium dioxide (TiO_2) nanostructures is the most popularly investigated metal-oxide in addressing variety of environmental problem likes water purification (Kim *et al.*, 2011; Chambers *et al.*, 2017; Azizi-Lalabadi *et al.*, 2019). It is notable for its significant photocatalytic effects that decompose organic chemical and kill bacteria (Nakata and Fujishima, 2012; Shao *et al.*, 2017). TiO_2 that has hydrophilic character that surrounded by hydroxyl group could interact well with water. Therefore, it has been applied to membrane modification for water purification (Kim *et al.*, 2003; Li *et al.*, 2009).

Membrane modification is one of the most versatile techniques to combat RO membrane fouling (Liu *et al.*, 2018). The introduction of surface modifiers, such as hydrophilic nanomaterials and antimicrobial polymers, renders excellent fouling resistance and enhanced membrane longevity and performance (McCloskey *et al.*, 2012; Jafarinejad, 2017; Zhang *et al.*, 2018; Zaidi, Mauritz and Hassan, 2019). In the last two decades, thin film nanocomposite (TFN) membranes have emerged as a new trend in membrane technology. As its name implied, TFN membrane is incorporated with various types of functional nanomaterials to obtain the desired physico-chemical properties. Two main approaches that have been widely explored to prepare TFN membrane are based on i) post modification of TFC membrane through surface coating and ii) direct incorporation of nanoparticles within PA layer or/and microporous substrate. However, the conventional coating led to experience in mass transfer resistance (Kasemset *et al.*, 2013). Surface modification with layer by layer (LbL) technique could address the trade-off between rejection and permeability (Xu *et al.*, 2015). The number of LbL layer could be carefully controlled during the deposition process to enhance its performance and develop high performance of membrane for water desalination by accounting the selectivity, the flux and fouling resistant.

1.2 Problem Statement

Owing to the complexity of PW content, handling both oil particles and dissolved ions using a single RO stage filtration is uncommon. In order to reduce the reliance of the pre-treatment system and improve the reliability of single stage RO for PW treatment, the physico-chemical properties of the TFC membrane must be carefully tailored to address the inherent fouling issues of the conventional RO membranes (Maphutha *et al.*, 2013). Surface modification of TFC membrane is a feasible approach to improve the performance of single-stage RO for PW treatment (Kasemset *et al.*, 2013). However, surface modification such as thin film coating and self-assembled coating usually generates thick and mass transfer resistance of polymer layer, thus lead to the reduction permeate flux (McCloskey *et al.*, 2012). Therefore, a more holistic approach is needed to strike a good balance between the separation performance and fouling resistance. In this study, LbL technique has been implemented in order to prepare thin membranes with fewer defects, more hydrophilicity, stability and lower mass transfer resistance (Halakoo and Feng, 2020).

LbL technique has been feasibly used to form the PA selective layer of TFC RO membranes as it demonstrates several advantages compared with the conventional interfacial polymerization technique in terms of the control over the thickness and composition of the thin film in molecular level. However, when thin film nanocomposite (TFN) membrane is concerned, the direct incorporation of the nanomaterials in one of the polyelectrolytes or both during LbL assembly may be destructive. The presence of nanomaterial may induce the formation of defective selective layer due to the interrupted interaction between the anionic and cationic polyelectrolytes (Long *et al.*, 2018). The unique features of the nanomaterials to tackle fouling issue based on their antimicrobial properties and ability to form hydration layer could not be maximized when these nanomaterials were embedded within the LbL assembled polyelectrolyte thin film layer (Wang *et al.*, 2017b). In order to address these limitations, the LbL deposition of nanomaterials on the as-fabricated PA TFC membrane has been attempted in this study. The surface of PA TFC membrane allows the maximum nanomaterial loading where more accessible nanomaterial active sites are available to alter the surface properties (Liu *et al.*, 2013; Goh and Ismail, 2014).

In recent years, TiO_2 nanomaterial has received great attention as it offers many unique properties such as excellent antimicrobial properties, high hydrophilicity (Jhaveri and Murthy, 2016), low toxicity (Safarpour, Khataee and Vatanpour, 2015), high chemical stability and photocatalytic properties (Munafò *et al.*, 2014). However, the usage of TiO_2 nanoparticles in membrane is often limited by the insufficient surface hydrophilicity and high surface roughness (Nakata and Fujishima, 2012). By contrast, two-dimensional (2D) nanostructure presents the new potential of TiO_2 nanomaterial to render more fascinating properties such as ultrathin thickness, smooth surface, large surface area, and offers higher surface hydrophilicity owing to the presence of a large quantity hydroxyl functional group compared to its 1-dimensional counterpart (Sasaki *et al.*, 1996; Sáringer, Rouster and Szilágyi, 2019). These properties could help in addressing low permselectivity and organic fouling by improving both hydrophilicity and surface roughness of membrane.

In this study, the hydrophilic titania in nanosheets (TNS) structure through LbL approach on the pre-formed TFC membranes was fabricated. Two types of TNS, i.e. positively and negatively charged TNS were prepared and deposited alternatively to form the bilayer. Various characterizations were conducted to study and correlate their physicochemical properties to the separation performances in RO. The resultant TFN performance was evaluated through PW desalination using low to high concentration of PW feed stream. Leaching and long-term study were performed to evaluate the stability of the bilayer deposited on the PA layer. Further, high concentration of saline oily and oily saline water was conducted to evaluate the excellent of membrane performance.

1.3 Research Objectives

As based on the preceding challenges and issues, the objectives of the research were set out:

(a) To evaluate the separation performance of TNS bilayer number and loading effect of negatively and positively charged TNS on PA layer through LbL assembly approach with oily saline water

(b) To investigate the stability of separation performance of TFN membrane with the best number of bilayer and loading onto PA layer via LbL technique.

(c) To study the antifouling property of the TFN membrane with the optimal bilayer using various feed concentration of saline oily water and oily saline water.

1.4 Scope of study

In order to achieve the objective that mentioned previously, the following scopes were drawn:

For Objective 1:

(a) Synthesizing of both pTNS and nTNS through solid-state calcination, acid ion exchange and exfoliation method.
(b) Characterizing and studying the structural properties of both TNS by using atomic force microscopy (AFM), Fourier transform infrared spectroscopy (FTIR), zeta potential, and high-resolution transmission electron microscopy (HRTEM) and x-ray diffraction spectroscopy (XRD) for height profile, functional group determination, surface charge and morphology and crystallinity, respectively.

(c) Evaluating the dispersibility of nanoparticles in aqueous solution using ultraviolet–visible (Uv-vis) spectrophotometer and Tyndall effect.

(d) Fabricating PSf support layer via solvent-nonsolvent phase inversion technique.

(e) Determining molecular weight cut-off (MWCO) of PSf.

(f) Forming PA layer with IP technique of using *m*-phenylenediamine (MPD) in aqueous solution and acryl chloride monomer (TMC) in hexane on top of the support layer.

(g) Depositing positively charged TNS and negatively charged TNS with the same loading of 0.05 mg/ml with different bilayer number from 0 to 4 onto PA layer via LbL technique to obtain stacked nanosheets structure where the number of pTNS/nTNS bilayer is controlled by the deposition cycle.

(h) Characterizing the fabricated membranes by using FTIR, contact angle goniometer, zeta potential, AFM and field emission scanning electron microscopy (FESEM), Energy Dispersive X-ray spectroscopy (EDX).

(i) Evaluating performances of the water permeability and rejection on six membranes including commercial TFC and self-fabricated membranes conducted by RO testing under 15 bar pressure by using feed of salt (NaCl aqueous solution; 2 mg/ml).

(j) Characterizing for the best bilayer number of TFN membrane by using X-ray photoelectron spectroscopy (XPS) and HRTEM.

(k) Evaluating performances of the water permeability and rejection on five selected membranes conducted by RO testing under 15 bar pressure by using feed of synthetic PW (NaCl aqueous solution; 2 mg/ml and crude oil; 1 mg/ml).

(l) Characterizing feed and permeate by using conductivity meter and Uv-vis spectrophotometer.

(m) Manipulating TNS loading in the range of 0.01-0.1 mg/ml of in aqueous solution with the optimal number of bilayer onto PA layer via LbL technique.

(n) Characterizing the fabricated membranes by using contact angle goniometer, zeta potential, AFM and FESEM.

(o) Evaluating performances of the water permeability and rejection on three type of membranes with different TNS loading conducted by RO testing under 15 bar pressure by using feed of synthetic PW (NaCl aqueous solution; 2 mg/ml and crude oil; 1 mg/ml.

For Objective 2:

(a) Evaluating long-term stability test on the TFN membrane with the TFC membrane and leaching of Ti.
(b) Characterizing the permeate of membrane separation by using inductively coupled plasma mass spectrometry (ICP-MS) analyser.

For Objective 3:

(a) Evaluating anti-fouling test with low to high concentration of saline oily water (2000 ppm, 5000 ppm and 10,000 ppm) and oily saline water (1000 ppm, 5000 ppm and 10,000 ppm) in saline oily water by comparing rate of oil rejection, salt rejection and water flux over periods of 960 min for repeated four cycles.

(b) Characterizing the feed and permeate water and tested membranes by using zeta sizer and SEM, respectively.

1.5 Significance of Study

This work presents the first attempt to perform a correlation study between LbL assembled TNS on TFC membrane with PW foulants. Surface coating through LbL assembly is an attractive post-coating method for preparing an ultrathin layer of composite membrane with tailored physicochemical. Through LbL assembly, the thickness of the thin film can be tailored and controlled. Moreover, the new features of TiO_2 in nanosheets dimensional could stimulate great interest in the use of LbL assembly technique for TFC membrane modification. Thus, exhibit much smoother and thinner surface which can induce a great flowing of water and increase rejection of salt and oil. This study also demonstrates the potential of LbL to be used for industry scaled membrane surface modification as it involves simple and straightforward approach of membrane modification. The technique promises the greener and very sustainable engineering membrane especially in oil and gas industry.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Ahmad, N. A**., Goh, P. S., Yogarathinam, L. T., Zulhairun, A. K. and Ismail, A. F. (2020) 'Current advances in membrane technologies for produced water desalination', *Desalination*, 493, pp. 114643. **(Q1, IF:9.501)**

2. **Ahmad, N. A**., Goh, P. S., Wong, K. C., Zulhairun, A. K. and Ismail, A. F. (2020) 'Enhancing desalination performance of thin film composite membrane through layer by layer assembly of oppositely charged titania nanosheet', *Desalination*, 476, pp. 114167. **(Q1, IF:9.501)**

3. **Ahmad, N. A**., Goh, P. S., Zulhairun, A. K. and Ismail, A. F. (2020) 'Antifouling property of oppositely charged titania nanosheet assembled on thin film composite reverse osmosis membrane for highly concentrated oily saline water treatment', *Membranes*, 10(9), pp. 1–19. **(Q1, IF:4.106)**

4. **Ahmad, N. A**., Goh, P. S., Karim, Z. A. and Ismail, A. F. (2018) 'Thin film composite membrane for oily waste water treatment: Recent advances and challenges', *Membranes*, 8(4), pp. 86. **(Q1, IF:4.106)**