THIN FILM NANOCOMPOSITE FORWARD OSMOSIS MEMBRANE INCORPORATED WITH BIMETALLIC OXIDE FOR HEAVY METAL REMOVAL

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ABSTRACT

Heavy metal contamination which has threatened human health is a critical environmental issue that caused by the uncontrolled discharge of heavy metal. Membrane technology has been justified as one of the favourable options in wastewater treatment for contaminants removal due to its high rejection to produce high quality of treated water. Recently, forward osmosis (FO) has emerged as an attractive alternative of conventional approaches such as adsorption and reverse osmosis (RO) for heavy metal removal. The advantages of FO process include lower energy consumption and less fouling tendency. During the separation processes, FO membrane ensures the water pass through the membrane while all the dissolved metal ions could be separated and filtered by the TFN FO membrane. Despite the advantages of FO, the major issue of FO membrane is to confront with interfacial concentration polarization (ICP) which causes the deterioration of flux over time. The reduction of critical ICP issue and increased water flux through the modification of TFC membrane by incorporating functional nanomaterials into the substrate layer of the FO TFC membrane has been proven as a feasible strategy to heighten the performance of FO membrane. In this study, thin film nanocomposite (TFN) FO membranes incorporated with titania nanotubes and magnetite oxide (TNT–Fe_3O_4) hybrid has been successfully fabricated. The TNT–Fe_3O_4 hybrid nanoparticles was synthesized through hydrothermal and coprecipitation method. In the first stage of this study, different types of nanoparticles i.e. TNT, Fe_3O_4, TNT–Fe_3O_4 were embedded into polysulfone (PSf) substrate. The performance of the TFN FO membranes were evaluated for heavy metal removal using the FO system at active layer facing feed solution (AL–FS) mode. Using the membrane incorporated 0.5 wt% of TNT–Fe_3O_4 hybrid nanoparticles, the FO water flux of 2.84 L/m^2.h and 2.54 L/m^2.h and the rejection of 98.06% and 88.37% were achieved for Cd^{2+} and Pb^{2+} removal, respectively. In the second stage, the effect of different loading of TNT–Fe_3O_4 embedded into PSf substrate was investigated. The loading of TNT–Fe_3O_4 hybrid plays a role in improving the FO water flux and rejection, particularly with the increasing loading of the nanofiller. The TFN 0.5 membrane exhibited good water permeability (A) with 3.60 L/m^2.h.bar, salt permeability (B) with 2.33 x10^{-8} m/s and 95.94% of salt rejection. TFN 0.5 achieved the small structural parameters with 1.45 mm and 1.60 mm in Cd^{2+} and Pb^{2+} removal, respectively. In addition, the optimized membrane exhibited high flux recovery and rejection which indicated a good quality of the TFN FO membrane. As a conclusion, modification of TFN FO membrane by incorporating TNT–Fe_3O_4 hybrid as nanofillers in the substrate layer is a promising approach to improve membrane permeability and selectivity.

ABSTRAK

Pencemaran logam berat yang mengancam kesihatan manusisa adalah isu kritikal dalam alam sekitar yang disebabkan oleh pelepasan logam berat yang tidak terkawal. Teknologi membran telah diiktirafkan sebagai salah satu pilihan yang menggalakkan dalam rawatan air sisa untuk penyingkiran bahan pencemaran akibat penolakannya yang tinggi untuk menghasilkan air terawat yang berkualiti tinggi. Dalam dekad kebelakangan ini, osmosis hadapan (FO) telah muncul sebagai pendekatan konvesional yang menarik seperti penjerapan dan osmosis berbalik (RO) untuk penyingkiran logam berat. Kelebihan proses FO termasuk penggunaan tenaga yang lebih rendah dan kekurangan kecenderungan terhadap kotoran membran. Semasa proses pemisahan, membran FO memastikan air melalui membran sementara semua ion logam yang terlarut boleh dipisahkan dan ditapis oleh membran TFN FO. Selain daripada kelebihan FO, kepekatan polarisasi dalaman (ICP) adalah isu utama untuk membran FO yang menyebabkan kemerosotan aliran air dari semasa ke semasa. Pengurangan isu kritikal ICP dan peningkatan aliran air (Jv) melalui pengubahsuaian membran TFC dengan menggabungkan bahan nanopartikel ke dalam lapisan substrat membran TFC FO telah terbukti sebagai strategi yang boleh dilaksanakan untuk meningkatkan prestasi membran FO. Dalam kajian ini, membran osmosis hadapan nanokomposit saput tipis (TFN) yang ditubuhkan dengan titania tiub nano dan magnetit oksida (TNT–Fe_3O_4) telah berjaya dihasilkan. Nanopartikel hibrid TNT–Fe_3O_4 disintesis melalui kaedah hidrotermal dan gabung pemendakan. Pada peringkat pertama kajian ini, pelbagai jenis nanopartikel iaitu TNT, Fe_3O_4, TNT–Fe_3O_4 telah dimasukkan ke dalam substrat polisulfona (PSf). Prestasi membran TFN FO sedang mengkaji penyingkiran logam berat menggunakan sistem FO pada mod AL–FS. Jv FO telah mencapai pada 2.84 L/m^2.h dan 2.54 L/m^2.h dan penolakannya mencapai 98.06% dan 88.37% dalam penyingkiran Cd^{2+} dan Pb^{2+} masing-masing apabila membran mengandungi 0.5% hibrid TNT–Fe_3O_4. Pada peringkat kedua, kesan pemuatan berbeza TNT–Fe_3O_4 yang dimasukkan ke dalam substrat PSf telah diselidiki. Pemuatan hibrid TNT–Fe_3O_4 memainkan peningkatan yang ketara dalam Jv FO dan penolakan ketika kandungan meningkat. Pada peringkat akhir, semua membran TFN FO disediakan telah menguji dalam sistem RO. Membran TFN 0.5 berbanding dengan membran kawalan menunjukkan kebolehtelapan air yang baik (A) dengan 3.60 L/m^2.h.bar, ketelapan garam (B) dengan 2.33 x10^{-8} m/s dan 95.94% penolakan garam. TFN 0.5 mengekalkan parameter struktur kecil dengan 1.45 mm dan 1.60 mm dalam penyingkiran Cd^{2+} dan Pb^{2+}. Di samping itu, membran yang dioptimumkan menunjukkan pemulihan aliran air dan penolakan yang tinggi yang menunjukkan kualiti membran TFN FO yang baik. Kesimpulannya, pengubahsuaian membran TFN FO dengan menggabungkan hibrid TNT–Fe_3O_4 sebagai nanofiller dalam lapisan substrat merupakan pendekatan yang menjanjikan untuk peningkatkan kebolehtelapan dan pemilihan membran.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
AL-FS	-	Active layer facing feed solution
DS	-	Draw solution
EDX	-	Energy dispersive spectroscopy
Fe_3O_4	-	Magnetite oxide
FESEM	-	Field emission scanning electronic microscope
FO	-	Forward osmosis
FS	-	Feed solution
HRTEM	-	High resolution transmission electron microscopy
ICP	-	Internal concentration polarization
IP	-	Interfacial polymerization
MPD	-	1,3-phenylendiamine
NF	-	Nanofiltration
PA	-	Particle Swarm Optimization
PSf	-	Polysulfone
PVP	-	Polyvinylpyrolidone
RO	-	Reverse osmosis
TFC	-	Thin film composite
TFN	-	Thin film nanocomposite
TMC	-	Trimesoyl chloride
TNT	-	Titania nanotubes
XRD	-	X-Ray diffractometer analysis

LIST OF SYMBOLS

A	-	Water permeability coefficient ($L/m^2.h$)
B	-	Salt permeability coefficient (m/s)
Js	-	Reverse salt flux ($g/m^2.h$)
Jv	-	Water flux ($L/m^2.h$)
M	-	Molarity (M)
R	-	Solute rejection (%)
S	-	Membrane structural parameter (mm)
T	-	Temperature (°C)
ΔP	-	Hydraulic pressure difference (bar)
$\Delta\pi$	-	Osmotic pressure difference (Pa)
wt%	-	Percentage of weight over volume

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Heavy metal contamination is a critical environmental issue that caused by the uncontrolled discharge of heavy metal which has threatened human health. The electronics, electroplating, machinery manufacturing, and metallurgy industries have produced an extensive amount of wastewater containing heavy metal which are eventually discharged into water bodies. Metal ions tend to penetrate inside human body through accumulation pathway and cause severe health effects and body dysfunction because its unable to be metabolized by human body or decompose easily (Cui et al., 2014). Compared to other toxic substances, heavy metals are stable and cannot be degraded naturally over long period of time. Therefore, the metal ions tend to accumulate in living organisms and bring tremendous ecological and physiological exposure. Thus, heavy metal removal from wastewater becomes a progressively important global issue (Zhu et al., 2015). Salam et al. (2019) reported that the geostatistical distribution and contamination status of heavy metals in the sediment of Perak river. Their study showed the range of heavy metals concentration in downstream areas were Pb (27.6–60.76 μg/g), Zn (49.04–160.5 μg/g), Cd (2.77–4.02 μg/g) and Cu (9.82–59.99 μg/g).

Various strategies for efficient heavy metal removal from water have been widely studied to reduce the negative impacts of heavy metal contaminations (Zhao & Liu, 2018). Increasing demand of pure water with low concentration of heavy metals makes it obligatory to effectively eliminate toxic heavy metals from industrial runoffs prior to their release into the ecosystem. Different conventional techniques are accessible for the decontamination of wastewater such as adsorption, ion exchange, chemical precipitation and solvent extraction (Wadhawan et al., 2020). Adsorption using suitable adsorbents is one of the most efficient, effective, fast, simple and economical method for the remove of metal ions from water and wastewater sources (Vojoudi et al., 2017). Chemical precipitation is one of the most widely used techniques for heavy metal removal from inorganic effluent in industry due to its simple operation (Gunatilake, 2015). Ion exchange process is another widely used approach in removing heavy metal from wastewater with ion exchange resin which has the specific uptake ability to exchange its cations with metals in wastewater, either synthetic or natural solid resin (Zhao et al., 2016a). Solvent extraction has been used for wastewater purification with using ionic liquids have shown good performance as an extractant phase in the separation of heavy metal ions (Platzer et al., 2017).

In the last decades, the development of membrane technology has been justified as one of the favourable options in wastewater treatment for contaminants removal due to its excellent removal efficiency. Membrane technology has been justified as a practicable option in wastewater treatment owing to its low fabrication cost and high rejection to contaminants such as organic compounds (Cui et al., 2014). Abdullah et al. (2019b) reported ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO) and forward osmosis (FO) are the membrane technologies that commonly used for treating heavy metal contaminated water. In essence, membrane filtration is a pressure driven separation process for heavy metals while it can be enhanced by treating with the membrane (Zhao et al., 2016b). The comparison of the NF and RO performance for treating industrial wastewater containing metal effluents at different operating pressure was reported by Liu et al. (2008a). The results showed that although RO could achieve an excellent metal rejection rate, however its water flux remained very low compared to NF. Nevertheless, both membrane processes require high energy operation and has high fouling tendency, which resulted in reduced productivity and extra operational cost (Abdullah et al., 2019b). The advantages of membrane technology mentioned above are high separation efficiency and excellent heavy metals rejection. However, the limitations in terms of high energy consumption, low water permeability and membrane fouling also hinder the sustainable implementation of membrane technology.

In recent years, FO has attracted a very great interest among researchers for heavy metals removal as it requires a very low energy, low fouling rate, satisfactory pure water permeation and high solute rejection (Cui et al., 2014). Hence, this provides the FO technique a hidden prospect for heavy metal removal in water and wastewater treatment (Liu et al., 2017a). Principally, FO is a membrane process driven by natural osmotic pressure created when draw solution and feed solution with different concentrations separated by a semipermeable membrane (Coday et al., 2014). FO membranes is nearly similar to NF and RO which comprised of substrate and selective thin film layer except does not utilized hydraulic pressure, FO can be potentially for this particular application (Abdullah, Tajuddin, & Yusof, 2019a). Bao et al. (2019) fabricated polyamidoamine (PAMAM) dendrimer grafted TFC FO membrane to achieve high NH_4^+ rejection and antifouling capacity in treating domestic wastewater. FO membranes are capable of rejecting most organics and phosphate ions by virtue of the size sieving effect (Chekli et al., 2016). On the other hand, Zhao et al. (2018) investigated the removal of Ni(II) from high salinity wastewater. The removal was carried out by comparing the performance of commercial cellulose triacetate (CTA) FO membrane and polyamide based TFC membrane. Nevertheless, internal concentration polarization (ICP) still remains as a major drawback for FO to be an applicable method for heavy metals removal. Currently, the ideal performance of FO membrane remains unachievable due to the inherent issues related to osmotically driven membranes such as high ICP which restricts the membrane from delivering optimum performance, particularly in terms of water flux and solutes rejection.

The recent development of FO membranes are focused on TFC membrane fabrication and their modification. The substrate structure and morphology significantly influence the degree of ICP within the porous layer; whereas the active layer controls the extent of solute and water fluxes across the membrane (Akther et al., 2019). Although PA-based TFC FO membranes have better performance compared to the cellulose-based membranes, the overall FO performance in terms of ICP, fouling resistance and chlorine tolerance is still an issue (Akther et al., 2015). On the other hand, substrate modification using inorganic nanofiller is one of the most common approaches to address ICP issue (Goh et al., 2019). The presence of nanomaterials within the substrate layer can alter the hydrophilicity and morphology, hence altering the water or solute transport behaviour during the FO process.

Since the first introduction of thin film nanocomposite (TFN), different nanomaterials such as titanium dioxide (TiO_2), silica dioxide (SiO_2), carbon nanotubes (CNTs) and graphene oxide (GO) have been incorporated into TFC FO membranes. The incorporation of GO into TFC substrate enhanced the membrane pore diameter, porosity and hydrophilicity which markedly increased the water permeability and antifouling properties (Sirinupong et al., 2018; Zinadini et al., 2014). The incorporation TiO_2 nanoparticles in the PSf substrate of PA TFC membrane using direct blending to control ICP in the substrate during FO operation (Emadzadeh, Lau, & Ismail, 2013). Magnetite oxide (Fe_3O_4) nanoparticles is used as inorganic nanofiller additive in membrane fabrication due to its numerous advantages, such as low toxicity, good biocompatibility, high surface area and chemical stability. Next, Fe_3O_4 nanoparticles also assisted for decreasing ICP of TFN FO membrane (Darabi et al., 2017). Titania nanotubes (TNTs) has been explored as nanofillers for fabricating TFN FO membranes due to its tubular form provides additional channel for water transport across the membranes. The advantages of TNT owing to hydrophilic properties, good stability, large pore sizes and surface area (Akther et al., 2019).

1.2 Problem Statement

In recent years, the utilization of heavy metal compounds has exponentially increased in various industrial processes. As such, it has also become a critical environmental issue due to heavy metal contamination of water source. Although heavy metal ions could be eliminated from wastewater by several conventional methods including adsorption, chemical precipitation and ion exchange (Atkovska et al., 2018), the major issues with these technologies are their effectiveness, energy consumption and long term sustainability. FO has gained interest as a membrane-based water treatment alternative owing to its low membrane fouling tendency and energy consumption, compared to other membrane techniques (Shibuya et al., 2018). However, the common issue encountered by most TFC FO membranes is the ICP. The diffusion of reverse salt and the intrinsically porous structure of the substrate layer induce ICP. This results in the decrease in the effective driving force and eventually the water flux declines sharply. The most common and effective strategy is by preparing TFN FO membrane which involves the incorporation of inorganic nanoparticles to alter the membrane substrates in order to reduce ICP phenomenon of TFC FO membranes (Zhang et al., 2018a). The nanoparticles provide their essential properties to address the ICP issues while maintaining the membrane performance (Lakhotia, Mukhopadhyay, & Kumari, 2019).

Currently, the inorganic nanomaterials that have been most widely used for FO membrane modification include zeolite, GO, CNTs, silica and TiO_2 (Akther et al., 2019). These nanomaterials are introduced into polymeric membranes matrices to improve the membrane hydrophilicity and reduce the surface roughness (Daraei et al., 2013). Nevertheless, most of the efforts in FO membrane modification were focused on using single nanomaterial to bring about the desired properties for desalination and wastewater treatment. For instance, TNT has been used to increase the hydrophilicity of the FO membranes in order to improve the flux and reduce fouling tendency. Darabi et al. (2017) were the first added Fe_3O_4 nanoparticles in polyehtersulfone (PES) substrate to mitigate ICP. The results revealed that the porosity and the hydrophilicity of the PES substrate were improved after addition of Fe_3O_4 leading to reducing in structural parameter (S) and water flux enhancement. As both hydrophilicity and ICP are equally important factors to be considered for a high-performance FO membrane, it is necessary to fabricate a membrane that can simultaneously address both issues. It is expected that the synergistic effect of TNT and Fe_3O_4 nanomaterials simultaneously address ICP and increase hydrophilicity. Hence, this study focused on the synthesis hybrid nanomaterials in order to mitigate ICP issue for water flux improvement as well as achieve high rejection of heavy metal removal in TFN FO membrane.

Based on recent open literature, most of the TFN membranes were prepared for desalination in RO or FO process. The TFN FO membranes that are particularly designed for heavy metal removal is still limited. Despite some works that showed the potential of using FO for heavy metal removal, these works were largely based on commercial membranes and their modifications (Vital et al., 2018; Zhao et al., 2016c). As the properties of FO membrane is one of the most crucial factor in determining the efficiency of the process, most emphasis should also be placed on the design and modification of membrane to further enhance the heavy metal removal efficiency.

Hence, this study is aimed to fabricate a TFN FO membrane which is incorporated with bimetallic oxide hybrid nanoparticles. TNT–Fe_3O_4 hybrid nanoparticles were synthesized via hydrothermal and coprecipitation method. The physio-chemical properties of TNT–Fe_3O_4 hybrid nanoparticles were investigated. Then, the TNT–Fe_3O_4 hybrid nanoparticles was embedded into polymer dope solution of polysulfone (PSf) substrate at different loading. The TNT–Fe_3O_4 incorporated PSf was formed by inversion phase and the polyamide selective layer was formed through interfacial polymerization. The physio-chemical properties of the fabricated membrane were evaluated in terms of surface roughness, water permeability and hydrophilicity. Finally, the separation and filtration performance of the TFN FO membranes were evaluated for heavy metals removal in terms of water permeability and separation behaviour.

1.3 Objectives of Study

The main aims of this research to fabricate a TFN FO membrane which consist of bimetallic oxide nanoparticles incorporated into the PSf substrate in order to form a TFN FO membrane for heavy metal removal. In order to solve the above-mentioned problems, the following objectives of have been outlined:

i. To investigate the physicochemical properties of TNT–Fe_3O_4 hybrid nanoparticle synthesized through hydrothermal and co-precipitation method.
ii. To investigate the physicochemical properties of TFN FO membranes incorporated with the TNT, Fe_3O_4 and different loading of TNT–Fe_3O_4 hybrid nanoparticle into substrate.
iii. To study the separation performance of TFN FO membrane for heavy metals removal in terms of flux, rejection and regeneration behaviour.

1.4 Scope of Study

In order to achieve the main objectives of this study, the scopes of the study are identified as below:

i. Synthesizing the hybrid bimetallic oxide nanoparticles by hydrothermal and coprecipitation method using titanium dioxide (TiO_2) and ferrous sulphate ($FeCl_2 \cdot 4H_2O$) and ferric chloride ($FeCl_3 \cdot 6H_2O$) at molar ratio 1:2. The hybrid bimetallic oxide nanoparticles TNT and Fe_3O_4 was fixed at weight ratio of 1:1.

ii. Conducting the morphological and physicochemical characterization of TNT–Fe_3O_4 hybrid nanoparticles using high resolution transmission electron microscope (HRTEM), field scanning electron microscope (FESEM), X-ray diffraction (XRD) and zeta potential.

iii. Embedding TNT–Fe_3O_4 hybrid nanoparticles into the PSf substrate dope. The loading of TNT–Fe_3O_4 hybrid nanoparticles was varied from 0.1 wt% – 0.7 wt%. The dope formulation was maintained constant at 16.5 wt% polysulfone (PSf), 0.5 wt% polyvinylpyrrolidone (PVP) and 83 wt% n-methyl-pyrrolidone (NMP).

iv. Fabricating PSf substrate via phase inversion and polyamide layer via interfacial polymerization using 2.0 wt% m-phenylenediamine (MPD) and 0.1 wt% of trimesoyl chloride (TMC) as monomers

v. Conducting the morphological, surface roughness, hydrophilicity and surface charge characterization of the TFN FO membrane by scanning microscope (SEM), atomic force microscope (AFM), water contact angle, porosity and zeta potential, respectively.

vi. Conducting the cross-flow RO experiment at 1 bar hydraulic pressure.

vii. Evaluating the intrinsic properties of TFN FO membrane in terms of water permeability, salt permeability and salt rejection in RO system.

viii. Preparing a 10 mg/L heavy metal feed solution from each $Cd(NO_3)_2$ and $Pb(NO_3)_2$ powder.

ix. Conducting the FO experiment in active layer facing feed solution (AL-FS) orientation with 10 mg/L of $Cd(NO_3)_2$ as feed solution and 1 M of NaCl as draw solution.

x. Evaluating the filtration performance of TFN FO membrane in terms of water flux, reverse solute flux and structure parameter in FO system.

1.5 Significance of Study

The development of a TFN FO membrane has offered significant contribution in water treatment for heavy metals removal due to their multifunctional properties of separation and filtration simultaneously with low energy consumption. Due to the variety of heavy metal pollutants present in wastewater from industry, the suitable nanoparticles must be selected for more efficient heavy metals separation and filtration. Thus, the fabricated membrane incorporated with bifunctional nanomaterials which is TNT–Fe_3O_4 hybrid nanoparticles that that can simultaneously address issues related to ICP and increase water flux. As this is the first attempt of modifying FO membrane for heavy metal removal through FO process, the findings from the correlation studies between the membrane properties and heavy metal removal performance will serve as the guideline for future related studies. With the promising features shown, the TFN FO membrane will be a promising heavy metals separation for water treatment.

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