IRON OXIDE MODIFIED POLYETHERSULFONE HOLLOW FIBER MEMBRANES WITH IMPROVED HYDROPHILICITY FOR WATER TREATMENT

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To my family and friends.

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ABSTRACT

Rapid industrialization has led to deterioration of water quality due to the increase in improper discharge of wastewater into receiving water body. Over the years, membrane technology has emerged as a potential alternative to treat various types of water and wastewater. However, fouling and flux declination are still the major hindrances for the membrane application. To address this issue, the aim of this work was to develop a hollow fiber membrane with improved surface properties via incorporation of hydrophilic nanoparticles for water application. The objective of this work is to investigate the impacts of particle size of iron oxide (Fe_3O_4) and the effects of different surface-functionalized Fe_3O_4 nanoparticles on the properties of polyethersulfone (PES) hollow fiber membranes for ultrafiltration process. All of the membranes were synthesized via a dry-jet wet spinning process followed by a series of instrumental characterization before proceeding to filtration performance assessment. The results showed that the addition of smaller Fe_3O_4 particles (50 nm) into PES dope solution could produce a membrane with better hydrophilicity (contact angle: 75.77°) and consequently better pure water flux (PWF) (102.74 L/m^2.h.bar) compared to the pristine PES membrane (82.55 L/m^2.h.bar) and membrane incorporated with larger Fe_3O_4 particles of 5 µm (91.55 L/m^2.h.bar). This is due to better particle dispersion in the PES matrix that subsequently enhances the membrane permeability. In addition, the membrane modified by 50-nm Fe_3O_4 also displayed good filtration performance by rejecting 80.43% bovine serum albumin (BSA) from aqueous solution. Surface coating of 50-nm Fe_3O_4 nanoparticles using polydopamine (PDA) could further improve its dispersion and stability in dope solution, leading to improved membrane performance. Compared to the membrane incorporated with neat Fe_3O_4, the addition of PDA-coated Fe_3O_4 into PES membrane showed 17.58% improvement in water flux and 6.08% higher BSA rejection, reaching 121.19 L/m^2.h.bar and 85.32%, respectively. This can be attributed to the improvement in terms of membrane hydrophilicity and porosity. The incorporation of dual functionalized Fe_3O_4 into PES membrane using amine were found to greatly promote membrane permeability compared with using sulfonic acid due to enhanced synergistic interaction between Fe_3O_4/PDA-amine and PES polymer. The PES/Fe_3O_4/PDA-amine membrane recorded the highest PWF of 137.15 L/m^2.h.bar and BSA rejection of 92.16%. This membrane also achieved excellent chemical oxygen demand (95.17%) and color reduction (89.86%) when tested using river water sample. In conclusion, the developed PES/Fe_3O_4/PDA-amine membrane showed an excellent separation performance and antifouling property for water treatment, overcoming the drawbacks of PES membrane.

ABSTRAK

Perkembangan perindustrian yang pesat menyebabkan kemerosotan kualiti air disebabkan oleh peningkatan pengendalian pembuangan air sisa yang tidak betul ke sumber air. Dalam tempoh beberapa tahun, teknologi membran telah muncul sebagai alternatif yang berpotensi untuk merawat pelbagai jenis air dan air sisa. Walau bagaimanapun, pengotoran dan pengurangan fluks masih menjadi penghalang utama bagi aplikasi membran dalam bidang ini. Untuk mengatasi masalah ini, tujuan penyelidikan ini adalah untuk membangunkan membran gentian berongga yang mempunyai sifat permukaan yang lebih baik melalui penggabungan nanozarah yang hidrofilik untuk aplikasi air. Objektif kerja ini adalah untuk mengkaji impak saiz zarah ferum oksida (Fe_3O_4) dan kesan nanozarah Fe_3O_4 yang berfungsi dengan permukaan yang berbeza pada sifat membran serat berongga polietersulfon (PES) untuk proses ultraturasan. Semua membran disintesis melalui proses putaran jet basah-kering diikuti dengan siri analisa instrumental sebelum meneruskan penilaian prestasi penapisan. Hasil kajian menunjukkan bahawa penambahan zarah Fe_3O_4 yang lebih kecil (50 nm) ke dalam larutan polimer PES dapat menghasilkan membran yang lebih hidrofilik (sudut sentuh: 75.77°) dan menyebabkan fluks air tulen meningkat (PWF) (102.74 $L/m^2.h.bar$) berbanding dengan membran PES yang tulen (82.55 $L/m^2.h.bar$) dan membran yang digabungkan dengan zarah Fe_3O_4 yang lebih besar, 5 µm (91.55 $L/m^2.h.bar$). Ini disebabkan oleh penyebaran zarah yang lebih baik dalam matriks PES yang seterusnya meningkatkan kebolehtelapan membran. Selain itu, membran yang diubah suai oleh Fe_3O_4 50-nm juga menunjukkan prestasi penurasan yang baik dengan penolakan 80.43% albumin dari serum lembu (BSA) dari larutan berair. Lapisan permukaan nanozarah Fe_3O_4 50-nm yang diubah menggunakan salutan polidopamina (PDA) dapat meningkatkan penyebaran dan kestabilannya dalam larutan polimer, yang membawa kepada peningkatan prestasi membran. Berbanding dengan membran yang digabungkan dengan Fe_3O_4 tulen, penambahan Fe_3O_4 bersalut PDA ke dalam membran PES menunjukkan peningkatan fluks air sebanyak 17.58% dan penolakan BSA 6.08% lebih tinggi, dimana masing-masing mencapai 121.19 $L/m^2.h.bar$ dan 85.32%. Ini boleh dikaitkan dengan peningkatan dari segi kehidrofilikan membran dan keliangan. Penggabungan Fe_3O_4 fungsi duaan ke dalam membran PES menggunakan amina didapati dapat meningkatkan kebolehtelapan membran berbanding dengan penggunaan asid sulfonik kerana interaksi kesekerjaan yang baik antara Fe_3O_4/PDA-amina dan polimer PES. Membran PES/Fe_3O_4/PDA-amina mencatatkan PWF tertinggi iaitu 137.15 $L/m^2.h.bar$ dan penolakan BSA sebanyak 92.16%. Membran ini juga mencapai keperluan oksigen kimia (95.17%) dan pengurangan warna (89.86%) ketika diuji dengan sampel air sungai. Kesimpulannya, membran PES/Fe_3O_4/PDA-amina yang dibangunkan menunjukkan prestasi pemisahan dan sifat anti-kotoran untuk rawatan air yang sangat baik, sekaligus dapat mengatasi kekurangan membran PES.

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LIST OF ABBREVIATIONS

Al_2O_3	-	Aluminium oxide
APTES	-	(3-aminopropyl) triethoxysilane
AT-POME	-	Aerobically treated palm oil mill effluent
BET	-	Brunauer-Emmett-Teller surface area analysis
BSA	-	Bovine serum albumin
CA	-	Cellulose acetate
CH_2Cl_2	-	Dichloromethane
$ClSO_3H$	-	Chlorosulfonic acid
CNTs	-	Carbon nanotubes
DA	-	Dopamine hydrochloride
Da	-	Dalton
DMAc	-	*N,N*-dimethylacetamide
DMF	-	*N,N*-dimethylformamide
EDX	-	Energy dispersive x-ray
Fe	-	Iron
Fe^{2+}	-	Ferrous ion
Fe^{3+}	-	Ferric ion
Fe_3O_4	-	Iron oxide particles
Fe_3O_4/PDA	-	Polydopamine-coated Fe_3O_4 particles
Fe_3O_4/PDA-Amine	-	Amine-modified polydopamine-coated Fe_3O_4 particles
Fe_3O_4/PDA-Sulf	-	Sulfonic acid-modified polydopamine-coated Fe_3O_4 particles
FESEM	-	Field emission scanning electron microscopy
FTIR	-	Fourier-transform infrared
FRR	-	Flux recovery rate
GO	-	Graphene oxide
KBr	-	Potassium bromide
MF	-	Microfiltration
$MgSO_4$	-	Magnesium sulfate

MWCNT	-	Multi-walled carbon nanotubes
Na_2SO_4	-	Sodium sulfate
NF	-	Nanofiltration
NMP	-	*N*-methylpyrrolidone
NIPS	-	Non-solvent induced separation
O	-	Oxygen
O^{2-}	-	Oxygen ion
OCMCS	-	O-carboxymethyl chitosan
PAA	-	Poly (acrylic acid)
PAN	-	Polyacrylonitrile
PANI	-	Polyaniline
PDA	-	Polydopamine
PEG	-	Polyethylene glycol
PEO	-	Polyethylene oxide
PES	-	Polyethersulfone
PEES	-	Poly (ether ether) sulfone
PVA	-	Polyvinyl alcohol
PSf	-	Polysulfone
PVC	-	Polyvinylchloride
PVDF	-	Polyvinylidene fluoride
PVP	-	Polyvinylpyrrolidone
PWF	-	Pure water flux
PWP	-	Pure water permeation flux
RO	-	Reverse osmosis
SiO_2	-	Silica dioxide
TEM	-	Transmission electron microscopy
TGA	-	Thermal gravimetric analysis
TiO_2	-	Titanium dioxide
TIPS	-	Temperature induced phase separation
UF	-	Ultrafiltration
XRD	-	X-ray diffractometry
ZnO	-	Zinc oxide

LIST OF SYMBOLS

A	-	Effective area of membrane (m^2)
C_p	-	Concentration of permeate (mg/L)
C_f	-	Concentration of protein feed (mg/L)
$C_{f,i}$	-	Initial permeate concentration (mg/L)
$C_{p,i}$	-	Parameter's final concentration in the permeate (mg/L)
d_w	-	Density of water (g/cm^3)
J_p	-	Flux under steady state (L/m^2h)
Jw_1	-	Initial pure water flux (L/m^2h)
l	-	Membrane thickness (m)
ΔP	-	Transmembrane pressure (Pa),
Q	-	Permeate volume water per time (m^3/s)
R	-	Removal rate (%)
R_a	-	Mean surface roughness
R_{FD}	-	Flux decline
R_{ir}	-	Irreversible fouling (%)
R_r	-	Reversible fouling (%)
R_t	-	Total flux loss
r_m	-	Mean pore radius
t	-	Permeation time (h)
Wt_i	-	Initial weight of sample (mg)
Wt_f	-	Final weight of sample (mg)
Wt_{loss}	-	Weight loss of the sample (mg)
ε	-	Overall porosity
ω_1	-	Weight of wet membrane (g)
ω_2	-	Weight of dry membrane (g)
μ	-	Viscosity of water (mpa.s)

CHAPTER 1

INTRODUCTION

1.1 Research Background

Clean water is essential to life. Over the past decade, clean water scarcity has become a rising global issue due to rapid industrialization and urbanization. Despite its contribution to global economic growth, the improper discharge of untreated or partially treated wastewater into receiving water body that deteriorates water resource quality remains a main concern to public. Industrial effluent, domestic sewage, and agriculture run-off are among the sources of the major pollutants that could pose a substantial threat to surface and underground water if not treated and managed appropriately (Khanzada et al., 2020). World Wildlife Fund (WWF) predicted that two-thirds of the world population would encounter water shortages by 2025 due to the declining available quality and quantity of clean water that has become inevitable (WWF, 2021).

To address the increasing demand for clean water, wastewater treatment and reclamation have become viable alternatives. However, conventional wastewater treatment methods are constantly limited by the requirement of large land, high cost, long retention time, and inability to meet the discharge limit (Fane et al., 2011; Khanzada et al., 2020). As such, membrane technology has garnered attention as a promising wastewater treatment process due to its high separation performance, small system footprint, ease of operation, and cost-effectiveness (Goh and Ismail, 2018; Pendergast and Hoek, 2011). In light of its potential application for various water/wastewater treatment processes, the utilization of membrane technology has undergone rapid development in the last decade.

Membrane can be described as a selective barrier and interphase between two bulk phases that permits passage of specific components while rejecting others (Baker, 2004). Among pressure-driven membrane processes, ultrafiltration (UF) is commonly used in large-scale industrial wastewater treatment processes, owing to its high efficiency, high water production rate, low operation and maintenance cost, and low energy requirement (Nazri et al., 2015; Rambabu and Velu, 2014). Since its commercialization in the 1960s, the global market of UF membranes is expected to grow to nearly $4.6 billion in 2021 (Ramamurthy, 2016). The strict environmental legislation and global water crisis have led to the swift rise of UF membranes, particularly in wastewater treatment. However, membrane surface fouling remains a key challenge for its industrial adoption in the past and present.

In pushing the boundaries to improve membrane surface characteristics, various membrane modification strategies have been explored. This led to an increase in nanocomposite UF membranes, which comprise the dispersion of nanoparticles into the polymer matrix. Many reports have modified UF membrane with nanoparticles such as titanium dioxide (TiO_2) (Hosseini et al., 2018), graphene oxide (GO) (Zinadini et al., 2015), and carbon nanotubes (CNTs) (Rameetse et al., 2020). Besides these nanoparticles, recent studies have reported on the viability of utilizing iron oxide (Fe_3O_4) nanoparticles in enhancing membrane fouling resistance. Fe_3O_4 offers good characteristics such as good hydrophilicity, large surface area, low toxicity, and chemically stable (Gholami et al., 2014; Hosseini et al., 2019). Most importantly, Fe_3O_4 is very cheap and available in the commercial market.

Several studies have attempted to modify membranes with Fe_3O_4 nanoparticles for various wastewater treatment applications, such as removing salts, organic matter, dye, and heavy metal ions (Lakhotia et al., 2019; Rambabu and Velu, 2014). Thus, there are vast opportunities for in-depth research on Fe_3O_4-modified membranes. In view of this, this study aims to provide better insight into the impacts of incorporating Fe_3O_4 on the surface properties of polyethersulfone (PES) hollow fiber membrane for water treatment.

1.2 Problem Statement

UF membranes have been employed in various wastewater treatment processes due to their efficiency in removing suspended and dissolved particles present in industrial wastewater (Rambabu and Velu, 2015). However, membrane surface fouling remains a critical challenge that hinders broader applications of UF membranes in wastewater treatment processes. Various materials have been used as the primary material for the development of UF membranes, such as polyvinylidene fluoride (PVDF) (Tan et al., 2017b), polysulfone (PSf) (Zainol Abidin et al., 2019), and PES (Vatanpour et al., 2019). PES is a common material used in fabricating commercial UF hollow fiber membranes as it has excellent chemical resistance, a wide range of pH tolerance (0 to 14), and high thermal resistance (up to 95˚C) (Sterlitech, 2020). In addition, it shows the best compatibility among other membranes (Lalia et al., 2013).

Despite its advantages, PES-based membranes still suffer from fouling due to its hydrophobic nature, which causes a decline in fluid transport properties during operation and consequently lead to an increase in energy consumption. To address this issue, surface modification using hydrophilic nanoparticles is one of the important strategies to tackle the hydrophobicity of PES membrane while providing other features such as improved surface characteristics (Homayoonfal et al., 2013; Quemener et al., 2017).

Among a wide range of nanoparticles, Fe_3O_4 nanoparticles have shown to be a promising nanosized filler to improve UF membrane (Xu et al., 2012). The abundance of hydroxyl functional groups on its surface makes it highly hydrophilic, which is the key feature in fabricating membrane with excellent filtration performance and fouling resistance. The incorporation of Fe_3O_4 nanoparticles into polymer matrix during membrane preparation has been widely used to modify membrane functionality. Most importantly, the price of commercial Fe_3O_4 (~USD 1.63/g) (Iron (II,III) oxide powder, <5 μm, 2021) is much cheaper compared to the carbon-based particles such as GO (up to USD 500/g) (Graphene oxide sheets, 2021) and CNTs (~USD 40/g) (Carbon nanotube, 2021). This makes the commercial Fe_3O_4 much more competitive and practical.

Nevertheless, typical Fe_3O_4 nanoparticles tend to agglomerate in dope solutions due to strong Van der Waals forces (Ng et al., 2013). To reduce the high surface energies due to having a large surface area, these nanoparticles tend to aggregate. Agglomeration of nanoparticles in dope solution would lead to poor distribution and dispersion in the membrane matrix, negatively affecting membrane structural integrity and applications (Liu et al., 2011). Moreover, agglomerated nanoparticles also increase the propensity of Fe_3O_4 leaching out from the membrane. This would not only cause decline in membrane performance, but also pose hazardous health risks for the end-users. Although reports on Fe_3O_4 nanoparticles leaching from nanocomposite membranes are rare, important strategies are needed to address the agglomeration of Fe_3O_4 issue. Thus, many studies recommended introducing only a small number of nanoparticles (0.1-0.5 wt%) to modify the membrane (Ansari et al., 2016; Daraei et al., 2013).

Besides the quantity of nanoparticles used, another main factor that can result in the formation of different membrane properties is the size of nanoparticles (Ng et al., 2013). In general, particles of smaller size are more favorable as it provides larger surface area for interfacial interactions between nanoparticle and membrane matrix (Bankura et al., 2012; Wang et al., 2019a). As of now, the size effects of Fe_3O_4 on membrane properties have yet been reported in the literature. As such, this study also intends to compare the influence of particles sizes of Fe_3O_4 (50-100 nm and <5 μm) on the properties of PES hollow fiber membranes for water treatment. With respect to price, 5-μm Fe_3O_4 costs only ~USD 1.08/g (Iron (II,III) oxide powder, <5 μm, 2021), i.e., ~34% cheaper than Fe_3O_4 of 50 nm (~USD 1.63/g).

However, it must be pointed out that incorporating particles with a size <100 nm into the polymer matrix is challenging due to strong surface interactions that induce agglomeration between particles. Thus, some studies suggested functionalizing the surface of Fe_3O_4 to enhance its distribution in dope solution (Ghaemi et al., 2015; Rowley and Abu-Zahra, 2019). Surface modification of Fe_3O_4 nanoparticles by surface coating is the most common approach in improving its stability as nanofillers. Coating material such as polydopamine (PDA) is one of the promising candidates that can offer good compatibility, facile surface modification, and ease of preparation, hence enhancing nanoparticle's hydrophilicity and dispersibility without compromising its intrinsic properties (Ramezanpour et al., 2019). The abundance of the functional group of PDA enables it to undergo secondary surface modification to improve further nanoparticle stability in organic solvents and effects on the membrane surface properties.

To achieve secondary surface modification, agents such as (3-aminopropyl) triethoxysilane (APTES) and sulfonic acid could be potentially used to enhance nanoparticle stability so as it can efficiently reduce nanoparticle agglomeration by demonstrating a synergistic effect, hence improving the interactions between nanofillers and membrane matrix (Veisi et al., 2016; Zhang et al., 2019). However, only a few researches have been reported on the UF membranes modification with PDA-coated Fe_3O_4 nanoparticles as a nanofiller. Nonetheless, it is necessary to fully comprehend the roles of different types of surface-functionalized Fe_3O_4 nanoparticles in modifying PES membrane for enhanced separation performance. In addition, this study also intends to provide important insight on underlying issues related to membrane fouling resistance, aiming to develop an effective membrane for water and wastewater treatment.

1.3 Research Objectives

This study aims to develop new type of nanocomposite PES hollow fiber membrane for water treatment. The objectives of this study can be further elaborated as follows:

1. To investigate the influence of particle sizes of Fe_3O_4 (50 nm and <5 μm) on the surface properties and filtration performance of PES hollow fiber membranes for water treatment.

2. To evaluate the effects of different surface functionalization on the best particle size of Fe_3O_4 in improving hydrophilicity and antifouling properties of PES nanocomposite membranes for water treatment.

1.4 Scope of Study

To achieve the objectives above, the following scopes of this study are identified:

1. Characterization of commercial Fe_3O_4 particles (particle size: 50 nm and <5 μm) by transmission electron microscopy (TEM), Brunauer-Emmett-Teller (BET) surface area analysis, Fourier transform infrared (FTIR), X-ray diffractometry (XRD), and dispersion study.

2. Preparation of polymeric dope solutions composed of 20 wt% PES and 0.1 wt% of Fe_3O_4 of different particle sizes by conventional blending method.

3. Fabrication of PES hollow fiber membranes with and without inorganic particles by non-solvent induced separation (NIPS) technique at constant spinning conditions (air gap: 10 cm, dope extrusion rate: 6 mL/min, bore fluid rate: 2.0 mL/min, and spinneret i.d/o.d.: 0.8 mm/1.2 mm).

4. Characterization of fabricated PES hollow fiber membranes by field emission scanning electron microscopy (FESEM), energy dispersive x-ray (EDX), thermal gravimetric analysis (TGA), Fourier transform infrared (FTIR), contact angle, membrane porosity, and tensile strength.

5. Evaluation of fabricated PES hollow fiber membranes filtration performance with respect to pure water flux and solute rejection against 500 ppm bovine serum albumin (BSA) at 1 bar to identify the best particle size of Fe_3O_4.

6. Surface functionalization of best particle size of Fe_3O_4 with PDA through direct addition of coating solution containing dopamine and Tris buffer (10mM, pH 8.5) to produce Fe_3O_4/PDA particles.

7. Secondary surface modification of Fe_3O_4/PDA particles with (3-aminopropyl) triethoxysilane (APTES) and sulfonic acid to obtain amine-modified polydopamine-coated Fe_3O_4 particles (Fe_3O_4/PDA-Amine) and sulfonic acid-modified polydopamine-coated Fe_3O_4 particles (Fe_3O_4/PDA-Sulf), respectively.

8. Characterization of surface-functionalized Fe_3O_4 particles by TEM, BET surface area analysis, FTIR, XRD, and dispersion study.

9. Preparation of PES hollow fiber membranes dope solution using different types of surface-functionalized Fe_3O_4 nanoparticles at fixed loading (0.1 wt%) by conventional blending method.

10. Fabrication of PES hollow fiber membranes by NIPS technique at fixed spinning conditions (air gap: 10 cm, dope extrusion rate: 6 mL/min, bore fluid rate: 2.0 mL/min, and spinneret i.d/o.d.: 0.8 mm/1.2 mm).

11. Characterization of fabricated PES hollow fiber membranes by FESEM, EDX, TGA, FTIR, contact angle, membrane porosity, and tensile strength.

12. Evaluation of fabricated PES hollow fiber membranes filtration performance with respect to pure water flux and solute rejection against 500 ppm BSA and river water at 1 bar.

13. Assessment of fouling resistance of selected PES hollow fiber membranes with respect to flux stability and flux recovery rate.

1.5 Significance of Study

This study intends to better understand the underlying principle of the fabrication of UF membranes with enhanced antifouling properties for water treatment by considering the changes in the morphological structure of membrane and separation performance due to the addition of nanoparticles into the polymer matrix. It is known that the extent of fouling depends on the surface roughness, hydrophilicity, and morphological structure of a membrane. Therefore, hydrophilic membranes with excellent fouling resistance can be fabricated by identifying the ideal properties of UF membrane. Currently, works related to the effects of particle sizes on membrane properties are limited to other nanoparticles such as TiO_2. The findings of this study are expected to provide insight into the influences of particle sizes of Fe_3O_4 on the properties of PES hollow fiber membranes. This study would also fill the knowledge gap on the use of surface-functionalized Fe_3O_4 in improving the hydrophilicity and antifouling properties of the resultant membranes. The outcome of this research would be helpful to comprehend further the application of nanocomposite UF membranes for wastewater treatment, which could offer great potential for the broader utilization of UF membranes.

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LIST OF PUBLICATIONS

Journal

1. **Nawi, N. S. M.**, Lau, W. J., Yusof, N., Said, N., & Ismail, A.F. (2021). Surface Modification of PES Hollow Fiber Membranes using Iron Oxide Particles for Water Treatment: Does Particle Size Really Matter?. *Malaysian Journal of Fundamental and Applied Sciences*. 17(5), 621-635. **(SCOPUS)**
2. **Nawi, N.S.M.**, Lau, W.J., Yusof, N., & Ismail, A.F. (2022). The Impacts of Iron Oxide Nanoparticles on Membrane Properties for Water and Wastewater Applications: A Review. *Arabian Journal for Science and Engineering*. **(IF: 2.334)**
3. **Nawi, N.S.M.**, Lau, W.J., Yusof, N., Said, N., & Ismail, A.F. (2022). Enhancing Water Flux and Antifouling Properties of PES Hollow Fiber Membranes via Incorporation of Surface-functionalized Fe_3O_4 Nanoparticles. *Journal of Chemical Technology & Biotechnology*. **(IF: 3.174)**

Conference

1. **Nawi, N.S.M.**, Lau, W.J., & Yusof, N. (2021). Hollow Fiber Membranes Incorporated with Iron Oxide Nanoparticles for Palm Oil Mill Effluent (POME) Treatment, *Regional Congress on Membrane Technology 2020 & Regional Conference Environmental Engineering 2020 (RCOM 2020 & RCEnvE 2020)*, 16-17th January 2021. Virtual Conference.