SUPERHYDROPHILIC POLYETHERSULFONE-BASED ELECTROSPUN NANOFIBROUS ULTRAFILTRATION MEMBRANES FOR OILY WASTEWATER TREATMENT

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DEDICATION

In the name of Allah, the most Gracious, the most Merciful. May Allah's benedictions and salutations be upon to His beloved messenger Muhammad, members of His household and His companions.

This work is dedicated to my parents, brothers, sisters, wife and kids for their love, prayers and support.

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ABSTRACT

Lately, oil polluted water and its separation have received much attention in the context of growing environmental concerns in broad. In this regard, technology based on polymeric electrospun nanofibrous membranes (ENMs) has become an emerging solution for oil effluents, water filtrations and water treatments. However, highly porous structure and inadequate mechanical integrity of ENMs could result in great fouling tendency towards rapid flux deterioration in oil-water separation, unless inhibited. Thus, in this work, some novel ultrafiltration (UF) ENMs incorporated with iron oxide (Fe_3O_4) and hydrous manganese dioxide (HMO) nanoparticles (NPs) were synthesized. Subsequently, these UF-ENMs were characterized to determine their effectiveness for the removal of oil from industrial wastewater. The mechanical property and hydrophilicity of polyethersulfone (PES)-based ENMs were improved by three strategies. First, *N*-methyl-pyrrolidinone (NMP) was added to dimethylformamide (DMF), wherein the solvent stimulated fusion of the inter-fiber junctions was enhanced. Second, Fe_3O_4-HMO NPs were inserted into the ENMs to improve their anti-fouling resistance against oil molecules. Third, hot press technique was used to strengthen the electrospun mat. The morphology, structure, mechanical strength, water contact angle (WCA), water permeability and oil emulsion in water filtration capacity of these prepared ENMs were evaluated. The results disclosed an enhancement in the mechanical strength, hydrophilicity, permeability and selectivity of these ENMs due to the inclusion of Fe_3O_4-HMO NPs. Compared to pristine ENMs, the tensile strength and the elongation-at-break of the Fe_3O_4-HMO NPs incorporated ENMs were correspondingly improved by 225.88% and 40.91%, whereas the WCA was reduced by 89.8% (from 123.27° ± 0.23 to 12.34° ± 0.19). It was also found that the hot pressed PE/Fe_3O_4-HMO ENMs possessed smaller pore diameter (61.05 nm) compared to pristine PES ENMs (97.15 nm). The Fe_3O_4-HMO NPs modified ENMs were found to exhibit excellent oil elimination (98.37% ± 0.49 and 91.02% ± 0.69) and outstanding water flux recovery performance (87.32% ± 0.70 and 74.65% ± 0.73) when tested with synthetic oil solution (12,000 ppm) and oily effluents (7,000 ppm), respectively. Water productivity of over 4,000 L/m^{2}h was achieved without forfeiting the rate of oil removal under gravity. The observed improved low flux declination revealed by the proposed ENMs was attributed to their improved surface resistance mediated oil anti-fouling qualities. The enhanced mechanical and oil anti-fouling traits of the prepared UF-ENMs were established to be a prospective for the treatment of diverse industrial oily effluents, especially emulsions of oil–water.

ABSTRAK

Sejak kebelakangan ini, air tercemar dengan minyak dan pemisahannya telah mendapat banyak perhatian dalam konteks kesedaran penjagaan alam sekitar yang semakin meningkat secara keseluruhan. Dalam hal ini, teknologi yang berasaskan membran polimer serat nano elektroputaran (ENMs) telah muncul sebagai satu penyelesaian bagi rawatan efluen minyak, penapisan air dan rawatan air. Walau bagaimanapun, struktur ENMs yang sangat berliang dan kekuatan mekanikal yang tidak mencukupi boleh menyebabkan kecenderungan yang besar untuk berlaku fouling ke arah kemerosotan fluks yang cepat dalam pemisahan minyak-air, melainkan jika ia dihalang. Oleh itu, dalam kajian ini, beberapa ENMs ultrapenapisan (UF) baharu yang menggabungkan nanopartikel (NPs) ferum oksida (Fe_3O_4) dan mangan oksida hidrous (HMO) telah disintesis. Seterusnya, UF-ENMs ini telah dicirikan untuk menentukan keberkesanannya dalam penyingkiran minyak daripada air sisa industri. Sifat mekanikal dan hidrofilik ENMs berasaskan polietersulfon (PES) telah dipertingkatkan dengan menggunakan tiga strategi. Pertama, *N*-metil-pirolidinon (NMP) ditambahkan dengan dimetilformamida (DMF), di mana gabungan dirangsang-pelarut bagi persimpangan antara serat telah dipertingkatkan. Kedua, NPs Fe_3O_4-HMO dimasukkan ke dalam ENMs untuk meningkatkan rintangan anti-fouling terhadap molekul minyak. Ketiga, teknik tekanan panas digunakan untuk mengukuhkan lapisan elektroputaran. Morfologi, struktur, kekuatan mekanikal, sudut sentuh air (WCA), kebolehtelapan air dan emulsi minyak dalam kapasiti penapisan air bagi ENMs yang disediakan telah dikaji. Hasil kajian mendapati penambahbaikan dalam kekuatan sifat mekanikal, hidrofilik, kebolehtelapan dan kepilihan ENMs disebabkan oleh penambahan NPs Fe_3O_4-HMO. Berbanding dengan ENMs yang murni, kekuatan tegangan dan pemanjangan pada pecahan bagi NPs Fe_3O_4-HMO yang digabungkan dengan ENMs masing-masing telah meningkat sebanyak 225.88% dan 40.91%, manakala WCA berkurang sebanyak 89.8% (dari 123.27° ± 0.23 hingga 12.34° ± 0.19). Ia juga mendapati ENMs PE/Fe_3O_4-HMO yang ditekan-panas mempunyai diameter liang yang lebih kecil (61.05 nm) berbanding dengan ENMs PES yang murni (97.15 nm). Didapati ENMs yang diubahsuai dengan NPs Fe_3O_4-HMO menunjukkan penyingkiran minyak yang sangat baik (98.37% ± 0.49 dan 91.02% ± 0.69) dan prestasi pemulihan fluks air yang sangat baik (87.32% ± 0.70 dan 74.65% ± 0.73) apabila diuji dengan larutan minyak sintetik (12,000 ppm) dan efluen berminyak (7,000 ppm). Produktiviti air melebihi 4,000 L/m^2h telah dicapai tanpa menjejaskan kadar penyingkiran minyak di bawah tarikan graviti. Penurunan fluks rendah yang baik yang didedahkan oleh ENMs yang dicadangkan ini disebabkan oleh penambahbaikan kualiti anti-fouling minyak melalui rintangan permukaan. Peningkatan ciri-ciri mekanikal dan anti-fouling minyak bagi UF-ENMs yang disediakan ini dipercayai boleh dijadikan sebagai satu prospektif untuk rawatan pelbagai efluen berminyak industri, terutamanya emulsi minyak-air.

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LIST OF ABBREVIATIONS

Ag	-	Silver
Al_2O_3	-	Aluminium oxide
APAN	-	Amination of polyacrylonitrile
BOD	-	Biological oxygen demand
CNF	-	Carbon nanofiber
CNTs	-	Carbon nanotubes
CA	-	Cellulose acetate
COD	-	Chemical oxygen demand
CVD	-	Chemical vapor deposition
CO	-	Crude oil
DI	-	Deionized
DMSO	-	Dimethyl sulfoxide
DMAc	-	Dimethylacetamide
DMF	-	Dimethylformamide
DO	-	Dissolved oxygen
DWEs	-	Downward electrospinning setups
DLS	-	Dynamic light scattering
ETP	-	Effluent treatment plant
ED	-	Electrodialysis
ENMs	-	Electrospun nanofibrous membranes
EDS	-	Energy dispersive x-ray
FESEM	-	Field emission scanning electron microscope
F-PBZ/SiO_2	-	Fluorinated polybenzoxazine (F-PBZ) - incorporated silicon dioxide (SiO_2)
FO	-	Forward osmosis
FTIR	-	Fourier transform infrared spectroscopy
GO	-	Graphene oxide
HR	-	High resolution

HAO	-	Hydrous aluminium oxide
HMO	-	Hydrous manganese dioxide
IC	-	Inorganic carbon
IP	-	Interfacial polymerisation
Fe_3O_4	-	Iron oxide
IPA	-	Isopropyl alcohol
LEP	-	Liquid entry pressure
LMH	-	Liters per meter squared per hour, metric flux units
MgO	-	Magnesium oxide
MD	-	Membrane distillation
MF	-	Microfiltration
Mw	-	Molecular weight
MNEs	-	Multi-needle electrospinning
MWCNTs	-	Multiwalled carbon nanotubes
PU ($PU-C_{18}H_{37}$)	-	N-alkyl-substituted
NMs	-	Nanofibrous membranes
NF	-	Nanofiltration
NPs	-	Nanoparticles
N_2	-	Nitrogen
NMP	-	N-methyl-pyrrolidone
NDIR	-	Non-dispersive Infared detector
PU ($PU-C_8F_{17}$)	-	N-perfluorooctyl-substituted
OCMCS	-	O-carboxymethyl chitosan
OCA	-	Oil contact angle
Ppm	-	Parts per million
PV	-	Pervaporation
PDO	-	Petroleum development Oman
PS	-	Phase separation
PLA	-	Poly(lactic) acid
PVA	-	Poly(vinyl alcohol)
PAM	-	polyacrylamide

PMA	-	Polyacrylamide
PAN	-	Polyacrylonitrile
PEGDA@PG NF	-	Polyacrylonitrile/polyethylene glycol nanofibrous
PBZ	-	Polybenzoxazine
PDMS- -*b*-P4VP	-	polydimethylsiloxane -block- poly (4-vinylpyridine)
PES	-	Polyethersulfone
PEG	-	Polyethylene glycol
PEO	-	Polyethylene oxide
PET	-	Polyethylene terephthalate
PLLA	-	Poly-L-lactide acid
PIM-1/POSS	-	Polymer of intrinsic microporosity (PIM-1)/polyhedral oligomeric silsesquioxane (POSS)
PP	-	Polypropylen
PS	-	Polystyrene
PSF	-	Polysulfone
PSU	-	Polysulfone
PTFE	-	Polytetrafluoroethylene
PU	-	Polyurethane
PVA–MWCNTs/PAN	-	Polyvinyl alcohol (PVA)- multiwalled carbon nanotubes (MWCNTs)/polyacrylonitrile (PAN)
PVC	-	Polyvinyl chloride
PVDF	-	Polyvinylidene fluoride
PVDF-HFP	-	Polyvinylidene fluoride-co-hexafluoropropylene
PVP	-	Polyvinylpyrrolidone
RH	-	Relative humidity
RO	-	Reverse osmosis
RT	-	Room temperature
SNM	-	Silica nanofiber membrane
SiO_2	-	Silica oxide
SiO_2-CNFs	-	Silicon dioxide (SiO_2)-coated with carbon nanofibers (CNFs)
SNE	-	Single needle electrospinning
SDS	-	Sodium dodecyl benzenesulfonate

SS	-	Suspended solids
TGA	-	Thermo-gravimetric analysis
TO	-	Thermo-osmosis
TiO_2	-	Titanium dioxide
TC	-	Total carbon
TOC	-	Total organic carbon
TMP	-	Trans-membrane pressure
TEM	-	Transmission electron microscopy
UF	-	Ultrafiltration
UV	-	Ultraviolet
UWEs	-	Upward electrospinning setups
WWTP	-	Wastewater treatment plant
WCA	-	Water contact angle
XRD	-	X-ray diffraction
ZnO	-	Zinc oxide

LIST OF SYMBOLS

$R_{oil}\%$	-	Oil rejection efficiency
ρ_{H_2O}	-	Density of the pure water (0.998 g/cm^3)
ρ_{PES}	-	Density of the PES polymer (1.37 g/cm^3)
µS	-	Microsecond
Å	-	Angstrom
bbl	-	Barrel (unit)
C	-	Concentration
C_{feed}	-	Solute concentration in feed solutio
$C_{oil,F}$	-	Oil content (mg/L) in the feed solution
$C_{oil,P}$	-	Oil content (mg/L) in the permeate solution
cP	-	Centipoise
$C_{permeate}$	-	Solute concentration in permeate solution
Jw	-	Pure water permeation flux
kDa	-	Kilodaltons
kV	-	Kilovolts
L	-	Membrane thickness
mm	-	Millimeter
mN/m	-	Millinewton/meter
MPa	-	Megapascal
ms	-	Millisecond
nm	-	Nanometer
p	-	Pressure
Pa s	-	Pascal-second
R_{FR}	-	Flux recovery rate
rpm	-	Revolutions per minute
T	-	Temperature
T_B	-	Boiling temperature
T_d	-	Decomposition temperature

Tg	-	Glass-transition temperature
Tm	-	Melting temperature
V	-	Mass average velocity
V	-	Molar volume of water
W_d	-	Weight in dry state
W_w	-	Weight in wet state
ε	-	Porosity
μ	-	Viscosity of liquid
μL	-	Microlitre
μm	-	Micrometer
ρ	-	Mass density (mass per volume)

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CHAPTER 1

INTRODUCTION

1.1 Research Background

In recent years, separation of oil from contaminated water has received much attention due to the growing environmental problem wherein water pollution by oil is very common in countries like Oman. Large amount of oil contaminated water as waste are produced as oil-water emulsion by diverse industries including oil, petrochemical, metallurgy, pharmaceutical etc., (Lai *et al.* 2017; Gohari *et al.*, 2014). Such oil contaminated wastewater contains different chemicals and minerals that are hazardous to human health and have negative impact on the ecosystem. To mitigate this problem, government agencies are increasingly setting up strict regulations to eliminate these hazardous elements from wastewater prior to discharge (Ong, 2015; Gohari *et al.*, 2014; Ong *et al.*, 2014).

The conventional methods for oil-water separation including gravity, skimming and flotation are helpful for free oil and water mixtures when oil droplet sizes are above 150 μm and the range of dispersed oil particle size is between 20 to 150 μm. However, these techniques are impractical for oil and water emulsions containing small size particles below 20 μm. In fact, the poor efficiency and expensive operation process limits the wide applicability of traditional techniques to separate oil from polluted water (Prince *et al.*, 2016; Ong *et al.*, 2014). Thus, advanced methods are required for effective separation of oil from various oil and water emulsions. Meanwhile, separation processes based on advanced polymer membranes have emerged. These membranes are produced by electrospinning procedures for enhancing the efficacy of oil separation from water and maximizing the recycled water and oil resources (Kota *et al.*, 2012; Shang *et al.*, 2012).

At present, two types of membranes are utilized for oil-water separation depending on the specific liquid mediated surface wettability of electrospun nanofibrous membranes (ENMs). The first kind is called superhydrophobic (water-hating) ENMs surfaces, wherein such membranes repel water when it enters the pores, allowing the penetration of oils and thereby eliminating oils from the oil/water emulsion (Zhang *et al.*, 2015 a; Xue *et al.*, 2014). The other type is superhydrophilic (water-loving) ENMs surfaces, which selectively detach water from oil/water emulsion, repelling the oil to enter into the pores (Ma *et al.*, 2016; Xue *et al.*, 2014; Zhu *et al.*, 2014). The superhydrophobic ENMs have problems related to the adherence of oils with high viscosity to the membrane surface thereby extra chemicals are needed to remove the oil (Zhang *et al.*, 2019). On the top, the high turbulent flow is required to drive the oil emulsion towards the membrane, which is energy intensive and costly for the system. Superhydrophilic ENMs, due to their water affinity and high surface energy, can readily form hydrogen bonds up on contact with water, thus repelling the hydrophobic oil particles including hydrocarbons, surfactants, grease, and so on.

To the best of amid knowledge, studies on ultrafiltration (UF) ENMs for the separation of industrial oily effluents at large scale have not been reported yet. The asymmetric UF membranes have been prepared using phase inversion methods. Moreover, the high porosity of ENMs leads to poor mechanical properties, causing a tendency to break easily, which in turn, causes rapid fouling with a considerable loss of flux. Therefore, the oil-water separation performances of ENMs from the treatment of industrial effluents are affected (Choong, 2015, Yoon *et al.*, 2009). In this regard, the membrane materials are significant for successful realization of UF-ENMs technology to treat oily wastewater. It is important to mention that the Oman marine environment is the primary source of potable water (Price *et al.*, 1993) and operates as a significant reservoir for seafood including fish and other biota (De Mora *et al.* 2004). So far, no study has been carried out on the Effluent Treatment Plant (ETP) at Petroleum Development Oman (PDO), Mina Al Fahal, Sultanate of Oman. According to Oman marine environment, this site is important because a huge amount of industrial oily effluents is discharged annually before being treated properly.

Considering the urgent necessity of treating the contaminated oil/water emulsion from diverse industrial effluents, this work is an attempt to prepare some novel UF-ENMs by mixing polyethersulfone (PES) and hydrous manganese dioxide (HMO) to enhance UF process to treat oily wastewater efficiently. In the past, several industries have intensively used PES (excellent permeability, good selectivity, high mechanical and thermal stability, and strong chemical resistance) as asymmetric UF membrane material owing to its distinct properties (Lee *et al.*, 2016; Sun, 2010). Moreover, the eventual fouling associated with the hydrophobic character of PES-based membranes is the main problem that limits the applicability (Salimi *et al.*, 2016; Gohari *et al.*, 2014). The poor mechanical strength of ENMs is responsible for such fouling trend, leading to rapid flux deterioration (Choong, 2015). It has been established that the surface hydrophilicity and mechanical integrity of the PES based ENMs are vital for the pure water flux and antifouling resistance against oil deposition and/or adsorption. Consequently, dedicated efforts have been made to progress the surface hydrophilicity and mechanical integrity of the polymeric ENMs. To this end, surface micro-nano surface textures were developed for effective separation of oil from water, resulting in superhydrophilic surfaces with low adhesion of oil (Zhu *et al.*, 2014; Zhang *et al.*, 2013; Kota *et al.*, 2012).

Polymeric membranes based superhydrophilic nanomaterial's including HMO and iron oxide (Fe_3O_4) NPs have been focused owing to their large surface area, high mechanical strength, and good chemical stability. Meanwhile, HMO and Fe_3O_4 NPs, owing to their capacity to remove toxic agents and pathogens, appeared promising for wastewater treatment and environmental safety (Koushkbaghi *et al.*, 2018; Zuluaga *et al.*, 2017; Li *et al.*, 2016 a; Ghaemi *et al.*, 2015; Mittal *et al.*, 2014; Wan *et al.*, 2014; Jamshidi Gohari *et al.*, 2013; Jin *et al.*, 2013; Su *et al.*, 2010; Ling *et al.*, 2010; Guo *et al.*, 2011; Daraei *et al.*, 2012; Hakami *et al.*, 2012). To treat oil-water emulsions efficiently, Fe_3O_4 NPs and HMO have been used to improve the asymmetric UF characteristics of membranes prepared using phase inversion approach (Lai *et al.*, 2017; Gohari *et al.*, 2014; Rahimi *et al.*, 2014). The superhydrophilic nature of HMO and Fe_3O_4 NPs are advantageous for the attachment of large number of OH functional groups to their surface. The HMO and Fe_3O_4 NPs incorporated membrane surfaces have improved hydrophilicity, thereby showing enhanced water permeability and strong anti-fouling resistance towards oil-water emulsion treatment. Despite some studies conducted on the UF-based superhydrophilic ENMs materials synthesis and characterizations for separating oil from water, a clear understanding on the mechanisms of oil removal remains deficient. Moreover, a high performing novel UF-PES based ENMs for effective treatment of oil-water effluents is far from being achieved.

In this study, ultra-wetting HMO and Fe_3O_4 NPs combined ENMs were prepared to examine their oil-water separation performance. These novel UF-ENMs were synthesized using hot press technique. In this process an appropriate amount of hydrophilic polyvinylpyrrolidone (PVP) polymer, HMO and hydrophilic Fe_3O_4 NPs were added into the PES solution to achieve enhanced oil removal efficiency of UF-ENMs. Furthermore, the oil separation traits of these proposed UF-PES-ENMs were evaluated by subjecting them to synthetic oily solution and industrial oily effluents wherein different processing conditions were adopted. Properties of UF-PES-ENMs such as long-term stability with respect to water flux, oil rejection, and recovery rate features were analyzed to get an understanding of the mechanism of oil removal from polluted water. As a case study, Effluent Treatment Plant (ETP) at Petroleum Development Oman (PDO) in Mina Al Fahal in the Sultanate of Oman has been selected as the perfect site wherein the samples were collected for the treatment of industrial oily effluents before being discharged into the marine environment.

1.2 Problem Statement

With increasing environmental awareness and tighter regulations, it is imperative to develop new technologies and new materials for oil/water process, targeting industrial wastewaters and, polluted oceanic waters. UF membrane technology is one of the most effective ways to separate oil–water mixtures for a wide range of industrial effluents and has been widely applied in various separation processes. UF membranes with pore sizes between 0.002 and 0.05 μm have been used to remove stable oil particles from wastewater, owing to its high water oil separation efficiency and permeability under low operating pressure (Chakrabarty *et al.*, 2010; Ong *et al.,* 2014; Ong, 2015). However, the filtration performance was found to vary with pore diameter of the membranes, and decreasing oil rejection was observed as the pore size was enlarged (Ong, 2015). In addition, typical oil rejection of UF ranges from 80% to less than 99%, hence, the UF membrane surface is tended to be easily fouled at high oil concentration (Ong, 2015; Ju *et al*., 2008).

In this regard, the water flux deterioration and mechanical integrity are major issues for oil-water separation related to the traditional membranes that need to be resolved. Generally, the existing UF membranes face practical problems during the oily water treatment of industrial effluents, revealing strong fouling tendency of the membrane surface for prolonged usage due to the adsorption and accumulation of oil particles. Such shortcomings of UF membrane have a negative impact on its water permeability thereby make it less favourable for oily wastewater treatments (Ong, 2015; Gohari *et al.,* 2014). Consequently, membranes with stronger anti-fouling capacity need to be developed to overcome the problem related to oily wastewater treatment wherein the adsorption of oil droplets on the membrane surface must be minimized. For efficient treatment of oil-water separation, the surface of the membranes needs to be designed either with superhydrophobic or superhydrophilic properties (Zhang *et al*., 2015). Most of the commercially available UF-ENMs are commonly made of hydrophobic materials; therefore, such membranes are easily inclined to oil fouling. To fulfil such demand, synthesis of membrane surface with improved hydrophilic or superhydrophilic properties is required in order to inhibit its fouling by lowering the adsorption of oil droplets on the surface.

The selection of membrane materials is the most significant aspect towards achieving high performance UF membrane technology in oily wastewater treatments. For UF application, superhydrophilic electrospun fibrous attributes with water loving tendency are needed. In this regard, PES based ENMs have emerged as suitable system. Over the years, diverse strategies have been developed to improve the mechanical strength and oil anti-fouling traits for PES based ENMs. Due to their many distinguished properties, the PES material has intensively been used as an element of asymmetric UF membrane in different industrial applications (Liu *et al.*, 2012). It was demonstrated that the hydrophobic features of PES based membranes make them sub-optimal to separate water from oil/water mixtures and susceptible to fouling when applied to remove hydrophobic oil molecules. Consecutively, the water-flux stability and durability of the membranes was enhanced significantly. Despite some efforts, PES based high performance UF-ENMs for oil-water effluent treatment is far from being achieved.

Due to several drawbacks associated with PES based UF-ENMs for removing oil from industrial effluents, these membranes have not yet been exploited at commercial scale. The main constraint was attributed to the inability of ENMs to withstand high pressures commonly used in the UF processes. This could be due to the electrospinning mediated improper alignments of the polymer chains and inadequate interactions among nanofibers in ENMs (Panatdasirisuk *et al.*, 2017; Yoon *et al.*, 2009). Yet again, the high porosity and poor mechanical integrity of ENMs are responsible for a strong fouling trend towards speedy flux weakening (Ma *et al.*, 2016; Ahmed *et al.*, 2015). Conversely, the low solidity of ENMs due to the presence of flexible and very thin fibers, which can offer infinite flux even when the membranes operate at low pressures, is detrimental to applications (Panatdasirisuk *et al*, 2017).

Therefore, the ENMs prepared using PES only cannot be used directly in the UF processes to eliminate oil from oil/water mixtures to mitigate the water pollution caused by industrial effluents. The inclusion of Fe_3O_4 and HMO NPs mixture into the ENMs dope solution could enhance their superhydrophilic properties, resulting in a high oil rejection rate in treating industrial oily effluent during UF processes. Thus, improvement of these ENMs in terms of their mechanical strength, interfacial stability and enhance their superhydrophilic properties is mandatory to obtain enhanced oil removal rate (treatments with high performance) from various oily effluents generated by diverse industries. Particularly, no investigation has been made for treating oily effluents generated by Effluent Treatment Plant (ETP) at Petroleum Development Oman (PDO) in Mina Al Fahal in the Sultanate of Oman.

1.3 Research Objectives

Based on the above mentioned research gaps, the following objectives emerged for the present work:

i. To fabricate the polyethersulfone electrospun nanofibrous membranes (PES-ENMs) with improved attributes, which include enhanced mechanical traits and interfacial stability using mixed solvents united hot press technique.

ii. To develop a new type of superhydrophilic PES-ENMs doped with nanoparticles of iron oxide (Fe_3O_4) and hydrous manganese dioxide (HMO) blend for enhanced ultrafiltration (UF) process in treating synthetic oily solution and industrial oily effluents samples.

iii. To determine the water productivity and oil removal capacity of the resultant UF-PES-ENMs in treating the synthetic oily solution under different conditions.

iv. To validate the enhanced oil-water separation performance of the developed optimum UF-PES-ENMs using industrial oily effluents under different conditions for verifying their stability with respect to the water flux, oil rejection and recovery rate.

1.4 Scope of the Study

To accomplish the above said objectives, the following research scope is designed and presented in two stages. The first stage dealt with the synthesis, and characterizations of UF-PES-ENMs that are efficient in removing oil from the industrial oily effluents. The second stage involved the evaluation of oil-water separation performance of PES-ENMs and PES/NPs-ENMs using cross flow UF system.

Stage I: Synthesis and characterizations of ENMs and NPs incorporated ENMs with enhanced physiochemical properties such as mechanical strength, interfacial stability and hydrophilicity. Two mixed solvents and a hot press technique was used to attain enhanced UF process for effective oil removal which was achieved following the steps described below:

i. Preparing PES, PES/PVP, PES/Fe_3O_4, PES/HMO and PES/Fe_3O_4-HMO ENMs doped solutions using fixed ratio of DMF/NMP solvents (50:50 wt%) followed by electrospinning technique. Using fixed electrospinning operating parameters including applied voltage, flow rate, drum's rotation speed and spinning distance to produce optimum ENMs.
ii. Enhancing the interfacial stability of the resultant ENMs by using hot press technique (at temperature of 210°C and pressure of 1 bar for the duration of 10 min).
iii. Determining surface morphology, structure and physical properties of the prepared ENMs including mechanical strength, thermal stability and surface hydrophilicity of ENMs via scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS), transmission electron microscopy (TEM), fourier transform infrared spectroscopy (FTIR), thermo-gravimetric analysis (TGA), water contact angle (WCA), tensile testing machine and pore size measurement.

Stage II: The evaluation of oil-water separation performance of PES-ENMs and PES/NPs-ENMs using cross flow UF system which was achieved following the steps described below:

i. Testing the performance of the fabricated ENMs, in terms of their water productivity using cross flow UF system before subjecting to oily solution or oily effluents.
ii. Measuring the physicochemical characteristics of the synthetic oily solution and industrial oily effluents samples such as pH, viscosity, salinity, sizes of emulsion droplets, chemical oxygen demand (COD) and total organic carbon (TOC).

iii. Evaluating the separation characteristics of the prepared PES-ENMs in cross flow UF mode to confirm its stability for continuous two hours of filtration test for treating two different kind of samples, separately, as follows:

- Testing of the prepared PES-ENMs and the proposed nanomaterial incorporated PES-ENMs for treating the standard oily solution (12,000 ppm).
- Validating the oil separation performance rate of the developed optimum PES/NPs-ENMs for the industrial oily effluents (7,000 ppm).

iv. Analyzing the separation performance of the prepared PES-ENMs against the standard oily solutions (12,000 ppm) and industrial oily effluents (7,000 ppm) in cross flow UF mode (from stage iii) with respect to water permeability flux, oil rejection, and water flux recovery ratio properties within 2 hours of experimental period under various operating conditions.

v. Measuring oil concentration in the feed and permeate solutions by using the UV-vis spectrophotometer.

1.5 Significance of Study

This study is very important from the perspective of a new type of UF-PES-ENMs development, which is useful for protecting the marine environment worldwide in general and Oman in particular from the contamination caused by industrial oily effluents before being discharged into marine environment. This is the first ever study related to the development of promising technique for the treatment of industrial oily effluents in Oman and neighbouring regions. The implementation of prepared UF-PES-ENMs for selective oil-water separation being a relatively new technology is shown to offer considerable benefits for oily wastewater treatments. In this way, the separation efficiency was improved, and high permeable membranes were created for oil–water separation with reduced energy consumption. Thus, superhydrophilic ENMs can generate renewed interests and open up new avenues for industrial oily effluent treatment. Furthermore, this research has helped understand the mechanism behind the separation process and as a result new body of knowledge has been generated. These kinds of membranes were shown to have strong resistance against fouling by oils, leading to high permeability and rejection rates in separating water from oil-water mixtures.

So far, there is a lack of literature on separation performance of UF-ENMs for treating the large scale industrial oily effluents. Thus, a comparison on the oil separation performance efficiency of the produced UF-PES-ENMs was made with the asymmetric UF membranes via phase inversion technique. The major issue related to the poor pressure withstanding capacity of the traditional ENMs generally used in the UF process was resolved. The highly porous nature of ENMs was the main reason for extremely low pressure (less than 1 bar) requirement for their operation, which was surmounted by enhancing the mechanical integrity of the membranes. Besides, this high porosity of ENMs that was responsible for strong fouling tendency and thus rapid flux deterioration was overcome. This in turn could improve the oil-water separation performance of ENMs such as water-flux stability and durability during the filtration process of oily effluents. This is the first report on the synthesis of UF PES-based ENMs following two approaches where mixtures of DMF/NMP solvents were combined. Such unification and the use of hot pressing technique provided notable advantages to strengthen the electrospun mat layer during liquid UF processes. Thus, the mechanical traits such as the tensile strength of the electrospun membrane mat were enhanced without altering the fiber morphology (size, shape and dimension). Furthermore, the use of PVP as hydrophilic additive for the fabrication of asymmetric UF membranes made via phase inversion technique was beneficial. Inspired by this rationale, a specific quantity of hydrophilic of PVP was added into the PES-ENMs doped solution to enhance pore density and to induce a hydrophilic character of the membrane for improved water flux filtration.

Most importantly, the inclusion of Fe_3O_4 NPs and HMO mixture by changing the ratio between NPs into the ENMs dope solution could enhance their superhydrophilic properties, resulting in a high oil rejection rate in treating industrial oily effluent during UF processes. Furthermore, the combination of hydrophilic

Fe_3O_4 NPs and HMO led to interesting attributes of the UF-PES-ENMs in treating industrial oily effluents and made them good candidate for many applications due to their excellent mechanical and thermal stability. In short, the blend of hydrophilic Fe_3O_4 NPs and HMO into the ENMs dope solution was innovative towards the development of UF-PES-ENMs. Through this research work, it was possible to identify various operating conditions suitable to achieve optimum ENMs surfaces and inorganic NPs with small particle sizes distribution capable of industrial effluents treatments. In the long run these UF-PES-ENMs are expected to be a practical solution in the industrial effluents treatment, thus an environmental remedy for water pollution. The proposed UF PES-based ENMs can lead to diverse practical applications and thus open up new directions for in-depth studies into this field.

1.6 Thesis Outline

The present thesis consists of five chapters, and they organized as follows:

Chapter 1 presents a brief background of the current research with its rationale and the need to conduct this work. The research gap (problem statement), objectives, scope and significance of this study are outlined.

Chapter 2 provides a critical and comprehensive overview of relevant literature. Literature on the development of various nanofiber membranes for oily wastewater treatment is analysed. The available electrospinning technique for fabricating the nanofiber membranes of oil removing or water removing types with superwetting surfaces is discussed. The recent progress on the oil-water filtration by ENMs incorporated with advanced nanomaterial for enhancing membrane performance is highlighted.

Chapter 3 underscores the detail research methodology to accomplish the proposed research objectives. The techniques used for sample preparations, material acquisition, testing the oil removal capacity of prepared UF-PES-ENMs are all explained. The principles behind all analytical techniques used for the sample characterizations are described.

Chapter 4 presents the results, analyses and detail discussions on the obtained results. The optimization of the prepared ENMs and production of the nanomaterial incorporated ENMs together with the influence of various electrospinning parameters on the membrane properties and performance are discussed. The results of the characterization determined some distinct properties and performance of the proposed UF-ENMs are underscored.

Chapter 5 concludes the thesis by showing the accomplishments of the proposed objectives. This research opened up many new avenues that are worth looking at in the future. It ends with the further outlook demonstrating the further need of research in this field. In short, some environmental friendly nanomaterial based superwetting superhydrophilic-superoleophobic membrane surfaces were fabricated for sustainable applications, especially for separating high-viscous oil-water mixtures from industries that commonly cause environmental pollution.

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LIST OF PUBLICATIONS

Journals

i. **Al-Husaini, I**., Yusoff, A., Lau, W., Ismail, A., Al-Abri, M., Al-Ghafri, B. and Wirzal, M. (2019). Fabrication of polyethersulfone electrospun nanofibrous membranes incorporated with hydrous manganese dioxide for enhanced ultrafiltration of oily solution. *Separation and Purification Technology*, 212, 205-214. **(Q1, IF: 5.107)**

ii. **Al-Husaini, I.S**., Yusoff, A.R.M., Lau, W.J., Ismail, A.F., Al-Abri, M., Z., and Wirzal, M.D.H. (2019). Iron oxide nanoparticles incorporated polyethersulfone electrospun nanofibrous membranes for effective oil removal. *Chemical Engineering Research and Design,* 148, 142-154. **(Q2, IF: 3.073)**

Chapter Book

i. **Issa Sulaiman Al-Husaini,** Abdull Rahim Mohd Yusof, Woei-Jye Lau, Ahmad Fauzi Ismail, Mohammed Al-Abri. (2019). *Nanofiber Membranes for Oily Wastewater Treatment. Nanofiber Membranes for Medical, Environmental, and Energy Applications, CRC Press, 87-102.*

Conference Proceedings

i. *Produced* Water Conference, Oman Water Society (OWS) in partnership with Produced Water Society Middle East and Petroleum Development Oman (PDO), Oman, Muscat, 23-24 October 2019, http://producedwatermiddleeast.com.