POLYVINYLIDENE FLUORIDE INCORPORATED WITH TITANIA-ZIRCONIA DUAL LAYER HOLLOW FIBER PHOTOCATALYTIC MEMBRANE FOR OILY WASTEWATER TREATMENT

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DEDICATION

This thesis is dedicated to my husband and children, who taught me that sacrifice is the key to my life success, to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake and to my late mother, who taught me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

The need for a more effective oily wastewater treatment is necessary to minimize oil and grease content in the wastewater and produce maximum amount of treated water that is suitable to be discharged into open water course. Even though degradation of oily wastewater was found to be promising through photocatalysis process, excellent performance can be achieved through the combination of photocatalysis with membrane separation process. In this study, zirconium dioxide (ZrO_2) was combined with titanium dioxide (TiO_2) to improve the specific surface area of TiO_2. The TiO_2-ZrO_2 hybrid photocatalysts were then embedded in the outer layer of the polyvinylidene fluoride (PVDF) dual layer hollow fiber (DLHF) membrane to produce a photocatalytic membrane for oily wastewater treatment. In the first stage of the study, the coupling of ZrO_2 content from 1 to 20% into TiO_2 was designed to enhance the oily wastewater adsorption capacity and the photodegradation performance. The 1% TiO_2-ZrO_2 hybrid photocatalysts synthesized in this study revealed a higher (second highest) specific surface area of 136.7 m^2/g in comparison to single TiO_2 (39.9 m^2/g). This characteristic is desired as it can boost the photocatalytic activity. The hybrid photocatalysts also displayed reduced optical band gap energy which is desirable as it allows better absorption of photons to excite the electrons into the valence band. The second stage of the study involved fabrication of PVDF DLHF membrane embedded with 1 wt.% of TiO_2-ZrO_2 hybrid photocatalysts in the outer layer of the membrane (DL-ZT1). The fabricated membrane was optimized in terms of air gap from 5 cm to 50 cm. The membrane spun at lower air gap of 5 cm showed the formation of long finger-like structure around 65.0 ± 3.3 μm in length on the outer layer of the membrane due to the immediate phase inversion on the outer side of the fiber. Cross-sectional image of the membrane showed that the membrane is free from delamination which indicated mutual diffusion of polymer during co-extrusion. The membrane displayed lowest contact angle of 71.70° ± 2.58°. The low contact angle was attributed to the low air gap of 5 cm that promoted the growing of microvoids on the outer layer. Under crossflow filtration condition, the membrane also demonstrated highest water and oily wastewater permeation flux as well as oil rejection percentage of 85.4% without UV light irradiation. In the third stage of the study, the membrane was optimized in terms of photocatalysts loading from 0 to 1 wt.% in the outer layer dope composition. The photocatalytic activity of the membrane was investigated using the submerged membrane photoreactor (sMPR) at oily wastewater concentration of 1000 and 10,000 ppm. At 1000 ppm concentration, DL-ZT1 was found to have initial oily wastewater permeation flux of 97.71 $L/m^2.h$ without UV light irradiation and the flux increased to 321.62 $L/m^2.h$ under UV light irradiation. As a result, DL-ZT1 recorded total organic carbon (TOC) degradation of 91.8%. Despite showing reduced TOC degradation at higher oily wastewater concentration of 10,000 ppm, DL-ZT1 recorded oil rejection percentage of 96%. DL-ZT1 exhibited a great potential of photocatalytic membrane for oily wastewater treatment. In comparison to single layer hollow fiber membranes, the DLHF membranes has better performance due to the embedded nanomaterials localized on the outer layer and which made possible reduction in membrane fouling.

ABSTRAK

Keperluan rawatan air sisa berminyak yang efektif adalah penting bagi mengurangkan kandungan minyak dan gris di dalam air sisa dan menghasilkan air terawat dalam jumlah yang maksimum dan sesuai untuk dilepaskan ke sumber air terbuka. Walaupun proses foto-pemangkinan telah menunjukkan kebolehan untuk mendegradasi air sisa berminyak, akan tetapi prestasi yang lebih baik boleh dicapai dengan menggabungkan proses foto-mangkin dengan proses pemisahan menggunakan membran. Dalam kajian ini, zirkonium dioksida (ZrO_2) telah digabung dengan titanium dioksida (TiO_2) untuk meningkatkan luas permukaan tertentu TiO_2. Foto mangkin hibrid TiO_2-ZrO_2 kemudiannya dimasukkan ke dalam lapisan luar gentian geronggang dwi-lapisan (DLHF) polivinilidena florida (PVDF) untuk menghasilkan membran foto-pemangkin bagi merawat air sisa berminyak. Pada peringkat pertama kajian, penambahan kandungan ZrO_2 sebanyak 1 sehingga 20% ke dalam TiO_2 telah dibentuk bagi meningkatkan keupayaan penjerapan air sisa berminyak dan prestasi fotodegradasi. Didapati bahawa foto mangkin hibrid TiO_2-ZrO_2 yang disintesis dengan 1% mempunyai luas permukaan tertentu yang lebih tinggi (kedua tertinggi) sebanyak 136.7 m^2/g berbanding TiO_2 tunggal (39.9 m^2/g). Ciri ini sangat diperlukan kerana ia dapat menggalakkan aktiviti foto-mangkin. Foto mangkin hibrid juga menunjukkan penurunan jurang jalur optikal yang akan meningkatkan penyerapan foton bagi melonjakkan elektron ke jalur valensi. Peringkat kedua kajian merangkumi pembuatan membran PVDF DLHF yang digabung dengan foto mangkin hibrid TiO_2-ZrO_2 sebanyak 1 wt.% pada lapisan luar membran dwi-lapisan (DL-ZT1). Pembuatan membran telah dioptimumkan dari segi kesan ruang udara daripada 5 cm sehingga 50 cm. Membran yang dipintal pada ruang udara terendah iaitu 5 cm menunjukkan pembentukan struktur jejari yang panjang sekitar 65.0 ± 3.3 µm pada lapisan luar membran yang disebabkan oleh penyongsangan fasa secara serta merta pada bahagian luar gentian. Imej keratan rentas membran menunjukkan bahawa membran adalah bebas daripada delaminasi yang menandakan gabungan polimer yang baik berlaku sewaktu penyemperitan bersama. Membran juga menunjukkan sudut kontak terkecil iaitu 71.70° ± 2.58°. Sudut kontak yang kecil ini disebabkan oleh kesan ruang udara terendah iaitu 5 cm yang menggalakkan tumbesaran liang mikro pada lapisan luar. Menerusi penurasan aliran silang, membran yang terhasil berjaya mencatatkan ketelapan fluks tertinggi bagi kedua-dua air dan juga air sisa berminyak selain menunjukkan peratusan minyak tersingkir sebanyak 85.4% tanpa penyinaran sinar UV. Pada peringkat ketiga kajian, membran dioptimumkan dari segi bebanan foto mangkin daripada 0 sehingga 1 wt.% di dalam komposisi larutan lapisan luar. Aktiviti foto-mangkin oleh membran yang dikaji menggunakan reaktor foto-mangkin membran tenggelam (sMPR) dengan air sisa berminyak pada kepekatan 1000 dan 10,000 ppm. Pada kepekatan 1000 ppm, DL-ZT1 mencatatkan ketelapan fluks air sisa berminyak sebanyak 97.71 $L/m^2.h$ tanpa penyinaran sinar UV dan fluks meningkat kepada 321.62 $L/m^2.h$ di bawah penyinaran sinar UV. Akibatnya, DL-ZT1 merekodkan pendegradasian jumlah karbon organik (TOC) sebanyak 91.8%. Di sebalik penurunan pendegradasian TOC pada kepekatan air sisa tinggi 10,000 ppm, DL-ZT1 mencatatkan peratusan minyak tersingkir sebanyak 96%. DL-ZT1 mempamerkan potensi yang sangat besar sebagai membran foto-pemangkin bagi tujuan rawatan air sisa berminyak. Prestasi membran DLHF didapati lebih baik berbanding membran gentian geronggang satu lapisan kerana bahan nano yang digabung berpusat pada permukaan luar membran dan dapat mengurangkan kotoran pada membran.

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LIST OF ABBREVIATIONS

3DOM	-	Three-dimensionally ordered microporous
A/D	-	Acceptor/Donor
AFM	-	Atomic force microscopy
Al_2O_3	-	Aluminum oxide
API	-	American Petroleum Institute
AO	-	Ammonium oxalate
AO7	-	Acid Orange 7
AOP	-	Advanced oxidation process
AR1	-	Acid red 1
ASTM	-	American Society for Testing and Materials
AT-POME	-	Aerobic treated palm oil mill effluent
BET	-	Brunauer-Emmett-Teller
BJH	-	Barrett-Joyner-Halenda
BPA	-	Bisphenol-A
BQ	-	1,4-benzoquinone
BSA	-	Bovine serum albumin
C_3N_4	-	Carbon nitride
CA	-	Cellulose acetate
$CaCO_3$	-	Calcium carbonate
CB	-	Conduction band
CBZ	-	Carbamazepine
CR	-	Congo Red
CTA	-	Cellulose triacetate
CuBTc	-	Copper benzene-1,3,5-tricarboxylate
DLHF	-	Dual layer hollow fiber
DLS	-	Dynamic light scattering
DMAc	-	N, N-Dimethylacetamide
DMF	-	N, N-Dimethylformamide
DMSO	-	Dimethylsulfoxide

DP	-	Diphenhydramine
EDX	-	Energy dispersive X-ray
EPS	-	Extracellular polymeric substances
FA	-	Fulvic acid
$FeCl_3$	-	Ferric chloride
Fe_2O_3	-	Ferric oxide
Fe_3O_4	-	Iron oxide
FESEM	-	Field emission scanning electron microscopy
FS	-	Flat sheet
FTIR	-	Fourier transform infrared
GO	-	Graphene oxide
HA	-	Humic acid
HAO	-	Hydrous aluminum oxide
HF	-	Hollow fiber
HFO	-	Hydrous ferric dioxide
HMO	-	Hydrous manganese
HNT	-	Halloysite nanotube
IMO	-	International Maritime Organization
IPA	-	Isopropyl alcohol
KBr	-	Potassium bromide
La_2O_3	-	Lanthanum oxide
LiCl	-	Lithium chloride
MARPOL	-	International Convention for the Prevention of Marine Pollution
MB	-	Methylene blue
MCU	-	Melamine, cyanuric acid and urea
MF	-	Microfiltration
MMM	-	Mixed matrix membrane
MNA	-	Methylnicotinamide chloride
MO	-	Methyl Orange
MoS_2	-	Molybdenum disulphide
MWCNT	-	Multi-walled carbon nanotubes
NA	-	Not available

NF - Nanofiltration
NHE - Normal hydrogen electrode
NIR - Near infrared
NMP - N-methyl pyrrolidone
NOM - Natural organic matter
NOx - Nitrogen oxide
P4VP - Poly-4-vinyl pyridine
PAA - Phenoxyacetic acid
PAI - Polyamide imide
PAN - Polyacrylonitrile
PANCMI - Polyacrylonitrile-co-maleimide
PEGMA - Poly(ethylene glycol) methacrylate
PEI - Polyetherimide
PES - Polyethersulfone
PF127 - Pluronic F127
PL - Photoluminescence
PMMA - Poly(methyl methacrylate)
PMR - Photocatalytic membrane reactor
PNP - *p*-nitrophenol
POSS - Polyhedral oligomeric silsesquioxane
PP - Polypropylene
PPEES - Poly(1,4-phenylene ether ether sulfone)
ppm - Part per million
PPSU - Polyphenylsulfone
PS - Polystyrene
PS4VP - Poly(styrene-b-4-vinylpyridine)
PSf - Polysulfone
PV - Pervaporation
PVC - Polyvinyl chloride
PVDF - Polyvinylidene fluoride
PVP - Polyvinylpyrrolidone
RB5 - Reactive Black 5

rGO	-	Reduced graphene oxide
RhB	-	Rhodamine B
RO	-	Reverse osmosis
rpm	-	Rotation per minute
SDBS	-	Sodium dodecylbenzenesulfonate
SDG	-	Sustainable Development Goal
SEM	-	Scanning electron microscopy
SEOM	-	Secondary effluent organic matter
SiO_2	-	Silicon dioxide
SLHF	-	Single layer hollow fiber
sMPR	-	Submerged membrane photoreactor
SnO_2	-	Stannic oxide
SOM	-	Seawater organic matter
SOW	-	Synthetic oily wastewater
sPPSU	-	Sulfonated polyphenylsulfone
TEM	-	Transmission electron microscopy
THM	-	Trihalomethane
TiO_2	-	Titanium dioxide or titania
TMP	-	Transmembrane pressure
TNT	-	Titanate nanotubes
TOC	-	Total organic carbon
TOG	-	Total oil and grease
TPZ	-	Tetrapropyl zirconate
TroCs	-	Trace organic contaminants
TTIP	-	Titanium tetraisopropoxide
UATR	-	Universal attenuated total reflectance
UF	-	Ultrafiltration
UV	-	Ultraviolet
UV-A	-	Ultraviolet-A
UV-Vis	-	Ultraviolet-visible
VB	-	Valence band
XPS	-	X-ray photoelectron spectroscopy

XRD - X-ray diffraction
ZIO - Zinc-iron oxide
ZnO - Zinc oxide
ZnS - Zinc sulfate
ZrO_2 - Zirconium dioxide or zirconia

LIST OF SYMBOLS

A - Effective area of the membrane in m^2
Å - Angstrom
A - Absorption coefficient
c - Speed of light in m/s
C - Reactant concentration in mg/L
C_e - Equilibrium concentration of oily wastewater in mg/L
C_F - Feed concentration in ppm
C_o - Initial concentrations of oily wastewater in mg/L
C_P - Permeate concentration in ppm
C_t - Oily wastewater concentration at time t in mg/L
$\mathrm{D_{BET}}$ - Average particle diameter in nm
e - Charge of electron
E - Energy
$\mathrm{E_{CB}}$ - Conduction band edge potential in eV
$\mathrm{E_g}$ - Optical band gap energy in eV
$\mathrm{E_{VB}}$ - Valence band edge potential in eV
ε - Membrane porosity
F(R) - Kubelka-Munk function
hv - Photon energy
$\mathrm{h^+}$ - Hole
J_{w_1} - Water permeation flux in L/m^2.h
J_{w_2} - Recalculated water permeation flux in L/m^2.h
k - Energy independent constant
$\mathrm{k_{aap}}$ - Apparent rate constant in min^{-1}
$\mathrm{k_f}$ - Reaction rate constant in mg/L.min
$\mathrm{K_{ad}}$ - Adsorption constant in L/mg
Kα - K-alpha
λ - Wavelength in Å
n - Type of transition
$\mathrm{O_2^{-}}\bullet$ - Superoxide radical

$\bullet OH$ - Hydroxyl radical

s - Scattering factor

Q - Amount of permeated water in L

Q_e - Adsorption capacity in mg/g

r - Reaction rate in mg/L.min

R - Raw reflectance data

R - Oil rejection percentage

R_a - Membrane surface roughness in nm

R_{FR} - Flux recovery ratio

R^2 - Coefficient of correlation

ρ - Powder sample density in g/cm^3

ρ_w - Water density in g/cm^3

ρ_p - Polymer density in g/cm^3

t - Time

v - Frequency in s^{-1}

V - Volume of model pollutant solution in L

$\tilde{v}$ - Wavenumber in m^{-1}

W - Weight of photocatalysts in g

w_{dry} - Membrane weight at wet condition

w_{wet} - Membrane weight at dry condition

wt.% - Weight percentage

X - Electronegativity of the semiconductor in eV

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Oily wastewater is characterized as an extremely complex substance, usually consisting of high concentrations of dispersed oil, grease, suspended particles (Sun *et al.*, 2017), fats, hydrocarbons, and parts of petroleum such as diesel oil, gasoline, and kerosene (Jamaly, Giwa and Hasan, 2015). Nowadays, oily wastewaters are produced by many industries such as petrochemical complexes, oil refineries, oil distribution companies, food producers, machining factories, metal manufacturers, leather, and textiles industries (Yu, Han and He, 2017). In the oil and gas industry, oily wastewater is known as produced water; a by-product produced during the extraction of oil and natural gas. Oily wastewater that has been discharged without proper treatment can influence groundwater, saltwater, or drinking water. The percolation of contaminants in the produced water could dissolve into the water resources underneath the earth. Hence, the understanding of the various methods used to treat oily wastewater is needed to lessen the undesirable effects of oily wastewaters (Jamaly, Giwa and Hasan, 2015).

According to the 2011 estimation on oil and gas production quantity in Malaysia (Hock Lee, 2013), the crude oil production at 603,400 barrels/day added to the production of oily wastewaters that constitute of deadly matters for instance phenols, petroleum hydrocarbons, and polyaromatic hydrocarbons. In addition, the high values of oil and grease content as well as chemical and biochemical oxygen demand that present in the oily wastewaters does not comply with the standards set by the regulatory bodies of many countries (Alade *et al.*, 2011). For instance, the acceptable conditions for the discharge of industrial effluent set by the Department of Environment Malaysia under Standard A and B of Environment Quality (Industrial Effluent) Regulations 2009 only allow the industrial effluent with oil and grease content not exceeding 1 and 10 ppm, respectively. During oil exploration and extraction processes, oily wastewater concentration around 4000 to 6000 ppm of oil and grease was produced. Meanwhile, oily wastewater concentrations in the ranges of 100 to 5000 ppm were produced by the metal processing industries comprising of grinding oils, cutting oils, lubrication fluids, and coolant oils in the form of soluble and emulsified oil (Putatunda *et al.*, 2019).

Oily wastewater treatment using various methods which include electrochemical, floatation, coagulation, adsorption, and membrane filtration have been reported (Yu, Han and He, 2017). However, the particle size of oil droplets, which is less than 10 µm in most cases, has limited the role of conventional treatment such as floatation and coagulation methods in treating oily wastewater (Jamshidi Gohari *et al.*, 2014). Besides the high operating cost (Putatunda *et al.*, 2019), floatation and coagulation were reported to be less effective since these methods can only remove dispersed oil and floating oil with oil droplet size in the range of 20 to 150 µm (Nascimbén Santos *et al.*, 2020). Therefore, a technology that can overcome these drawbacks is required. Most researchers agree that a single method might not be able to achieve the industrial effluent standard (Jamaly, Giwa and Hasan, 2015). This is due to the method might not be able to accommodate a high volume of effluent as well as the complex nature of the oily wastewater itself.

Membrane technology has emerged as an eminent technology in oily wastewater treatment due to its high separation efficiency (Saini, Sinha and Dash, 2019), high effluent quality (Cebeci and Gökçek, 2018), and no chemical additive is needed to break the emulsion. Microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and pervaporation (PV) membranes are frequently employed to remove oil-water emulsion (Ahmad, Guria and Mandal, 2018) owing to the high efficacy of the membranes in the removal of oil droplets (Ong, Lau, Goh and Ismail, 2014). However, the high fouling propensity resulted from oil adsorption and deposition on the surface of these polymeric membranes has hindered the membrane filtration performance (Lay, Wang and Chew, 2021) hence limited their long-term usage (Jamshidi Gohari *et al.*, 2014). Many researchers devoted their research to investigate ways to boost the antifouling properties of polymeric membranes like polyvinylidene fluoride (PVDF), polysulfone (PSf), polyehersulfone (PES), and cellulose acetate (CA). Modification of polymer membrane surface through various strategies have been accomplished to tailor the surface hydrophilicity, charge, and roughness of the membrane to mitigate the effects of fouling. The incorporation of nanomaterials such as hydrophilic titanium dioxide or titania (TiO_2) (Ong, Lau, Goh, Ng and Ismail, 2014; Mishra *et al.*, 2014; Moghadam *et al.*, 2015), hydrophilic aluminum oxide (Al_2O_3) (Yan *et al.*, 2009), lithium chloride (LiCl)/TiO_2 (Yuliwati and Ismail, 2011), hydrous manganese (HMO) (Jamshidi Gohari *et al.*, 2014), hydrous aluminum oxide (HAO) (Jamshidi Gohari *et al.*, 2015), graphene oxide (GO) (Tang *et al.*, 2015), and silicon oxide (SiO_2)-g-poly(ethylene glycol) methacrylate (PEGMA) (Saini, Sinha and Dash, 2019) into both flat sheet (FS) and hollow fiber (HF) UF membranes to treat oily wastewater has endowed the membrane surface with improved hydrophilicity and good antifouling properties. Ong et al. (2013) observed the deterioration of antifouling property of PVDF-TiO_2 composite HF membrane when it was tested at a high concentration of oily solution of 1000 ppm, however the composite membrane was still promising to treat discharged containing oily solution from industries without having to suffer severe flux decline since the concentration of oil that originates from industrial effluent normally falls in the range of 100 to 450 ppm.

TiO_2 is one of the most studied nanomaterials for membrane modifications (Imran Ali *et al.*, 2018). TiO_2 is not only known for its availability, notable physical and chemical properties, but also for its antifouling potential (Razmjou *et al.*, 2012) as well as its photocatalytic effect (Humayun *et al.*, 2018). The properties of TiO_2 can be further enhanced through metal-ion doping to improve the hydrophilicity of the nanomaterials (Hosseini, Sadeghi and Khazaei, 2018). Most of the polymeric HF membranes embedded with nanomaterials belong to mixed matrix nanocomposite membranes or single layer hollow fiber (SLHF) membranes whereby the inorganic nanomaterials are randomly distributed throughout the polymer matrix (Rezaei *et al.*, 2014; Koutahzadeh, Esfahani and Arce, 2016). The incorporation of inorganic nanomaterials such as metal oxides like TiO_2 resulted in remarkable separation performance but still facing problems related to aging and fouling (Davey, Leak and Patterson, 2016). Lately, dual layer hollow fiber (DLHF) membranes have attracted great interest due to their potential to improve water permeation flux, reduce material cost through the usage of less costly material as the substrate, and improve antifouling performance through the addition of nanomaterials (Shi, Xue, Gao and Zhou, 2016). The presence of embedded TiO_2 nanomaterials in the PVDF outer layer of the DLHF membrane promoted the formation of microporous structure which allowed uniform nanomaterials dispersion (Dzinun *et al.*, 2016).

1.2 Problem Statement

TiO_2 is the most conventionally used photocatalyst in the photodegradation of organic contaminants in water and air due to its excellent oxidative properties and high photocatalytic activity. Based on the literature review, TiO_2-based heterojunction photocatalysts demonstrate desirable characteristics in organic pollutant degradation due to the highly reactive hydroxyl (•OH) radical generated. However, TiO_2 suffers from the recombination of a large amount of the photo-activated electrons and holes. Coupling TiO_2 photocatalysts with other metal oxides can help resolve the electron-hole recombination issue faced by TiO_2. Metal oxide such as zirconium dioxide or zirconia (ZrO_2) offers attractive characteristics like high thermal stability and able to delay phase transformation in TiO_2, which makes ZrO_2 appropriate to be coupled with TiO_2. Coupling lower content of ZrO_2 into TiO_2 can enhance the TiO_2 properties through the production of TiO_2-ZrO_2 hybrid photocatalysts with high specific surface area and boost the photocatalytic activity. Lower photodegradation activity was reported when coupling higher content of ZrO_2 into TiO_2. TiO_2-ZrO_2 was investigated for the oxidation of ethylene (Fu *et al.*, 1996) as well as degradation of 4-chlorophenol (Neppolian *et al.*, 2007), phenol (Kambur, Pozan and Boz, 2012), azo-dye (Pirzada *et al.*, 2015), nitrogen oxide (NOx) (Kim *et al.*, 2010) and rhodamine B (RhB) (Li *et al.*, 2015). So far, no research has been reported on the usage of TiO_2-ZrO_2 for the degradation of oily wastewater. There were some contradictory findings on the optimal ratio of TiO_2 to ZrO_2 in their hybrid, due to the different preparation methods used which led to the non-consistent of the small ZrO_2 content reported (Wang, Patel and Liang, 2018). Therefore, it is necessary to investigate the optimal ratio of TiO_2 to ZrO_2 based on the application explored in this study. The amount of ZrO_2 in the hybrid can significant influence the physicochemical of the resultant TiO_2-ZrO_2 hybrid photocatalysts, which in turn affect the photocatalytic activity.

SLHF membrane is the most widely used HF membrane configuration. However, the emerging of DLHF membranes that offers the usage of a high-performance material or functional material as the outer layer and a relatively cheap material as the inner layer has attracted interest from many researchers to study it (Zhu *et al.*, 2014). Meanwhile, the addition of nanomaterials in the outer dope solution can improve the transport properties in the outer layer of the DLHF membranes (Amaral *et al.*, 2016). Since the membrane structures affects the separation performance of a membrane (Han, Wan and Chung, 2018), the alteration of spinning parameters such as dope flow rate, air gap, coagulation bath temperature, type of bore fluid and coagulation media will affect the membrane structures. Based on literature review, the influence of air gap on membrane morphology and permeation were sometimes found conflicting between membranes with different formulation (Ahmad and Mohd Shafie, 2017). This makes air gap an important factor as the parameter influences the thickness of the outer skin layer and the membrane pore size (Wang *et al.*, 2018) during the fabrication stage. Although extensive research on the effect of air gap on morphology and structure of HF membranes has been carried out, limited efforts have been made to study the effect of air gap in DLHF membranes. The influence of air gap in SLHF and DLHF membranes could result in different outcomes. Therefore, the nanocomposite membranes produced in this study were optimized in terms of their air gap. Air gap has significant influence on the structure and morphology of the PVDF DLHF membranes, especially the outer layer.

The distribution of the nanomaterials is difficult to be controlled as the nanomaterials were randomly distributed within the polymer matrix of an SLHF membranes (Davey, Leak and Patterson, 2016). The modification is seen as a wastage since the nanomaterials are not evenly distributed at the outer surface of the nanocomposite membranes. On the other hand, DLHF membranes has better performance over SLHF membranes due to the embedded nanomaterials were localized on the outer layer and was able to combat the fouling problem better than before. The microporous structure formed in the DLHF membranes helps in the uniform dispersion of the nanomaterials. Based on literature review, immobilizing nanomaterials in the outer layer of DLHF membranes through the production of nanocomposite DLHF membranes can reduce the hydrophobicity of the polymer membrane while the photocatalytic effect of the nanomaterials from the semiconductor nanomaterials type or photocatalysts can assist in the chemical reaction through the reaction of the produced reactive substance with the pollutant to form a less toxic compound. Immobilizing a high load of nanomaterials in the polymer matrix was reported to improve the membrane properties. However, agglomeration of nanomaterials, as well as leaching of nanomaterials at high loading could raise hazardous health risks to the end-user of the treated water (Mahmoudi *et al.*, 2019). Immobilizing a small load of nanomaterials such as TiO_2-ZrO_2 hybrid photocatalysts in the outer layer of the DLHF membranes could provide a solution to those issues. Even though the incorporation of nanomaterials into the membranes was reported to enhance the membrane's performance but the optimal loading depends on the properties of the nanomaterials and the membrane composition (Wen *et al.*, 2019). Therefore, the PVDF DLHF membranes were optimized in terms of the TiO_2-ZrO_2 hybrid photocatalysts loading prior to the photocatalytic testing using a submerged photoreactor under UV light irradiation for oily wastewater treatment. Varying the TiO_2-ZrO_2 hybrid photocatalysts loading into the outer layer of the DLHF membranes will influence the rate of oily wastewater degradation. The main difference between this study with other studies that uses the same TiO_2-ZrO_2 hybrid photocatalyst is that this study embedded the photocatalysts on the membrane outer surface of a PVDF DLHF membrane rather than suspending the photocatalysts in the oily wastewater and so far, has not been investigated yet. Despite the great research works conducted, only a few efforts have been made to develop a technique that is capable to treat oily wastewater without secondary treatment. The nanocomposite membrane can preserve a close interaction between the oily wastewater and the photocatalysts while simplifying the photocatalysts recovery process. The photocatalytic nanocomposite membrane fabricated in this study was expected to enhance photocatalytic properties by suppressing the electron-hole pairs recombination and minimize membrane fouling through the degradation of foulants. This study also provides in-depth information on the performance of DLHF and SLHF membranes embedded with the same hybrid photocatalysts that would affect the photodegradation of oily wastewater.

1.3 Research Objectives

The objectives of the research are as follows:

1. To investigate the effects of ZrO_2 and TiO_2 contents in TiO_2-ZrO_2 hybrid photocatalysts on the photodegradation of oily wastewater.
2. To assess the effect of air gaps on the structures and properties of PVDF/TiO_2-ZrO_2 DLHF photocatalytic nanocomposite membranes applied for oily wastewater treatment.
3. To evaluate the effect of TiO_2-ZrO_2 hybrid photocatalysts loading on the oily wastewater photodegradation performance using a submerged photoreactor under UV light irradiation and to compare the performances of DLHF and SLHF membranes prepared and tested under the same experimental conditions.

1.4 Research Scope

The above-mentioned research objectives are accomplished through the subsequent scopes of studies that have been finalized as follows:

(a) Synthesizing TiO_2-ZrO_2 hybrid nanomaterials with various ZrO_2 content (1, 5, 10, 15, and 20%) in TiO_2 via sol-gel method. The TiO_2-ZrO_2 hybrid photocatalysts with various ZrO_2 content (20, 50, and 80%) were initially prepared and the produced nanomaterials were analyzed using XRD and UV-Vis to determine the inclination of the ZrO_2 content. The preliminary results from XRD and UV-Vis were found to incline towards the lower ZrO_2 content in TiO_2. The findings were used to reduce the design decision and the ZrO_2 content coupled with TiO_2 in this study was limited to 20% and below.

(b) Determining the TiO_2-ZrO_2 hybrid nanomaterials properties such as crystallite sizes and crystal phase using X-ray diffractometer (XRD), specific surface area, pore volume, pore radius, and pore size distribution using Brunauer-Emmett-Teller (BET), particle size using transmission electron microscopy (TEM), surface morphology using field emission scanning electron microscopy (FESEM), reflectance spectra and optical band gap energy (E_g) using ultraviolet-visible (UV-Vis) spectroscopy, phase composition using Fourier transform infrared (FTIR) spectroscopy, chemical composition using X-ray photoelectron spectroscopy (XPS), electron-hole pairs transition using photoluminescene (PL) spectrometer as well as particle size distribution and zeta potential by using dynamic light scattering (DLS).

(c) Conducting adsorption and photocatalytic test using suspended TiO_2-ZrO_2 hybrid photocatalysts of different ZrO_2 content in oily wastewater at 100 ppm for 7 h (2 h to calculate the adsorption capacity (Q_e) of pollutant adsorbed on adsorbate under dark condition and 5 h to determine the total organic carbon (TOC) degradation under UV light irradiation).

(d) Identifying the active species produced by the hybrid photocatalysts during adsorption-photodegradation of oily wastewater at 100 ppm through the radical scavenging experiment for 7 h.

(e) Fabricating DLHF nanocomposite membranes using dry-jet wet phase inversion technique at different air gaps ranged from 5 to 50 cm. The membranes spun from the optimal air gap were embedded with different loading capacities of TiO_2-ZrO_2 hybrid photocatalyst ranging from 0 to 1 wt.%. The photocatalysts loading and air gap were manipulated while the outer and outer and inner dope flow rate, bore fluid flow rate and spinneret geometry used were kept constant to narrow down the scope of the study. The highest photocatalysts loading used to spin the DLHF membranes was fixed at 1 wt.% due to the limitation of the spinneret used which consist of small hole (die-gap) size for the outer dope solution.

(f) Characterizing the DLHF membranes using scanning electron microscopy (SEM) for morphological analysis, energy dispersive X-ray (EDX) for the analysis of nanomaterials dispersion on the membrane outer layer, atomic force microscopic (AFM) for surface roughness analysis, universal attenuated total reflectance (UATR) for molecular structure analysis, contact angle measurement for membrane hydrophilicity analysis and porosity test for membrane porosity analysis.

(g) Measuring the water permeation flux, the oily wastewater permeation flux, the oil rejection efficiency, and the antifouling properties of the prepared membranes for oily wastewater at 1000 and 10,000 ppm. Synthetic cutting oil was used in the preparation of oily wastewater. The filtration and rejection tests were determined for the ideal case only.

(h) Constructing submerged membrane photocatalytic reactor (sMPR) with the dimension of 10 cm (width) x 25 cm (length) x 40 cm (height) equipped with a U-shape DLHF membrane module positioned at the base of sMPR, air compressor, air diffuser, air flow meter, permeate collection tank, peristaltic pump, and UV light.

(i) Measuring the water permeation flux before and after photocatalysis (1 h before and 1 h after), the filtration performance (oily wastewater permeation flux without UV light) for 4 h and the oily wastewater permeation flux under UV light irradiation in sMPR for a reaction time of 9 h (5 h of retention time and 4 h of photodegradation), the oily wastewater degradation and oil rejection percentage for oily wastewater at 1000 and 10,000 ppm.

(j) Determining the performance of DLHF and SLHF membranes that were embedded with the optimized content of TiO_2-ZrO_2 hybrid photocatalyst for the photodegradation of oily wastewater. The water and oily wastewater permeation flux at 1000 and 10,000 ppm were also determined. The membranes were characterized in terms of its elemental composition, morphology, porosity, hydrophilicity, and roughness.

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LIST OF PUBLICATIONS

1. **Nurshahnawal Yaacob**, Pei Sean Goh, Ahmad Fauzi Ismail, Noor Aina Mohd Nazri, Be Cheer Ng, Muhammad Nizam Zainal Abidin, Lukka Thuyavan Yogarathinam (2020). ZrO_2-TiO_2 Incorporated PVDF Dual-Layer Hollow Fiber Membrane for Oily Wastewater Treatment: Effect of Air Gap. *Membranes*, 10, 124 **(Q2, IF: 3.094)**.

2. **Nurshahnawal Yaacob**, Goh Pei Sean, Noor Aina Mohd Nazri, Ahmad Fauzi Ismail, Muhammad Nidzhom Zainol Abidin, Mahesan Naidu Subramaniam (2021). Simultaneous Oily Wastewater Adsorption and Photodegradation by ZrO_2-TiO_2 Heterojunction Photocatalysts. *Journal of Water Process Engineering*, 29, 101644 **(Q1, IF: 3.465)**.

3. **Nurshahnawal Yaacob**, Goh Pei Sean, Noor Aina Mohd Nazri, Ahmad Fauzi Ismail (2018). Structural and Potential Photocatalytic Properties of ZrO_2 co-doped TiO_2 Photocatalysts. In *National Congress on Membrane Technology.2018 (NATCOM 2018)*. 30th - 31st October 2018. Pulai Springs Resort, Johor Bahru, Malaysia.

4. **Nurshahnawal Yaacob**, Ahmad Fauzi Ismail, Goh Pei Sean, Noor Aina Mohd Nazri (2019). Performance of Combined Action of Oily Wastewater Adsorption and Photodegradation by ZrO_2-TiO_2 Heterojunction Photocatalysts. In *International Conference of Sustainable Environmental Technology (ISET 2019)*. 20th - 22nd August 2019. DoubleTree by Hilton Hotel, Johor Bahru, Malaysia, Malaysia.