PHOTOCATALYTIC DUAL LAYER HOLLOW FIBER MEMBRANE FOR COLOUR PIGMENT DEGRADATION OF AEROBICALLY TREATED PALM OIL MILL EFFLUENT

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DEDICATION

This thesis is dedicated to my father, Mr. Subramaniam, who taught me that I am the master of my own destiny, and my mother in heaven, Mrs. Kumari, who taught me to be kind to the people around me. It is also dedicated to my siblings, Sharmila, Prabagaran and Gowri, who supported me through the travails of life in every single way.

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ABSTRACT

Large amount of wastewater is produced during the processing of palm oil. The final form of effluent is aerobically-treated palm oil mill effluent (AT-POME), an odourless and oil-free brown solution. The brown pigments found in AT-POME pose high risk to eutrophication and water contamination if they are released into natural water bodies without further treatment. Current treatment methods to remove colour pigments such as nanofiltration face membrane fouling due to pore blockage. Therefore, a more efficient method to remove pigments from AT-POME has to be developed. Hence, this study focused on the fabrication of a photocatalytic dual layer ultrafiltration hollow fiber membrane for colour removal from AT-POME. Novel boron doped titania nanotubes (TNT-B) photocatalyst was incorporated on the outer layer of a polyvinylidene fluoride (PVDF) dual layer hollow fiber membrane (DLHFM). The molarity of boron doped into TNT was manipulated between the 0.25 M (TNT-B0.25), 0.5 M (TNT-B0.5) and 1.0 M (TNT-B1.0). TNT-B was prepared via a two-step hydrothermal method. The physicochemical properties of prepared TNT-B were characterized. The prepared photocatalyst were also tested for their photocatalytic activity under visible light irradiation for the photodegradation of lignin and tannic acid (TA), which are the two important constituents of AT-POME. TNT-B0.5 exhibited the best photocatalytic activity, where it was able to degrade lignin and TA up to 96.47% and 96.91%, respectively. High surface area (159.552 m^2/g) and visible light absorption have contributed to remarkably enhanced photocatalytic activity. TNT-B0.5 was then used as the photocatalyst to prepare PVDF DLHFM at a loading of 1 wt% the outer layer of membrane. The membranes were spun using a triple orifice spinneret, while three important spinning parameters, i.e. bore fluid flow rate (BFFR), outer dope solution flow rate (OLFR), air gap (AG) were manipulated to study the effect of these parameters on membrane characteristics and filtration performance. All membranes were analysed to understand their morphology and physicochemical properties. It was deduced that a BFFR of 3 ml/min, OLFR of 3 ml/min and AG of 10 cm were the optimum spinning conditions to prepare DLHFM with high flux and high rejection. The optimised membrane was then loaded with different photocatalyst loadings (1 wt%, 2 wt%, 3 wt%) on the outer layer. Then, the effect of different loading towards dynamic photocatalytic filtration and antifouling properties using synthetic AT-POME as model pollutant in a submerged membrane photo reactor (SMPR) was evaluated. With 2 wt% TNT-B0.5, the optimised DLHFM exhibited the highest flux of 51.29 L/m^2h and rejection of 79.42% when tested with synthetic AT-POME. The membrane also exhibited superior antifouling properties in which the flux was recovered by 95% after four filtration cycles of synthetic AT-POME. The optimised membrane was then used to treat real AT-POME for 20 days. The results showed that an increase in both flux and rejection over treatment time which was due to the synergistic effect of photocatalysis and membrane filtration.

ABSTRAK

Sejumlah besar air sisa dihasilkan semasa pemprosesan minyak kelapa sawit. Bentuk terakhir efluen yang dihasilkan ialah efluen kilang minyak kelapa sawit dirawat secara aerobik (AT-POME), iaitu larutan berwarna perang tanpa bau dan bebas minyak. Pigmen perang yang terdapat dalam AT-POME menimbulkan risiko tinggi seperti eutrofikasi dan pencemaran air jika ia dibebaskan ke dalam sumber air semula jadi tanpa rawatan lanjut. Kaedah-kaedah rawatan terkini bagi menyingkirkan pigmen warna seperti nanofiltrasi mengalami masalah pengotoran membran kerana penyumbatan liang. Oleh yang demikian, satu kaedah yang lebih cekap untuk menyingkirkan pigmen daripada AT-POME harus dibangunkan. Oleh itu, kajian ini memberi tumpuan kepada penghasilan membran ultrafiltrasi pemangkin foto gentian rongga dwi lapisan untuk penyingkiran pigmen daripada AT-POME. Pemangkin foto titania nanotiub bercampur boron baharu (TNT-B) telah digabungkan pada lapisan luar membran gentian rongga dwi lapisan polivinilidin florida (PVDF). Molariti boron yang dicampurkan dengan TNT dimanipulasi pada 0.25 M (TNT-B0.25), 0.5 M (TNT-B0.5) dan 1.0 M (TNT-B1.0). TNT-B telah disediakan melalui kaedah hidrotermal dua langkah. Sifat-sifat fizikokimia TNT-B yang disediakan telah dicirikan. Pemangkin foto yang disediakan juga telah diuji untuk mengenalpasti aktiviti pemangkinan foto di bawah sinaran cahaya bagi merawat lignin dan asid tanik (TA), iaitu dua komponen penting dalam AT-POME. TNT-B0.5 mempamerkan aktiviti pemangkinan foto terbaik, di mana ia dapat menghapuskan lignin dan TA sehingga 96.47% dan 96.91%. Kawasan dengan permukaan tinggi (159.552 m^2/g) serta penyerapan cahaya yang lebih baik telah mempertingkatkan aktiviti pemangkinan foto TNT-B0.5. TNT-B0.5 kemudiannya digunakan sebagai pemangkin foto untuk penyediaan PVDF DLHFM pada pemuatan 1 wt% pada lapisan luar membran. Membran telah dihasilkan menggunakan pemutar tiga orifis. Tiga parameter proses putaran penting, iaitu kadar aliran cecair penebuk (BFFR), kadar campuran polimer lapisan luar (OLFR) dan sela udara (AG) telah dimanipulasi untuk mengkaji kesan parameter terhadap ciri-ciri membran dan prestasi penolakan. Semua membran dianalisa untuk memahami sifat morfologi dan fizikokimia. BFFR 3 ml/min, OLFR 3 ml/min dan AG 10 cm merupakan keperluan proses putaran optimum untuk penyediaan DLHFM dengan fluks dan kadar penolakan yang tinggi. Membran yang dioptimumkan kemudian dimasukkan dengan kandungan pemangkin foto yang berbeza (1 wt%, 2 wt%, 3 wt%) pada lapisan luar membran dwi lapisan. Kemudian, kesan pemuatan yang berbeza terhadap penapisan pemangkinan foto dinamik dan sifat anticemar menggunakan AT-POME sintetik sebagai bahan cemar dalam reaktor foto membran tenggelam (SMPR) telah dinilai. DLHFM dengan 2 wt% TNT-B0.5, DLHFM menunjukkan aliran tertinggi sebanyak 51.29 L/m^2h dan penolakan sebanyak 79.42% apabila diuji dengan AT-POME sintetik. Membran juga mempamerkan sifat-sifat anti-pengotoran yang unggul di mana pemulihan aliran sebanyak 95% selepas empat kitaran penapisan AT-POME sintetik. Membran yang dioptimumkan kemudiannya digunakan untuk merawat AT-POME sebenar selama 20 hari. Hasilnya menunjukkan peningkatan dalam kedua-dua aliran dan penolakan dengan masa rawatan yang disebabkan oleh kesan sinergistik pemangkin foto dan penapisan membran.

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LIST OF ABBREVIATIONS

ADMI	-	American Dye Manufacturers Institute
AFM	-	Atomic force microscopy
AG	-	Air gap
AT-POME	-	Aerobically-treated palm oil mill effluent
BET	-	Brunauer-Emmett-Teller
BFFR	-	Bore fluid flow rate
BJH	-	Barrett-Joyner-Halenda
BOD	-	Biochemical oxygen demand
CB	-	Conduction band
CNT	-	Carbon nanotubes
COD	-	Chemical oxygen demand
CR	-	Congo red
CVD	-	Chemical vapor deposition
DER	-	Dope extrusion rate
DLHFM	-	Dual layer hollow fiber membrane
DOE	-	Department of environmental
DSFR	-	Dope solution flow rate
EDC	-	Endocrine disrupting chemical
EDX	-	Energy-dispersive x-ray spectroscopy
EFB	-	Empty fruit bunch
FESEM	-	Field emission scanning electron microscopy
FFB	-	Fresh fruit bunch
FTIR	-	Fourier transform infrared
GO	-	Graphene oxide
HA	-	Humic acid
HCBD	-	hexachlorobutadiene
HCH	-	Hexachlorocyclohexane
HPLC	-	High performance liquid chromatography
HRT	-	Hydraulic retention time
LPCVD	-	Low pressure chemical vapor deposition

MB	-	Methylene Blue
MF	-	Microfiltration
MO	-	Methylene Orange
MWCO	-	Molecular weight cut off
NF	-	Nanofiltration
NMP	-	N-methyl-2-pyrrolidone
OD	-	Outer diameter
OLFR	-	Outer layer flow rate
PCB	-	poly-chlorinated biphenyls
PES	-	Polyether sulfone
PKS	-	Palm kernel shell
PL	-	Photoluminescence
POME	-	Palm oil mill effluent
POP	-	Persistent organic pollutant
PPF	-	Palm pressed fiber
PSA	-	Particle size analyser
PSf	-	Polysulfone
PVDF	-	Polyvinylidene fluoride
PVP	-	Polyvinylpyrrolidone
RhB	-	Rhodamine B
RO	-	Reverse osmosis
SEM	-	Scanning electron microscopy
SMPR	-	Submerged membrane photo reactor
TEM	-	Transmission electron microscopy
TNT-B	-	Titania nanotube-boron
TOC	-	Total organic compound
TSS	-	Total suspended solids
TTIP	-	Titanium isopropoxide
UF	-	Ultrafiltration
UV	-	Ultraviolet
UV-Vis NIR	-	Ultraviolet-visible near infrared spectroscopy
VB	-	Valence band
XRD	-	X-ray diffraction

LIST OF SYMBOLS

d	-	Distance between layers
λ	-	Incident wavelength
θ	-	Diffraction angle
E_g	-	Band gap energy
h	-	Planck’s constant
c	-	Speed of light
λ_{red}	-	Red edge of adsorption
C_r	-	Reduction percentage
C_o	-	Initial absorbance
C_i	-	Final absorbance
q_e	-	Amount absorbed at equilibrium
q_t	-	Amount absorbed at given time
t	-	time
k_1	-	Rate constant of pseudo-first-order model
k_2	-	Rate constant for pseudo-second-order model
k_{app}	-	pseudo-first-order rate constant
ε	-	Membrane porosity
W_0	-	Weight of wet membrane
W_1	-	Weight of dry membrane
V	-	Volume
J_w	-	Pure water flux
A	-	Membrane effective area
Δt	-	Permeation time
R	-	Rejection
R_F	-	Removal efficiency
A_f	-	Absorbance value of feed
A_p	-	Absorbance value of permeate
F_R	-	Flux recovery
F_i	-	Initial flux

F_w	-	Final flux
R_R	-	Rejection recovery
R_i	-	Initial rejection
R_w	-	Rejection recovery

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CHAPTER 1

INTRODUCTION

1.1 Problem Background

Water has been the most important resource for mankind livelihood, with water being involved in many facets of daily routine of humans. Malaysia has been blessed with an abundance of fresh water which can be readily used for our daily routine and for the many industries while many countries such as United Arab Emirates, Oman, Japan and Australia are heavily relying on expensive processes such as desalination to procure clean water (Dawoud and Mulla, 2012). One prominent industry in South East Asia, especially in Malaysia and Indonesia which rely on fresh water for its processes is the palm oil industry. After Indonesia, Malaysia is the second largest producers and exporters of palm oil in the world with production volume of up to 17,500 metric tonnes (Otieno *et al.*, 2016). Furthermore, it has been expected that the production of global palm oil is to increase up to 140% (Otieno *et al.*, 2016). With the projected increase in palm oil production, the consumption of freshwater to sustain the higher volume of production is expected to increase too.

Findings have shown that approximately 1.75×10^{10} L of fresh water is used per year for the production of every tonne of palm oil in Malaysia (Shankar *et al.*, 2013). With this large water footprint, the palm oil production industry produces large amount of effluents known as palm oil mill effluent (POME). POME is characterised as an acidic, brownish and oily by-product of the palm oil industry (Poh *et al.*, 2010). POME constitutes to 96-97% water, made up with 0.6-0.7% oil and 4-5% of total solid (Azmi *et al.*, 2014). Real POME is highly polluted, with chemical oxygen demand (COD) and biochemical oxygen demand (BOD) values as high as 50,000 mg/L and 25,000 mg/L respectively (Rupani and Singh, 2010). Hence, releasing POME in its real form would be detrimental to the environment. Issues such as eutrophication (Petrenko *et al.*, 2016), algae bloom and pollution towards fresh water bodies can arise with unchecked release of such polluted wastewater. Hence, several treatment methods have been established by the palm oil industry to reduce these key parameters such as COD, BOD, turbidity, total suspended solids (TSS) and acidity. Currently, the established methods to treat raw POME includes ponding (Liew *et al.*, 2014), aerobic digestion (Tan *et al.*, 2014), physicochemical (Liew *et al.*, 2014) treatment and anaerobic digestion (Chou *et al.*, 2016). Among these, the ponding system and aerobic digestion methods have been combined and used prevalently by palm oil mills. With the current method, a secondary effluent, namely aerobically treated palm oil mill effluent (AT-POME) is produced. AT-POME is known to be less polluted compared to raw POME, with COD and BOD values amounting to 537 mg/L and 22 mg/L respectively and total organic carbon (TOC) values at 724 ppm (Tan *et al.*, 2014).

Even though these values meet the guidelines set by the Malaysia Department of Environmental (DOE), AT-POME exhibits a distinguished and intense brown colour. The brown colour can be attributed to the presence of various lignin and tannin complexes which has been broken down from the fragments of palm oil fruit itself (Poh *et al.*, 2010). Tannins and lignins are natural dyes which imparts brown coloration towards plants, especially tree barks (Shankar *et al.*, 2013). They are also found in the pulp and paper industry wastewater. With the presence of these natural dyes, the brown coloured AT-POME would pose significant harm towards the environment if they are discharged into the water bodies without further treatment. The pigments present in AT-POME can absorb and hinder light travelling through it, which can become an problem if this effluent is released into the environment. AT-POME can also increase the turbidity and concentration of pigments in receiving water body, which in turn, may hinder the passage of light to the bottom of the water body, such as rivers and lakes. The absence of sunlight at the bottom of water bodies may hinder photosynthesis carried out by underwater plants, which will subsequently reduce the amount of dissolved oxygen in the said water body. This phenomenon will accelerate the collapse of existing aquatic system.

Hence, to overcome this problem, a polishing mechanism is required to degrade or remove the natural pigments that are present in AT-POME. This would reduce its impact towards surrounding water bodies and allows its reusability, subsequently reducing freshwater consumption by the palm oil industry. Membrane technology has emerged as a very versatile technique for wastewater treatment (Yin and Deng, 2015). This technology has been extensively utilised to treat textile wastewater, which contains synthetic dyes created to colour clothing (Yin *et al.*, 2017). Membrane separation can separate synthetic dyes from textile effluent, hence reduce its negative impacts towards the environment. However, this creates a secondary pollutant as the pollutants are only removed physically from water, but not destroyed. An additional treatment step will be required to effectively remove or process this pollutant. Hence, the employment of photocatalytic membrane, which offers dual functionality of separating pollutants from water and destroy the pollutants concurrently via oxidative processes becomes an attractive alternative.

Photocatalytic membranes are polymeric or ceramic membranes that are embedded with photocatalyst. These catalysts are incorporated into the membrane matrix to produce strong oxidising species when in contact with water and photon sources and simultaneously oxidise and degrade any organic pollutant. Due to the dual functionalities, photocatalytic membrane can perform both filtration and organic matter degradation simultaneously. The incorporation of photocatalyst into membrane matrix can provide a synergistic effect to enhance water permeability and improve membrane anti-fouling capabilities. Adhesion of foulant on membrane surface can be minimized as the photocatalyst embedded in the membrane are able to degrade foulants that are built up on the membrane surface. This is important to maintain membrane usability on a long term. In conventional photocatalytic wastewater treatment, secondary treatment is required to separate the suspended used photocatalyst. Through the development of photocatalytic membranes, the immobilized photocatalyst avoids the necessity of secondary treatment and the risk of pollution.

Commonly, photocatalytic membranes are placed in a submerged membrane photo reactor (SMPR), which is an integrated system consists of light source, membrane modules and aeration system (Molinari *et al.*, 2017). Polymers such as polyether sulfone (PES) (Argurio *et al.*, 2018) and polyvinylidene fluoride (PVDF) (Dzinun *et al.*, 2015) have been successfully used to fabricate polymeric membranes with photocatalyst incorporated in them. Commercial titanium dioxide (TiO_2) P25

Degussa has been widely used and studied as a model photocatalyst due to its excellent photodegradation performance, high stability and low toxicity (Shi *et al.*, 2013). Recently, titanium nanotube (TNT) which is similar to TiO_2 P25 Degussa in terms of atomic composition but differ in physical and photocatalytic characteristics has been explored (Liu *et al.*, 2014). Owing to the structural properties, TNT exhibits more advantageous features compared to TiO_2, such as higher effective surface area and better photocatalytic degradation performance (Wong *et al.*, 2011). Currently, focus has also been placed in improving the photocatalytic performance of TiO_2-based photocatalysts through various modifications.

1.2 Problem Statement

The brown colour present in AT-POME hinders the reusability of the effluent. One method which can remove pigments present in AT-POME is via nanofiltration (NF) using polymeric membranes. However, the sheer amount of effluent would require a large amount of membrane surface area and pressure to force the feed through, which is energy intensive. Furthermore, AT-POME is known to be rich in organic compounds, which may also contribute towards membrane fouling which may reduce membrane usability and long-term performance. To counter this, a different approach is required, and this is where photocatalysis can play a significant role. Photocatalysis has been touted as a possible solution to degrade the pigments into smaller, less harmful particles and reduce the colour intensity of AT-POME. Commonly, photocatalysis is coupled with membrane technology in an SMPR. In this case, a membrane module is fitted as a separation unit, while photocatalyst is added into the effluent to form a slurry for photodegradation of pollutants present.

However, this method posed two significant issues. Firstly, a large amount of photocatalyst is added in the reactor to form the slurry. This requires an additional step to reclaim the used photocatalyst, which would make this method tedious. In addition, photocatalyst loss during treatment process is also another issue that needs to be considered. To overcome these issues, hybrid photocatalytic membranes is developed. Hybrid photocatalytic membrane is a class of membrane where photocatalysts are incorporated into the matrix of polymeric membrane. Current photocatalytic membranes are fabricated by mixing photocatalyst homogeneously into dope solution, which creates membranes with photocatalyst evenly dispersed throughout the membrane. With the photocatalyst embedded in the membrane matrix, the additional efforts to recycle the suspended photocatalyst employed in conventional (SMPR) can be avoided. Using this mechanism, membranes with photocatalyst embedded have shown good performance to remove oily wastewater (Ong *et. al*., 2014), natural organic matters (Choo *et al*., 2008, Yao *et al.*, 2009) and bacteria (Goei and Lim, 2014).

The second issue faced by conventional SMPR systems is that photocatalysts require constant ultraviolet (UV) light source for activation. Hence, the energy consumption may become a hindrance for its practical usage. To counter this issue, dopants such as carbon (Raza *et al.*, 2015), nitrogen (Nguyen and Bai, 2014), sulphur (Lisovski *et al.*, 2012), nickel (Mohseni-Salehi *et al.*, 2018) iron (Riaz, 2013; Craciun *et al.*, 2018) and graphene oxide (GO) (Song *et al.*, 2015) have been incorporated into photocatalyst to enhance photocatalytic performance under visible light irradiation. Among the dopants that have been explored, boron has shown great promise (Kamal *et al.*, 2019; Su *et al.*, 2019). Boron is a semi metal element, similar to semiconductors such as silicon and germanium which can vastly improve photocatalyst performance (Wang *et al.*, 2016; Zhang *et al.*, 2017).

The major concern during the fabrication of photocatalytic membrane is the difficulty to control the distribution of photocatalyst within the membrane matrix. Photocatalyst that are embedded within the inner parts of membrane matrix may not be activated due to the inability of photons to penetrate through the membrane matrix. Consequently, the photocatalytic efficiency is greatly reduced. The development of dual layer hollow fiber membranes (DLHFM) can potentially address this issue. DLHFM fabricated using a three-orifice spinneret form membrane structure that consists of two distinguished layers. Research conducted using dual layer membranes has shown good performance for forward osmosis (Setiawan *et al.*, 2012), gas separation (Amaral *et al.*, 2015) and photocatalytic degradation of endocrine disruption compounds (EDC) (Dzinun *et al.*, 2015). DLHFM allow greater control towards the physical and chemical properties of selective layer. By having two different functional layers, photocatalyst can be loaded on the outer layer, serving as a host for the photocatalyst and a selective layer, while the inner layer serves as a porous support to provide strength and allow quick permeation through the membrane. The ability to distribute photocatalyst only on the membrane surface allows more catalyst to be activated, hence enhancing photocatalytic efficiency.

Based on current literature, there is a dearth in studies regarding the development of photocatalytic DLHFM. There is also no study conducted on the effect of key spinning parameters towards membrane physical properties and the resultant permeation and rejection performance. Therefore, the main objective of this study is to fabricate a photocatalytic dual layer hollow fiber membrane which is embedded with a visible light responsive boron-doped TNT photocatalyst (TNT-B). TNT-B was synthesized via two-step hydrothermal method at different boron doping molarity, followed with characterisation. Doping at different molarity was performed to investigate its effect towards catalyst characteristics and photocatalytic activity. The photocatalyst was then incorporated into the outer layer dope solution at different loading for the fabrication of dual layer hollow fiber membrane with PVDF as the polymeric material. The dual layer hollow fiber membrane was produced by extruding the dope solutions prepared via a specially designed triple orifice spinneret using conventional dry/wet spinning technique, with water used as both internal and external polymer coagulant. For this, key spinning parameters, such as bore fluid flow rate (BFFR), outer layer dope solution flow rate (OLFR), air gap (AG) and photocatalyst loading were manipulated for optimisation purpose. Next, the membrane which exhibited superior filtration performance was installed into a SMPR to determine its efficiency to treat AT-POME. The membrane permeation, separation, anti-fouling attributes, and membrane recyclability were also evaluated.

1.3 Research Objective

The aim of this study is to develop photocatalytic DLHFM embedded with high performance photocatalyst which can be activated using visible light for AT-POME colour removal. Hence, this study embarked on the following objectives:

1. To create and characterize TNT-B photocatalyst which can perform photocatalytic activities under visible light activation.
2. To develop a series of TNT-B photocatalyst incorporated DLHFM by varying the three hollow fiber spinning parameters.
3. To examine the flux, colour removal efficiency and anti-fouling properties of the resultant PVDF/TNT-B photocatalytic DLHFM using AT-POME as model pollutant.

1.4 Scope of Study

To achieve the above-mentioned objectives, the following scopes of studies have been outlined:

1. Synthesis of TNT via hydrothermal method with a reaction temperature of 180 ºC, reaction time of 24 h and 10M NaOH used as alkali medium for synthesis
2. Doping of TNT with boron via hydrothermal method at different boric acid molarity, i.e. 0.25, 0.5, 1.0 M at 120 ºC for 6 h.
3. Study the properties of TNT-B such as morphology, particle size and charge, functional group changes, crystallinity, band gap, wavelength absorption and surface area using transmission electron microscopy (TEM), particle size analyzer (PSA), Fourier-transform infrared spectroscopy (FTIR), glancing angle x-ray diffraction (GA-XRD), ultraviolet–visible spectroscopy near infrared (UV-Vis NIR), photoluminescence (PL), brunauer–emmett–teller (BET) respectively.
4. Study the adsorption and photocatalytic activity of TNT-B under visible light irradiation using synthetic AT-POME as pollutant at different pH conditions of 4, 7, 10 and different initial concentration of 100 ppm, 200 ppm, 300 ppm, 400 ppm.
5. Preparation of inner dope solutions that consists of PVDF at 18 wt%, PVP at 5 wt% and NMP solvent at 77 wt%. Outer layer dope solutions were prepared with 15 wt% PVDF, 1wt% PVP, 85 wt% NMP and TNT-B loaded at different photocatalyst percentage i.e. 1 wt%, 2 wt% and 3 wt%.

6. Fabrication of photocatalytic DLHFM using a triple orifice spinneret, with water used as its internal and external coagulant at different parameters BFFR at 1 ml/min, 1.5 ml/min, 3 ml/min; outer layer dope flow rate at 3 ml/min, 4.5 ml/min and 6 ml/min; different AG at 5 cm, 10 cm, 15 cm and different photocatalyst loading on outer layer dope solution, at 1 wt%, 2 wt% and 3 wt%. Inner dope solution flow rate (DSFR) was maintained at 3 ml/min for all fabrication parameters.
7. Study of membrane properties such as surface and cross section morphology using scanning electron microscopy (SEM), particle distribution using energy-dispersive X-ray (EDX), surface roughness using atomic force microscopy (AFM), contact angle using contact angle goniometer, porosity, water uptake and membrane functional group analysis using Fourier-transform infrared spectroscopy (FTIR)
8. Evaluation of the intrinsic filtration properties of the photocatalytic DLHFM in terms of permeability and rejection using water, BSA, 1000 ppm lignin and TA as model solutions, respectively.
9. Study of the photocatalytic degradation of synthetic AT-POME using fabricated membranes in a SMPR under visible light conditions and different photocatalyst loading was evaluated.
10. Study of membrane fouling and membrane long term reusability for 20 days using real AT-POME effluent and evaluate the changes in membrane cross section and morphology using scanning electron microscope (SEM).

1.5 Significance of Study

Titania based photocatalyst, such as TiO_2 and TNT are very prominent in the field of photocatalysis due to their superior catalytic activity. Hence, most catalytic modification works have been done using such it as the model photocatalyst. Among the dopants used to enhance the photocatalytic activity of TNT, boron is one of the least studied upon dopant. Research using other dopants have shown that different doping concentration/molarity can significantly alter physical, chemical and photocatalytic properties of the developed photocatalyst. Doping TNT with different boron molarity would provide significant knowledge on the influence it has on the developed photocatalyst.

The second part of this work focuses on the development of a DLHFM where three key spinning parameters, which are BFFR, outer layer DSFR and AG were manipulated. Based on current literature, there is no detailed work conducted on the influence of different spinning parameters towards membrane physical characteristics and their performance in terms of permeation and rejection. Literature has shown that manipulation of key spinning parameters can significantly alter membrane characteristics, which influences their permeation and rejection capabilities. Hence, the study on these spinning parameters would shed light on the significance of such spinning parameters on the development of a DLHFM.

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Journal with Impact Factor

1. **Subramaniam, M. N.**, Goh, P. S., Lau, W. J., Ng, B. C., Ismail, A. F. (2018) AT-POME colour removal through photocatalytic submerged filtration using antifouling PVDF-TNT nanocomposite membrane. *Separation and Purification Technology, 191*, 266-275. https://doi.org/10.1016/j.seppur.2017.09.042. **(Q1, IF: 5.107)**

2. Rawindran, H., Lim, J. W., Goh, P. S., **Subramaniam, M. N.**, Ismail, A. F., Arzhandi M. R.D. (2019) Simultaneous separation and degradation of surfactants laden in produced water using PVDF/TiO_2 photocatalytic membrane. *Journal of Cleaner Production, 221*, 490-501. https://doi.org/10.1016/j.jclepro.2019.02.230. **(Q1, IF: 6.395)**

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4. **Subramaniam M. N.**, Goh P. S., Tan Y. H., Chiong S. J., Lau W. J., Ng B. C., Ismail A. F., Chuah J. Y., Lai, S. O. (2019) Antifouling improvement of polyethersulfone membrane incorporated with negatively charged zinc–iron oxide for AT_POME colour removal. *Arabian Journal for Science and Engineering, 44 (6)*, 5571-5580. https://doi.org/10.1007/s13369-019-03858-y. **(Q3, IF: 1.518)**

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7. **Subramaniam, M. N.,** Goh, P. S., Lau, W. J., Abidin, M. N. Z., Mansur, S., Ng, B. C., Ismail, A. F (2020) Optimizing the spinning parameter of titania nanotube-boron incorporated PVDF dual-layered hollow fiber membrane for synthetic AT-POME treatment. *Journal of Water Process Engineering, 36, 101372.* https://doi.org/10.1016/j.jwpe.2020.101372. **(Q1, IF: 3.465)**

8. **Subramaniam, M. N.**, Goh, P. S., Lau, W. J., Ismail, A. F., Karaman, M. (2020) Enhanced visible light photocatalytic degradation of organic pollutants by iron doped titania nanotubes synthesized via facile one-pot hydrothermal. *Powder Technology, 366, 96-106.* https://doi.org/10.1016/j.powtec.2020.02.052. **(Q1, IF: 4.142)**

Book Chapter

1. **Subramaniam, M. N.**, Goh, P. S., Lau, W. J., Ng, B. C., Ismail, A. F. (2019) Development of nanomaterial-based photocatalytic membrane for organic pollutants removal. *Advanced Nanomaterials for Membrane Synthesis and its Applications, 45-67.* Elsevier. https://doi.org/10.1016/B978-0-12-814503-6.00003-3