

SUPEROLEOPHOBIC AND SUPERHYDROPHILIC NANOCOMPOSITE  
COATED HOLLOW FIBER CERAMIC MEMBRANE FOR TREATMENT OF  
OILY WASTEWATER

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OILY WASTEWATER

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## ABSTRACT

Robust and efficient separation of emulsified oil and water mixtures is practically required as it still remains a global challenge. Existing conventional methods of oil-water separation using ceramic membranes still suffer some setbacks from membrane brittleness and fouling due to their innate inflexible ionic and covalent bonding and microfiltration pore size. Simultaneous superoleophobic and superhydrophilic surfaces are desired for various applications where emulsified oil separation is required. This study focused on improving the mechanical strength of the bare membrane (BM), addressing the prevailing fouling of membranes, and interactive effects of material properties and operating variables of the system. In the first stage, BM was successfully fabricated using phase inversion and sintering technique from kaolin-based ceramic material at different bore fluid flow rates (12.0-15.0 mL/min), kaolin content loadings (38-42 wt.%), and sintering temperatures (1300-1400 °C) and was examined for physicochemical properties. BM fabricated at 42 wt.% kaolin loading, 15 mL/min bore fluid flow rate and 1400 °C sintering temperature showed the best mechanical strength of 126.00 MPa, an average pore size of 3.50  $\mu\text{m}$ , and pure water flux of 108.57 L/m<sup>2</sup>.h. The result suggested an improvement in the mechanical strength and that BM was within range of microfiltration application. Due to its pore structures, it was prone to membrane fouling for oil-water separation. In the second stage, BM was modified through a simple dip-coating process with poly(diallyl dimethylammonium chloride)-alumina-perfluorooctanoic acid (PAP) and poly(diallyl dimethylammonium chloride)-perfluorooctanoic acid (PP) as interfacial materials. The interfacial materials were prepared using sol-gel method. Effects of coating layer cycles on BM were examined for surface wettability. Physicochemical properties of the PAP-BM and PP-BM including the surface morphologies and topologies were also studied. Highest average roughness, Ra of 1.042 was obtained for PAP-BM while BM with Ra of 0.665 showed the lowest. Oil contact angle (OCA) of PAP-BM was found at 155° while water contact angle (WCA) displayed time dependence mode as values decreased from 25° to 5° after 10 min of penetration. The third stage involved the performance evaluation using statistical software Design-Expert 7.0.0 based on response surface methodology. Three independent parameters (feed concentration, pH, and pressure) were examined for interactive effects on the two dependent parameters (oil rejection and water flux). Prior to that, the results of the antifouling test showed the total fouling resistance of ~66% and ~73%, reversible fouling resistance of ~52% and ~26% and irreversible fouling resistance of ~14% and ~48% for PAP-BM and BM, respectively. The ANOVA results revealed that the optimum conditions of the emulsified oily wastewater separation were obtained to be 600 ppm, 3.0 bar, and 9.0 for feed concentration, pressure and pH, respectively. Under these conditions, 174.35 L/m<sup>2</sup>.h of water flux and 98.63% of oil rejection were achieved experimentally. Oil rejection and water flux were found to be primarily affected by the feed concentration, pressure and pH. Hence, PAP-BM can tolerate and display intermittent superoleophobic and superhydrophilic surfaces, which was further evaluated based on its performance in a cross-flow filtration system.

## ABSTRAK

Pemisahan campuran minyak dan air yang cekap diperlukan secara praktikal, kerana ia masih menjadi satu cabaran global. Kaedah konvensional sedia ada untuk rawatan air sisa berminyak menggunakan membran seramik, masih mengalami banyak kekurangan dari segi kerapuhan membran dan pengotoran membran berpunca dari ikatan ionik dan kovalen tidak stabil serta saiz liang mikropenurasan. Permukaan oliofobik lampau dan hidrofilik lampau secara serentak dikehendaki untuk pelbagai aplikasi di mana pemisahan emulsi minyak diperlukan. Kajian ini juga bertujuan untuk meningkatkan kekuatan mekanikal membran terdedah (BM), menangani kekotoran membran, dan mengkaji kesan saling tindak antara sifat bahan dan pemboleh ubah operasi sistem. Pada peringkat pertama, BM telah berjaya dihasilkan menggunakan penyongsangan fasa dan teknik sinteran dari bahan seramik berasaskan kaolin pada kadar aliran cecair jara yang berbeza (12.0-15.0 mL / min), beban kandungan kaolin (38-42 wt.%), dan suhu sinteran (1300-1400 °C) dan telah diperiksa untuk sifat-sifat fisikokimia. BM yang dihasilkan pada 42 wt.% kaolin, 15 mL / min kadar aliran cecair jara dan suhu sinteran 1400 °C menunjukkan kekuatan mekanikal terbaik pada 126.00 MPa, purata saiz liang 3.50  $\mu\text{m}$ , dan fluks air tulen 108.57 L/m<sup>2</sup>.h. Keputusan kajian menunjukkan peningkatan pada kekuatan mekanikal dan BM berada dalam julat aplikasi mikropenurasan. Oleh kerana struktur liangnya, ia terdedah kepada kekotoran membran bagi pemisahan air minyak. Pada peringkat kedua, BM diubahsuai menerusi proses lapisan celup mudah bersama asid poli (dialil dimetilammonium klorida)-alumina-perflorooktanoik (PAP) dan poli (dialil dimetilammonium klorida)-perflorooktanoik (PP) sebagai antara muka bahan. Bahan antara muka disediakan menggunakan kaedah sol-gel. Kesan kitaran salutan ke atas BM diperiksa untuk kelembapan permukaan. Ciri-ciri fisikokimia PAP-BM dan PP-BM termasuk morfologi permukaan dan topologi juga dikaji. Purata kekasaran tertinggi, Ra 1.042 diperolehi untuk PAP-BM manakala BM dengan Ra 0.665 menunjukkan nilai terendah. Sudut sentuhan minyak PAP-BM ditemui pada 155° manakala sudut sentuhan air memaparkan mod bersandar masa kerana nilai menurun daripada 25° kepada 5° selepas 10 minit penembusan. Peringkat ketiga melibatkan penilaian prestasi menggunakan perisian statistik Design-Expert 7.0.0 berdasarkan kaedah sambutan permukaan. Tiga parameter tidak bersandar (kepekatan suapan, pH, dan tekanan) telah diperiksa untuk kesan interaktif pada kedua-dua parameter bersandar (penolakan minyak dan fluks minyak). Sebelum itu, keputusan ujian anti-kotoran menunjukkan jumlah kotoran sebanyak ~66% dan ~73%, kotoran berbalik sebanyak ~52% dan ~26% dan kekotoran yang tidak dapat dipulihkan masing-masing sebanyak ~14% dan ~48% untuk PAP-BM dan BM. Keputusan ANOVA menunjukkan bahawa keadaan optimum pemisahan air sisa berminyak diperolehi masing-masing bernilai 600 ppm, 3.0 bar, dan 9.0 untuk kepekatan suapan, tekanan dan pH. Di bawah syarat-syarat ini, 174.35 L/m<sup>2</sup>.h fluks minyak dan 98.63% penolakan minyak telah dicapai secara eksperimen. Penolakan minyak dan fluks minyak didapati terjejas terutamanya oleh kepekatan makanan, tekanan dan pH. Oleh itu, PAP-BM boleh mentoleransi dan memaparkan permukaan oliofobik super dan hidrofilik super yang berselang-seli berdasarkan prestasi PAP-BM dalam sistem penapisan aliran silang.

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## LIST OF ABBREVIATION

AFM	-	Atomic force microscopy
AG	-	Air gap
ASTM	-	American society for testing and materials
ANOVA	-	Analysis of variance
BBD	-	Box Behnken design
BOD	-	Biological oxygen demand
BM	-	Bare membrane
BN	-	Boron nitride
CA	-	Contact angle
CCD	-	Central composite design
COD	-	Chemical oxygen demand
CNTs	-	Carbon nanotubes
ED	-	Electrodialysis
EQR	-	Environmental quality regulations
EDX	-	Energy dispersive X-ray
FESEM	-	Field emission scanning microscopy
FTIR	-	Fourier transformation infrared
FO	-	Forward osmosis
GP	-	Gas permeation
GS	-	Gas separation
IF	-	Irreversible fouling
MB	-	Methylene blue
MIP	-	Mercury intrusion porosity
NMP	-	N-methylpyrrolidone
NP	-	Nanofiltration
NPs	-	Nanoparticles
OCA	-	Oil contact angle
OPS	-	Oil palm shell
PAM	-	Polyacrylamide

PAP	-	Poly(diallyldimethylammoniumchloride)- alumina-perfluorooctanoic acid
PEG	-	Polyethylene glycol
PFO	-	Perfluorooctanoate
PSA	-	Particle size analysis
PES	-	Polyethersulfone
FRP	-	Flux recovery percentage
POM	-	Palm oil mill
POME	-	Palm oil mill effluent
PP	-	Polypropylene
PS	-	Polystyrene
PTFE	-	Polytetrafluoroethylene
PVP	-	Polyvinylpyrrolidone
RF	-	Reversible fouling
RSM	-	Response surface methodology
$Q_{AO}$	-	Flow rate of the feed
$Q_{AP}$	-	Flow rate of the permeate
$Q_{AR}$	-	Flow rate of the retentate
RO	-	Reverse osmosis
RPM	-	Revolutions per minute
SDS	-	Sodium dodecyl sulfate
SEM	-	Scanning electron microscopy
TF	-	Total fouling
TEOS	-	Tetraethylorthosilicate
TGA	-	Thermogravimetric analysis
TiC	-	Titanium carbide
UF	-	Ultrafiltration
UTM	-	Universiti Teknologi Malaysia
WCA	-	Water contact angle
WHO	-	World Health Organization
XRD	-	X-ray diffraction
XPS	-	X-ray photoelectron spectroscopy

## LIST OF SYMBOLS

$\Delta t$	- Operating time (h)
$A$	- Effective area of membrane (m <sup>2</sup> )
$Q_f$	- Bending strength (MPa)
$\gamma$	- Surface tension mercury (N/m)
$C_{d,final}$	- Final oil concentration
$C_{d,initial}$	- Initial oil concentration (ppm)
$C_f$	- Feed concentration (ppm)
$C_p$	- Permeate concentration (ppm)
$J_o$	- Flux determined at the beginning in the filtration (L/m <sup>2</sup> .h)
$J_t$	- Flux determined at every hour in the filtration (L/m <sup>2</sup> .h)
$R_{oil}$	- Oil rejection rate (%)
$V$	- Volume of permeated water (mL)
$V_{d,final}$	- Final volume of draw solution (mL)
$V_{d,initial}$	- Initial volume of draw solution (mL)
$V_p$	- Volume of permeate passing across the membrane (mL)
Wt %	- Weight percentage (%)
$D_i$	- Inner diameter of the membrane (mm)
$D_o$	- Outer diameter of the membrane (mm)
$F$	- Force at which the membrane fracture (N)
$J_v$	- Permeate flux of the membrane (L/m <sup>2</sup> .h)
$L$	- Length of the membrane (cm)
$L_p$	- Water permeability (L/m <sup>2</sup> .h.bar)
$P$	- Transmembrane pressure (bar)
$P_a$	- Applied pressure (bar)
$R_a$	- Surface roughness (μm)
$Q$	- Volume of water permeate through the membrane (mL)
oC	- Degree Celsius (°C)
%	- Percentage (%)

$\bar{X}$	- Mean
$S$	- Standard deviation
$S^2$	- Variance
$S_{\bar{X}}$	- Margin of error
$N$	- Number of samples
$K_{cost}$	- Cost of kaolin
$Al_{cost}$	- Cost of alumina
$P_{cost}$	- Cost of Polyethersulfone (PESf)
$N_{cost}$	- Cost of N-methylpyrrolidone (NMP)
$A_{cost}$	- Cost of Arlcel P135
$A_{TF}$	- Surface area of tubular furnace (m <sup>2</sup> )
$\lambda_{TC}$	- Thermal conductivity of tubular furnace (W/m. K)
$T_{TF}$	- Thickness of tubular furnace (m)
$S_{TF}$	- Internal space surface area (m <sup>2</sup> )
$\lambda_{ITC}$	- Int. space thermal conductivity of tubular furnace (W/m. K)
$T_{ITF}$	- Internal space thickness of tubular furnace (m)
$C_p$	- Specific heat capacity (J/g. K)
$\rho$	- Density of air (g/L)
$T_{MTF}$	- Thickness of membrane tubular furnace (m)
$\dot{m}$	- Mass flow rate of air (g/h)
$t_{hr}$	- Heating time
$\Delta\theta$	- Temperature difference (K)
$AL_{mcost}$	- Material cost of alumina
$PDADMAC_{mcost}$	- Material cost PDADMAC
$NaPFO_{mcost}$	- Material cost NaPFO
$ETOH_{mcost}$	- Material cost ethanol

## LIST OF APPENDICES

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# CHAPTER 1

## INTRODUCTION

### 1.1 Background Introduction

Oily wastewater produced by industry has become a global issue affecting human and aquatic lives. Predominantly, oily wastewater contains different forms of oils such as stable emulsified oils, unstable dispersed oils and free-floating oils (Han *et al.*, 2015; Gohari *et al.*, 2015; Lu *et al.*, 2015; Zhu *et al.*, 2017). In contrast with free-floating oils that is the spilled oils on the deep-sea, dispersed oils are arbitrarily distributed in water. It has a strong propensity to amalgamate and instinctively develop into free-floating oils. Unlike, emulsified oils are relatively stable due to the existence of surfactant as in the case of asphaltenes in crude oil. Emulsified oils have small droplet sizes, usually not more than 10  $\mu\text{m}$  (Dickhout *et al.*, 2017; Tummons *et al.*, 2017; Zhang *et al.*, 2017; Zhu *et al.*, 2017). Conventional approaches to the treatment and separation of oil from oily wastewater, such as coagulation, floatation, gravity settling, and ultrasonic separation have been found to be ineffective mainly due to low separation efficiency, process separation units of equipment are complex, and high energy cost and secondary pollution (Das *et al.*, 2017; Hua *et al.*, 2007a; Cheryan & Rajagopalan, 1998; Du *et al.*, 2017; Rubio *et al.*, 2002). Still, these approaches can be used for treating free-floating oils and dispersed oils, majority of them are not appropriate for treating emulsified oils for the reason that the emulsified oils have small droplet sizes, low bulk difference compared to  $\text{H}_2\text{O}$  ( $150 \text{ kg/m}^3$ ) and high stability (Han *et al.*, 2015; Li *et al.*, 2017; Motin *et al.*, 2015). For example, oily wastewater from industries such as palm oil mill effluent (POME) is extremely common pollutant worldwide as it affects the environment and aquatic life. Figure 1.1 shows the global research trends on POME. Malaysia is currently leading with about 61% in this regard, followed by Indonesia with 12% and closely followed by Thailand with 8.17% (Scopus, 2 January 2021).



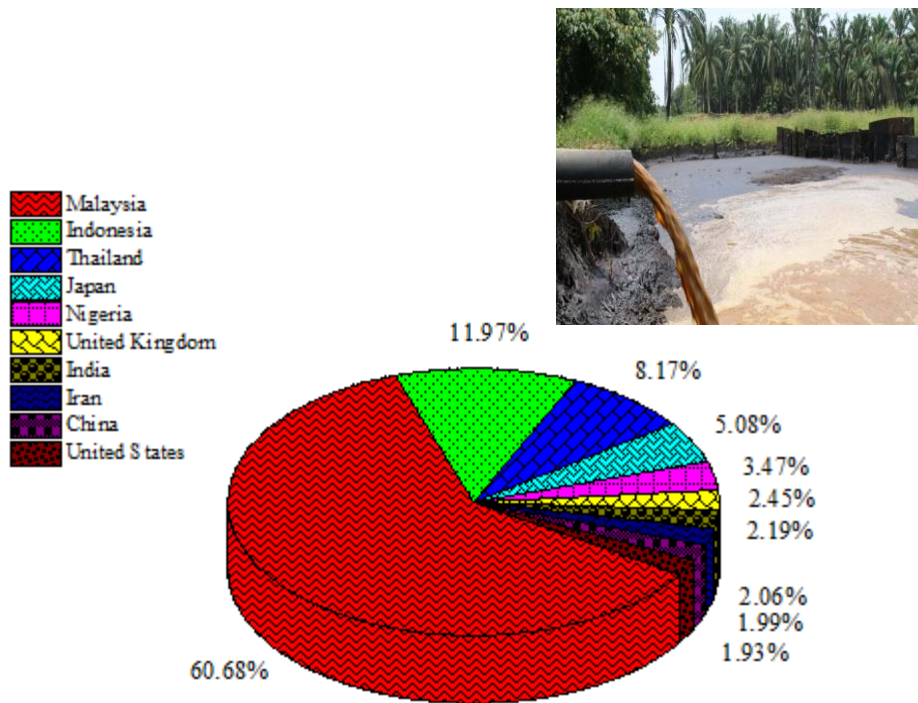


Figure 1.1 Global POME research output. Data were obtained from Scopus and accessed in January 2021

The effects can be attributed to the high demand for palm oil in the current market, globally. As shown in Figure 1.2, the production of palm oil increased more than double in the last one and half decades with an average growth of 6.8% per annum and accounted for 31% of the global oils and fats supply chain in 2018 (Ling, 2019). The effluent has a higher proportion of water (95 – 96%), with 0.6 – 0.7% oil, and the remaining 4.5% is total solid (Ahmad *et al.*, 2009; Azmi & Yunus, 2014; Chen & Xu, 2013; Kamyab *et al.*, 2018; Wang, 2006). This has generated tremendous concentration of biological oxygen demand (BOD), chemical oxygen demand (COD), oil and grease, as well as suspended solids; the level of this organic matter is due to the presence of unrecovered raw palm oil and its disposal to the water bodies without pre-treatment, posing threat to humans and the ecosystem (Ahmad *et al.*, 2005; Ahmad *et al.*, 2006; Jumadi *et al.*, 2021).

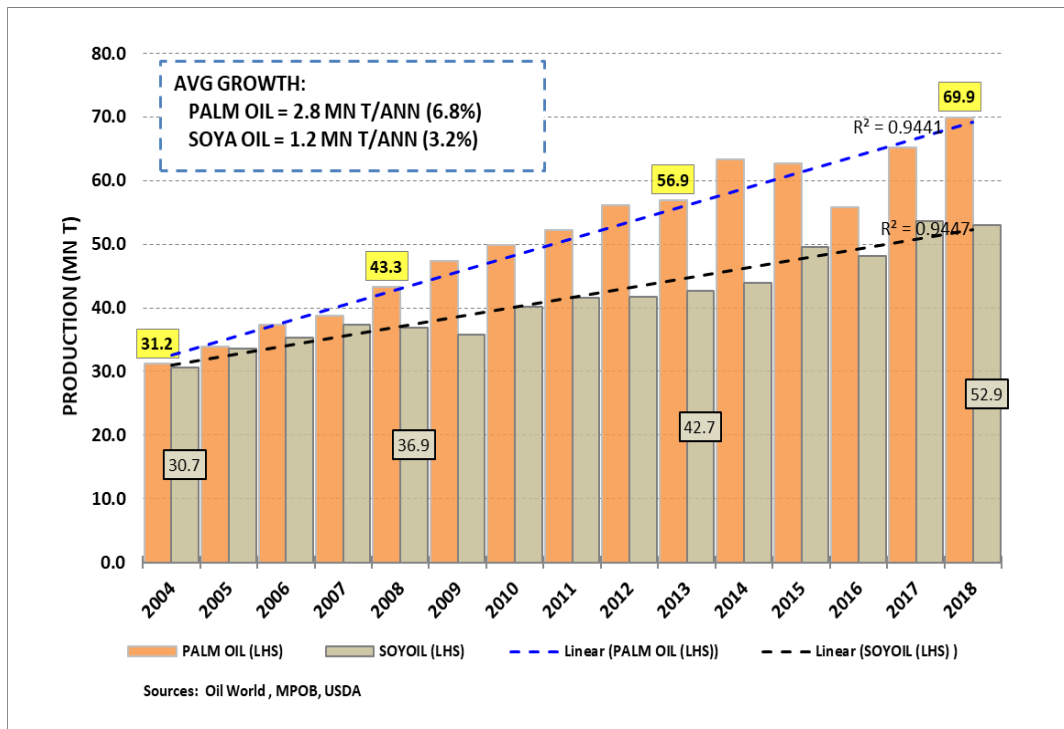


Figure 1.2 Global palm oil production with dynamic supply growth. An excerpt from Malaysia-China business forum, 4 March 2019, Kuala Lumpur and accessed in December 2020

However, the current strategy of using conventional biological anaerobic and aerobic systems, and faulty ponds treatment rely on the application of bacteria, which is unsafe, uneconomical, and unsustainable (Ahmad *et al.*, 2003), as it requires highly proper maintenance and assessment, and high labour and operational cost. Figure 1.3 gives insight into how rainfall patterns significantly influence BOD and SS concentration and how it affects quality of water. This is because water turbidity and TSS can be affected due to heavy rainfall during peak periods resulted in pond banks attrition, thereby resulting in SS suspension instead of settling at the bottom of the pond (Jumadi *et al.*, 2021). The current problems and challenges would require an advanced separation strategy such as microfiltration and ultrafiltration techniques to aid and reclaim a large proportion of water lost from palm oil production (Ahmad *et al.*, 2015). However, it is pertinent to protect the environment and develop systems that are economically viable and sustainable.

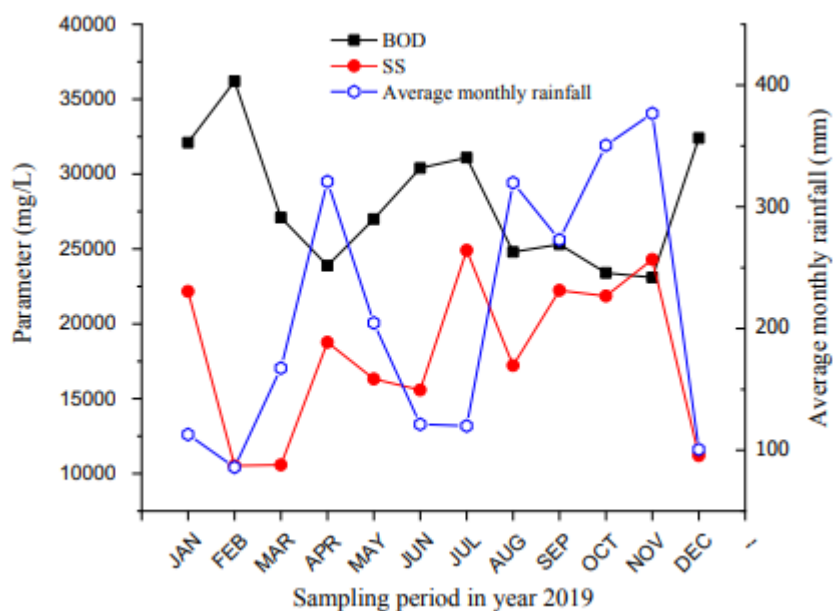


Figure 1.3 Raw palm oil mill effluent monitoring studies of BOD, SS and average monthly. 17<sup>th</sup> International symposium on solid oxide fuel cells. Digital meeting, July 18, 2021

One of the strategies that have been widely employed for oil-water separation with a different degree of oil concentration is the membrane separation technology. It is due to its efficiency and high selectivity structures. More of its merits are the high emulsion separation efficiency, no addition of chemicals and the ease of its operation. Inorganic (ceramic) membranes have been taken into consideration for presenting more advantages in terms of excellent solvent resistance, long lifetime, high thermal stability and exhibiting chemical inertness in the area of MF and UF processes (Xing, 2017). Other types of membranes from materials such as metal meshes, cotton, foams and nanofibers can be applied preferably as pre-treatment of oily wastewater (Du *et al.*, 2017; Han *et al.*, 2017; Li *et al.*, 2015; Obaid *et al.*, 2015; Wu *et al.*, 2017; Zhang *et al.*, 2015; Zhang *et al.*, 2017; Zhu & Chen, 2017).

Several articles have reported various strategies of oil and water separation, especially the use of ceramic membranes: alumina (Dong *et al.*, 2020),  $\alpha\text{Al}_2\text{O}_3$  (Abadi *et al.*, 2011), kaolin (Hubadillah *et al.*, 2018), sugarcane bagasse waste (Jamalludin *et al.*, 2019), kaolin/fly ash (Zou *et al.*, 2020), fly ash (Tai *et al.*, 2019), and ball clay (Abd Aziz *et al.*, 2019). Hence, a more robust, environmentally friendly and efficient

oil and water separation system made from reusable and durable material is proposed. The oil and water separation systems that can treat and provide a volume of water in oily wastewater, with high effectiveness in the flux rates, also address the impediment and challenge of fouling in membranes should have the abovementioned characteristics. According to the report from Scopus 2<sup>nd</sup> January 2021 (Figure 1.4) over the last ten years (2010 -2020), the studies showed an increase in the researches related to oil and water separation technologies, and further studies revealed that the development of surface wettability remains the focus and of interest to the researchers as it involves the ability of a liquid to maintain contact with solid surface (Gupta *et al.*, 2017; Tuteja *et al.*, 2007; Yong *et al.*, 2017). Figure 1.4 reveals the country-wise distribution of oil-water separation researchers as obtained from Scopus. The chart demonstrated that the attraction in oil-water separation research is growing world recognition with the increase in the number of researchers across the globe.

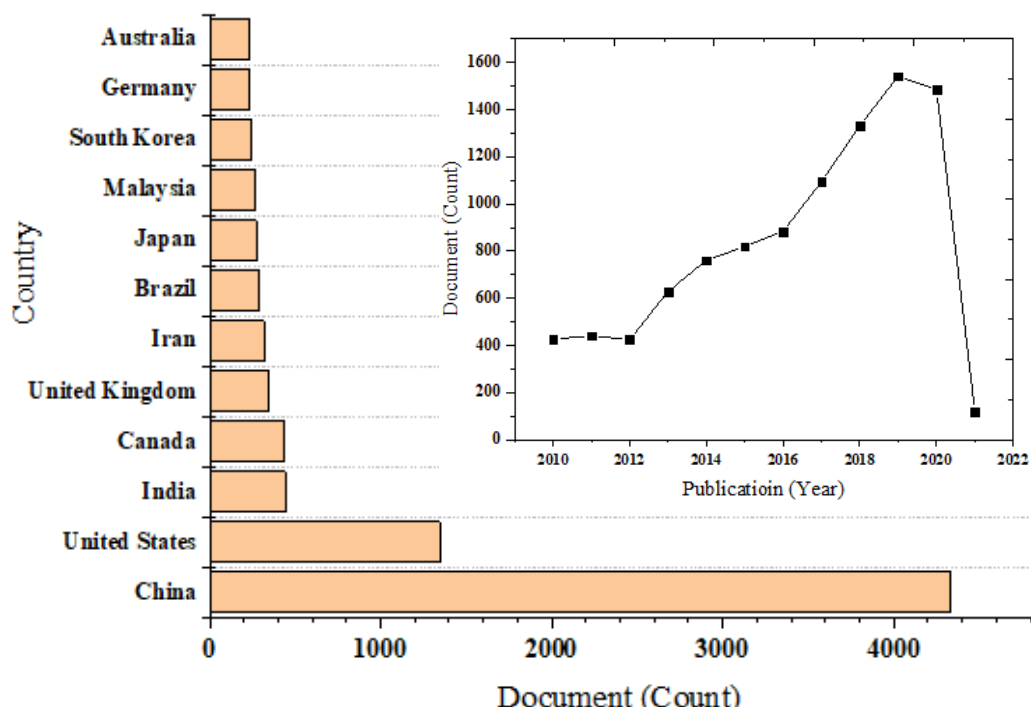


Figure 1.4 Research outlook on oil-water separation since 2010; output based on countries. Inset research publication on oil-water separation. Data were obtained from Scopus and accessed in February 2021

The application of ceramic membrane for oily water separation gives impetus to the physical structure and surface energy of membrane; in other words, it gave importance to the substrate with high surface roughness and suitable wettability (Gupta *et al.*, 2017). Both influence the concept of surface wettability behaviour, which usually expresses the ability of a material to get wetted when liquid encounters it. Super-wettability, such as for superhydrophilicity, has properties of water recovery from oily wastewater (Li *et al.*, 2011; Xue *et al.*, 2011; Jamaly *et al.*, 2015), whereas, superoleophobicity membrane material is still quite challenging to develop as oils with low surface tension tend to wet and spread across the membrane surfaces, and this leads to foulant generation, consequentially reduces flux rates, and ultimately leading to a decline in the operation and the performance of membrane (Liu *et al.*, 2014; Li *et al.*, 2011; Pan *et al.*, 2013; Tuteja *et al.*, 2007; Zhang *et al.*, 2011). However, surface functionalization of membrane enables alteration of the membrane surface's wetting properties for proper oil and water separation. One of such functionalization materials is an inorganic material, which is the most common because of its unique capability to exhibit surface roughness and efficiently make membrane function for oil and water separation (Wang & Guo *et al.*, 2013a,b).

## **1.2 Problem Statement**

Ceramic materials are very central to the numerous industrial applications involving severe conditions due to their unique properties such as thermal stability, chemical resistance, high mechanical strength and low surface area per unit volume, high permselectivity (Dai *et al.*, 2018; Tan *et al.*, 2001). Studies on inorganic membranes showed that hollow fibre membranes from such materials are comparatively scarce and too expensive as compared to ceramic materials (Liu *et al.*, 2001; Tan *et al.*, 2001). Hitherto, the intrinsic brittleness and fouling are the foremost drawbacks of ceramic membranes importantly in the oil and water separation. Firstly, membrane fabricated from ceramic materials is susceptible to breakable and rupture during permeation process due to innate inflexible ionic and covalent bonding (Basu & Balani, 2011), and in addition to the inappropriate ratio of

ratio ceramic material to a binder and small diameter of the hollow fibre during dope suspension preparation and fabrication process, respectively. This has many significant effects on the mechanical strength of ceramic membranes. Thus, high mechanical strength of ceramic membrane is very critical to membrane longevity (Xu *et al.*, 2014). For the stability and high efficiency of the membrane, 100 MPa is preferable (Abdulhameed *et al.*, 2017). However, most of the studies on the use of kaolin ceramic materials have not exceeded 100 MPa (Abdulhameed *et al.*, 2017; Hubadillah *et al.*, 2017, 2018; Mohtor *et al.*, 2017; Nandi *et al.*, 2008). But different studies suggested that varying the fabricating parameters will significantly improve the mechanical strength. Parameters such as ceramic material to the binder ratio (Liu *et al.*, 2001; Tan *et al.*, 2001), bore fluid flow rate (Adam *et al.*, 2019; Alobaidy *et al.*, 2017; Jamalludin *et al.*, 2018; Makhtar *et al.*, 2017a; Tai *et al.*, 2021), material content loading (Emani *et al.*, 2013; L.-F. Han *et al.*, 2011; Hubadillah, Harun, *et al.*, 2016), sintering temperature (Alves *et al.*, 2016; Chihi *et al.*, 2019b; Makhtar *et al.*, 2017b) are vital to the membrane structure and performance.

Secondly, ceramic membrane contains mineral oxides; its high surface energy (represents the equivalent attractive force between the liquid at the surface of a solid surface), makes it easily fouled by oil and other organic matter, which are quite challenging to remove, ruin membrane surfaces and block the membrane pores (Gao *et al.*, 2016). With such fouling, such surface hinders the smooth practical application of hydrophilic surface for oleophobicity. To address the challenges of the porogenic nature of ceramic membrane with respect to integrity with less resistance to fluid flow; by controlling the surface topology. Hence, this would require improving the surface structure and controlling the surface energy of ceramic membranes through surface modification of membranes' topology. The stimuli-responsive surface is accomplished by the simultaneous display of interaction between water as a polar phase and oil as a non-polar phase. In other words, water molecules would be able to penetrate the surface as a result of water-induced molecular rearrangement with the hydroxyl group of ceramic membrane attached to the interface.

On the other hand, oil at the interface of perfluoroalkyl group of the nanocomposite would experience low surface energy with oleophobicity because PFO has the tendencies to transforming the  $\text{Al}_2\text{O}_3$  surface by lowering its surface energy. The PDADMAC prevents agglomeration of alumina nanoparticles, and surface cracking of membrane surface, thereby improving the oleophobicity of the coating.

To the best of my knowledge, superoleophobic and superhydrophilic stimuli-response surfaces from kaolin-based ceramic membrane coated with fumed alumina nanocomposite have not been reported and used in the literature for the fabrication of hollow fiber ceramic membrane for the oil-water separation. Therefore, the current study aimed at creating superoleophobic and superhydrophilic hollow fiber ceramic membrane for the separation of synthesized oily wastewater. Firstly, the fabrication of the bare membrane would be carried out using spinning process and sintering technique from commercial kaolin as a starting material. Bare membrane fabrication is carried out at different bore fluid flow rate, kaolin content loading and sintering temperature to improve the mechanical strength of the membrane. Secondly, the surface modification of the bare membrane is functionalized using different composites such as PDADMAC-PFO (PP) and PDADMAC- $\text{Al}_2\text{O}_3$ /PFO (PAP) prepared by the sol-gel method using a simple dip-coating technique to address the prevailing fouling of ceramic membrane. The research problem is expected to solve by adhering to the research objectives outlined in Section 1.3. The development of ceramic membrane would be more rewarding and beneficial if the membrane obtained from the ceramic material fabrication is matched with the desired application to achieve excellent and efficient membrane performance. Hence, the research contributes to the field of knowledge and partly addresses the threats posed by oily wastewater to the environment.

### 1.3 Objectives of the Study

Based on the problem statement, the main objective of this thesis is to fabricate a superoleophobic and superhydrophilic hollow fiber ceramic membrane-based nanocomposite for treatment of oily wastewater. However, the specific objectives are:

- (a) To correlate the effect of bore fluid flow rates, kaolin loadings, and sintering temperatures to achieve an improved mechanical strength hollow fiber ceramic membrane via extrusion and sintering techniques.
- (b) To examine the superoleophobicity and superhydrophilicity of the bare membrane and  $\text{Al}_2\text{O}_3$  incorporation on the PDADMAC-PFO based layer by simple dip-coating method at different coating cycles, heat treatment and chemical resistance treatment.
- (c) To optimize the performance of the superoleophobic and superhydrophilic membranes through a cross-flow permeation with three independent parameters (feed concentration, pressure and pH) and two dependent parameters (oil rejection and water flux) using response surface methodology (RSM).

### 1.4 Scope of the Study

The following research activities have been selected as the scope of study to achieve the aforementioned specific objectives:

1. To correlate the effect of bore fluid flow rates, kaolin loadings, and sintering temperatures to achieve high mechanical strength hollow fiber ceramic membrane via extrusion and sintering techniques, the characterization of kaolin powder using PSA, XRD and FTIR, formulation of kaolin powder (KP) dope suspensions with the percentage by weight of KP1: 38 wt.%, KP2: 40 wt.%, KP3: 42 wt.% and the viscosity tests on the kaolin dope suspension loadings were conducted. Subsequently, the fabrication of bare membrane precursor via orifice spinneret using extrusion and a phase inversion technique with an air gap at 5 cm and



extrusion rates of 12 mL/min at different bore fluid flowrates: 12 mL/min, 13.5 mL/min, and 15 mL/min and the sintering process at different temperatures: 1300 °C, 1350 °C, 1400 °C to obtain the final bare membrane were investigated. In addition, the morphological studies and surface properties analyses: FESEM analysis for the surface area and cross-sectional area and pore size distribution and porosity test analysis of the bare membranes were examined. The Contact angle test for bare membranes 1300 °C, 1350 °C and 1400 °C was also conducted. The physical properties such as the mechanical strength analysis using three-point bending test analysis of the bare membranes was carried out and the permeability test to check for water flux capability was also investigated. The chemical properties such as FTIR analysis and XRD pattern of sintered bare membranes were examined.

2. To investigate the effect of coating cycle and Al<sub>2</sub>O<sub>3</sub> incorporation on the PDADMAC-PFO based layer by simple dip-coating method, the synthesis of PDADMAC-Al<sub>2</sub>O<sub>3</sub>/PFO complex polymer nanocomposites and the investigation on the effect of surface wettability at 5 cycles of dip coating on the surface of bare membranes were carried out. While the morphologies and topologies were investigated via FESEM, SEM-EDX, and AFM analyses for membranes cross-sectional and surface area. The modified bare membranes were investigated and analysed for the porosity and pore size distribution using mercury intrusion porosimetry and as well XRD, FTIR, and XPS. Subsequently, the wettability test via water and oil contact angle on the membranes was conducted. Then, the investigation on the effect of chemical resistance and thermal treatment on the coated bare membrane was examined.
3. Lastly, to evaluate the performance of the superoleophobic and superhydrophilic kaolin-based hollow fiber membranes through a cross-flow permeation system, first and foremost, the preliminary evaluation on the uncoated and coated membranes were investigated for oil rejection and water flux. Secondly, the antifouling tests were performed on the membranes. Then lastly, for this objective, the design of experiment was performed based on a statistical software Design-Expert 7.0.0 (Stat-Ease, 2005) with respect to the following factors and percentage

of oil rejection and water flux were considered as the responses: (a) concentration of lower (10 ppm) and upper (10000 ppm) limit, (b) operating pressure at lower (0 bar) and upper (3.0 bar) limit, (c) pH at lower (4) and upper (10) limit, (d) ANOVA test, and (e) Optimization test. In addition, cost analysis for membrane materials and energy was estimated.

## **1.5 Significance of the Study**

This study entails the process fabrication of superoleophobic and superhydrophilic hollow fiber ceramic membranes for oily wastewater treatment. One of the significances of this study is the use of local material that is readily available to produce ceramic membranes via spinning process and sintering technique. Another implication of this research is to overcome the limitation of conventional (biological and ponding) oily wastewater treatment with the use of hollow fiber ceramic ultrafiltration technology; water reclamation which would translate into a reduction in the use of chemical additives, lower operating cost and maintenance, and increase productivity. Outside these technical benefits, this study also promotes SDG 6 target 6.3 (2030), thereby improving water quality by reducing environmental pollution, eliminating dumping and minimizing release of hazardous chemicals and materials, halving the proportion of untreated wastewater and substantially increasing recyclability and safe reusability globally. Lastly, the outcome of this study would contribute significantly towards knowledge-driven research and contribute to the environmental sustainability of current oily wastewater.

## **1.6 Organization of the Thesis**

This thesis is divided into seven chapters including the preparation and characterization of high mechanical strength hollow fiber ceramic membranes via the spinning process and sintering technique at a high loading of kaolin and different sintering temperatures, the examination of the physicochemical characterization of

fabricated hollow fiber ceramic membrane, the modification of bare membrane for the superoleophobicity and superhydrophilicity property by dip-coating using PDADMAC- $\text{Al}_2\text{O}_3$ /PFO nanocomposite, and lastly, the performance evaluation of modified membrane using synthesized oily-wastewater through a cross-flow permeation system.

**Chapter 1** presents a brief background introduction on oily wastewater treatment using membrane technology. The statement of the problem, objectives, research scopes and significance of the study are highlighted.

**Chapter 2** explicitly provides elaborated literature reviews of the research topics. This includes background literature on oily wastewater, treatment technologies, membrane materials, fabrication techniques and application, membrane fouling and control, process optimization and also the research gap.

**Chapter 3** elaborates on the materials, techniques and working principles, characterization approaches and experimental step-up for the oily wastewater treatment.

**Chapter 4** formulates and describes the detailed fabrication of kaolin-based hollow fiber ceramic membrane via phase inversion and sintering technique. The effect of kaolin content loading and sintering temperatures are examined and carefully characterized and discussed. It also describes the physicochemical characterization of the fabricated membrane to include mechanical strength, porosity test, water permeation test, and as well as determining the functional elements and groups.

**Chapter 5** prepares and intercalates alumina-based NPs for the surface functionalization of kaolin-based membrane. Superoleophobic membrane obtained is characterized by contact angle, permeability test, and porosity test. Besides, surface morphologies and membrane antifouling processes are also studied.

**Chapter 6** evaluates the performance of the modified membrane through a cross-flow filtration system for oil rejection and water flux. The antifouling properties of the membranes were also investigated. The effect of oil concentrations, the effect of pH and operating pressure based on the design of experiment using RSM are also elaborated.

**Chapter 7** concludes each of the chapters contained in the thesis. The suggestions and recommendations for future work are also provided.

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## LIST OF PUBLICATIONS

### Journal with Impact Factor

1. **Raji, Y. O.**, Othman, M. H. D\*., Nordin, N. A. Md., Zhong, S. T., Usman, J; Mamah, S. C., Ismail, A. F., Rahman, M. A., Jaafar, J. (2020). Fabrication of magnesium bentonite hollow fibre ceramic membrane for oil-water separation. *Arabian Journal of Chemistry*, 13(7), 5996-6008 .  
<https://doi.org/10.1016/j.arabjc.2020.05.001>. (**Q2, IF:4.762**)
2. **Raji, Y. O.**, Othman, M. H. D\*., Nordin, N. A. Md., Adam, M. R., Zhong, S. T., Usman, J; Ismail, A. F. (2020). Surface matrix functionalization of clay-based ceramic membrane for oily water treatment: A mini-review. *Korean Journal of Chemical Engineering*, 37,1631-1641.  
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3. **Raji, Y.O.**, Othman, M. H. D\*., Nordin, N. A. Md., Kurniawan T. A., Ismail, F. A., Rahman M., Jaafar J., Adam M. R., Alftessi S. A., El-badawy T., Farag, T. M. (2021). Wettability improvement of ceramic membrane by intercalating nano- $\text{Al}_2\text{O}_3$  for separation of oil and water. *Surfaces and Interfaces*, 25,101178.  
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4. **Raji, Y.O.**, Othman, M. H. D\*., Nordin, N. A. Md., Adam, M. R., Ismail, F. A., Rahman M., Jaafar J., Said, K. A. M., Farag, T. M., Alftessi S. A., (2021). Synthesis and characterization of superoleophobic fumed alumina nanocomposite coated via the sol-gel process onto ceramic-based hollow fibre membrane for oil-water separation. *Ceramics International*. 47(11) 15367-15382. <https://doi.org/10.1016/j.ceramint.2021.05.319>. (**Q1, IF: 3.830**)

## Indexed Journal

1. **Raji, Y. O.**, Othman, M. H. D\*., Nordin, N. A. Md., Ismail, A. F., Rahman, M. A., Jaafar, Juhana, J., Usman, J; Mamah, S. C. (2020). Preparation and characterization of bentonite-based ceramic hollow fibre membrane, *Journal of Membrane Science and Research*, 7(2),95-101.  
<https://doi.org/10.22079/JMSR.2020.121902.1346> (**Indexed by Scopus**)
2. **Raji, Y. O.**, Othman, M. H. D\*., Nordin, N. A. Md., Ismail, A. F., Rahman, M. A., Jaafar, J., Ahmed, I., Mamah S. C., Usman J., Origomisan O. J. (2021). Effect of fluorosurfactant on alumina membrane for oil and water separation. *MaterialsToday: Proceedings*.46,1983-1989.  
<https://doi.org/10.1016/j.matpr.2021.02.596>

## Non-Indexed Conference Proceedings

1. **Raji, Y. O**; Othman, M. H. D\*; Nordin, N. A. H; Ismail, A. F; Rahman, M. A; Jaafar, J; Usman, J; Mamah, S.C. (2019). Preparation and characterization of bentonite-based ceramic hollow fibre membrane for treatment of oily wastewater. *International Conference of Sustainable Environmental Technology, 2019*, DoubleTree Hilton Hotel, Johor, Malaysia.
2. **Raji, Y. O**; Othman, M. H. D\*; Nordin, N. A. H; Ismail, A. F; Rahman, M. A; Jaafar, J; Ahmed, I; Mamah, S. C; Usman, J; Origomisan, O.J. (2020). Effects of Fluorosurfactant on Nanocomposite Prepared by Sol-Gel Dip-Coating Process for Oil and Water Separation. *Virtual- Regional Conference on Membrane Technology*, Johor, Malaysia.