FABRICATION AND CHARACTERISATION OF CERAMIC HOLLOW FIBRE MEMBRANE FROM METAKAOLIN AND CORN COB ASH FOR OIL–WATER SEPARATION

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UNIVERSITI TUN HUSSEIN ONN MALAYSIA

UNIVERSITI TUN HUSSEIN ONN MALAYSIA

STATUS CONFIRMATION FOR THESIS
DOCTORAL OF PHILOSOPHY

FABRICATION AND CHARACTERISATION OF CERAMIC HOLLOW FIBRE MEMBRANE FROM METAKAOLIN AND CORN COB ASH FOR FOR OIL–WATER SEPARATION

ACADEMIC SESSION: 2021/2022

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A thesis submitted in
fulfillment of the requirement for the award of the
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Special dedicated to my family and my best friends who give me support:

Kamarudin Bin Yusof

Noraini Binti Sulaiman

Nur Hafizah Binti Kamarudin

Nurfadilah Kamarudin

Siblings

Sahabat PROTEAM

ACKNOWLEDGEMENT

First of all, I deeply thank Allah, for his guidance and blessings. This thesis is the culmination of my journey of PhD which was just like climbing a high peak step by step accompanied with encouragement, hardship, trust and frustration. At this moment of accomplishment, I am greatly indebted to my research guide, Prof. Dr. Zawati Binti Harun, who accepted me as her PhD student and offered me her mentorship and care. Under her guidance I successfully overcame many difficulties and learnt a lot. The time spent with her will remain in my memory for years to come. Also, I greatly appreciate and acknowledge to Co Supervisors, Prof. Dr. Mohd Hafiz Dzarfan Bin Othman for his help, guidance, providing lab assistance equipment in Advanced Membrane Technology Research Centre (AMTEC), Universiti Teknologi Malaysia (UTM), and encouragement throughout my research progress.

Finally, I acknowledge the people who mean a lot to me, my parents, Noraini Binti Sulaiman and Kamarudin Bin Yusof and my supportive siblings, for their support, sacrifice, love and patience along years. I would never be able to pay back the love, help and affection showered upon by my sisters, my research buddies and also my best supporters; Nur Hafizah Binti Kamarudin and Nurfadilah Binti Kamarudin. Also, I am grateful to my best friends, Nurul Hidayah Binti Abd Latif, Nurfazliyah Binti Mohd Khozam, and Norhaslina Binti Hamid, who have consistently encouraged me throughout the study and the preparation of this thesis.

My sincere thanks go to all my lab mates from Integrated Material Process (IMP), Advanced Manufacturing and Material Centre (AMMC) and members in same area of research of membrane technology, Dr. Siti Khadijah Binti Hubadillah, Dr. Mohd Riduan Bin Jamalludin, Dr. Khairul Nazri Bin Yusof and also truthful research mate Dr. Najwa Binti Mustafa who have been very helpful by offering comment, advices and constructive discussion sessions. The most important persons in my research, technician labs, Mr. Anuar Bin Ismail, Mr. Mohd Tarmizi Bin Nasir, Mr. Fazlannuddin Hanur Bin Harith, Mr. Shahrul Mahadi Bin Samsudin, and Mdm. Faezahana Binti Mokhter for all the help. Finally, I would like to thank those who have contributed directly or indirectly toward the success of this research project.

ABSTRACT

Oily wastewater discharged into the environment causes serious global water pollution issues, which necessitates emerging recovery technologies, such as membranes for water purification. In this study, ceramic hollow fibre membrane (CHFM) derived from abundant waste materials of metakaolin (MK) and corn cob ash (CCA) were successfully developed via phase inversion/sintering techniques. As weakness of MK membrane is always associated to the brittleness property that lead to the reduction of strength value. Thus, further improvement by substituting a proportion of CCA into MK acts as pore-forming material and assist sintering mechanism in the preparing MK–CCA CHFM. The fabrication of CHFM involves CCA-based CHFM (H-NCA), MK–non-treated CCA-based CHFM (H-MNCA), and MK–treated CCA-based CHFM (H-MTCA) with processing parameters of ceramic powder ratio and ceramic powder content, bore fluid flow rate, and sintering temperature. Unstable H-NCA and H-MNCA membranes were observed during dope preparation and membrane spinning, owing to corn cob ash–potassium chloride (CCA–KCl) dissolution, which is significance as a green viscosity enhancer and pore agent in H-MNCA based on the precursor and sintered membrane matrix structure. The optimum MK:NCA ratio at 75:25 generated mechanical strength of 41.61 MPa and permeate water flux (PWF) of ~1159.93 L/m^{2}h. The final membrane preparation involved the TCA, produced the best H-MTCA asymmetric structure at MK–TCA of 45 wt%, bore fluid flow rate of 10 mL/min, and sintering temperature of 1150 °C. The performance tests showed a stable PWF (~266.52 L/m^{2}h), enhanced mechanical strength (82.78 MPa), and high oil–water separation efficiency (93.06%). Hence, CCA offered efficient sintering additive, not only formed in-situ reaction of phase network but also allow sintering at lower temperature, which preserved the sustainability technology.

ABSTRAK

Air buangan berminyak yang dibuang ke persekitaran menyebabkan masalah pencemaran air global yang serius yang memerlukan teknologi pemulihan seperti membran untuk pembersihan air. Dalam kajian ini, membran serat berongga seramik (CHFM) yang berasal dari bahan buangan metakaolin (MK) dan abu pulung jagung (CCA) telah dihasilkan melalui teknik penyongsangan fasa/persinteran. Kelemahan membran MK selalu dikaitkan dengan sifat kerapuhan yang menyebabkan penurunan nilai kekuatan. Oleh itu, penambahbaikan dibuat dengan mengantikan sebahagian CCA ke dalam MK yang bertindak sebagai bahan pembentuk liang dan membantu mekanisme persinteran dalam penyediaan MK–CCA CHFM. Pembuatan CHFM merangkupi CHFM berasaskan CCA (H-NCA), CHFM berasaskan MK–CCA yang tidak dirawat (H-MNCA) dan CHFM berasaskan MK–CCA yang dirawat (H-MTCA) dengan parameter pemprosesan iaitu kandungan/nisbah serbuk seramik, kadar aliran bendalir, dan suhu bakar. Ketidakstabilan membran H-NCA dan H-MNCA diperhatikan semasa penyediaan adunan dan penyemperitan membran disebabkan oleh penyahlarutan abu pulung jagung–kalium klorida (CCA–KCl) yang menunjukkan kesannya sebagai penambah kelikatan dan agen keliangan dalam struktur matriks bakar H-MNCA. Campuran komposisi membran yang optimum pada nisbah 75:25 (MK:NCA) mencapai kekuatan mekanikal 41.61 MPa dan kadar penyerapan air, (PWF) ~1159.93 L/m^{2}h. Penyediaan membran akhir melibatkan CCA yang dirawat, menghasilkan struktur asimetri H-MTCA yang terbaik pada kandungan seramik 45 wt%, kadar aliran bendalir 10 mL/ min, dan suhu bakar 1150 °C. Ujian prestasi menunjukkan PWF yang stabil (~266.52 L/m^{2}h), kekuatan mekanikal yang dipertingkatkan (82.78 MPa), dan kecekapan pemisahan minyak–air yang tinggi (93.06%). CCA bertindak sebagai bahan tambahan pensinteran yang cekap, bukan sahaja membentuk tindak balas *in-situ* rangkaian fasa tetapi juga pensinteran pada suhu yang lebih rendah serta dapat mengekalkan teknologi kemampanan.

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LIST OF SYMBOLS AND ABBREVIATIONS

A	Effective area of membrane (m^2)
^oC	Degree Celsius
c_f	Oil–water concentration in the feed stream (ppm)
c_p	Oil–water concentration in the permeate stream (ppm)
D_o/D_i	Outside diameter and the inner side diameter
F	Force
ρ_w	Density of distilled water at room temperature (g/cm^3)
L	Effective membrane length (cm)
t	Permeation time (h)
σ	Mechanical strength (MPa)
υ	Volume of membrane at in wet state (cm^3)
R	Rejection (%)
Ra	Surface roughness (µm)
Rb	Radius of spindle (cm)
Rc	Radius of container (cm)
V	Volume of permeated water (L)
w_d	Sample weight in dry condition (g)
w_w	Sample weight in wet condition (g)
ω	Angular velocity of spindle (rad/ec)
γ	Shear rate at surface of spindle (sec^{-1})
Al_2O_3	Alumina
$Al(OH)_3$	Alumina hydroxide
$AlF_3.3H_2O$	Aluminum fluoride trihydrate
AP	Additive polymer
BF	Bore fluid flow rate
BCFNO	$BaCo_{0.7}Fe_{0.2}Nb_{0.1}O_{3-\delta}$
BSE	Backscattered electron
CA	Cellulose acetate
CaO	Calcium oxide
CCA	Corn cob ash
CFA	Coal fly ash
CHFM	Ceramic hollow fibre membrane
CO	Carbon oxide
CO_2	Carbon dioxide
COD	Chemical oxygen demand
DPM	Diphenylmethane

DSC	Differential scanning calorimetry
FAS	Fluoroalkylsilane
$FeSO_4$	Iron (II) sulfate
FePc	Iron (II) phthalocyanine
Fe_2O_3	Iron oxide
$FeCl_3$	Iron (III) chloride
GC	Graphite composite
H-MNCA	Metakaolin–non-treated corn cob ash-based ceramic hollow fibre membrane
H-MTCA	Metakaolin–non-treated corn cob ash-based ceramic hollow fibre membrane
H-NCA	Corn cob ash-based ceramic hollow fibre membrane
HNO_3	Nitric acid
HPC	High performance concrete
K_2O	Potassium oxide
KCl	Potassium chloride\
LEPw	Liquid entry pressure
LiCl	Lithium chloride
LOI	Loss in ignition
MF	Microfiltration
MgO	Magnesium oxide
MK	Metakaolin
MIP	Mercury intrusion porosimetry
MMM	Mixed matrix membrane
MoO_3	Molybdenum oxide
MOR	Modulus of rupture
MTES	Methyltriethoxysilane
N	Nitrogen
Na_2O	Sodium oxide
NaOH	Sodium hydroxide
NaCl	Sodium chloride
NCA	Non-treated corn cob ash
NF	Nanofiltration
NH_4OH	Ammonium hydroxide
NMP	N-methyl-2-pyrrolidone
NS	Nanosphere
OM	Optical microscope
PACl	Poly aluminum chloride
PAN	Polyacrylonitrile
PESf	Polyethersulfone
POC	Palm oil clinker
POFA	Palm oil fiber ash
POTS	1H,1H,2H,2H-perflluorooctyltrir thoxysilane
PP	Polypropylene

PSF	Polysulfone
PSA	Particle size analyzer
PSD	Pore size distribution
PTFE	Polytetrafluoroethylene
PU	Polyurethane
PVA	Polyvinyl alcohol
PVC	Polyvinyl chloride
PVP	Polyvinylpyrrolidone
PVDF	Polyvinylidene fluoride
PWF	Pure water flux
RH	Rice husk
RHA	Rice husk ash
SCBA	Sugarcane baggase ash
SCC	Self-compacting concrete
SE	Secondary electrons
SEM	Scanning electron microscope
SiC	Silicon carbide
SiO_2	Silica
SPC	Sulfonated polycarbonate
TEOS	Tetraethylorthosilicate
TGA	Thermal gravimetric analyzer
TCA	Treated corn cob ash
TiO_2	Titanium dioxide
TMP	Transmembrane pressure
TOC	Total organic carbon
UF	Ultrafiltration
VOC	Volatile organic component
XRD	X-ray diffraction
XRF	X-ray fluorescence
Y_2O_3	Yttrium (III) oxide
γ-$Y_2Si_2O_7$	Yttrium disilicate
ZnO	Zinc oxide

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CHAPTER 1

INTRODUCTION

1.1 Research background

Oil–water mixtures are found in many industrial processes in form of a product, a by-product or a waste stream [1]. At a mean time, a large amount of water pollutants is discharged into the ground and surface water by the oily wastewater from industrial application on daily basis which causes severe ecological problems for humans, amphibious environments, and microorganism (as depicted in Figure 1.1). For instance, the long–term hydrophobicity of oily wastewater causes the pollution in agricultural soils and contaminated soils rarely absorb the water [2].

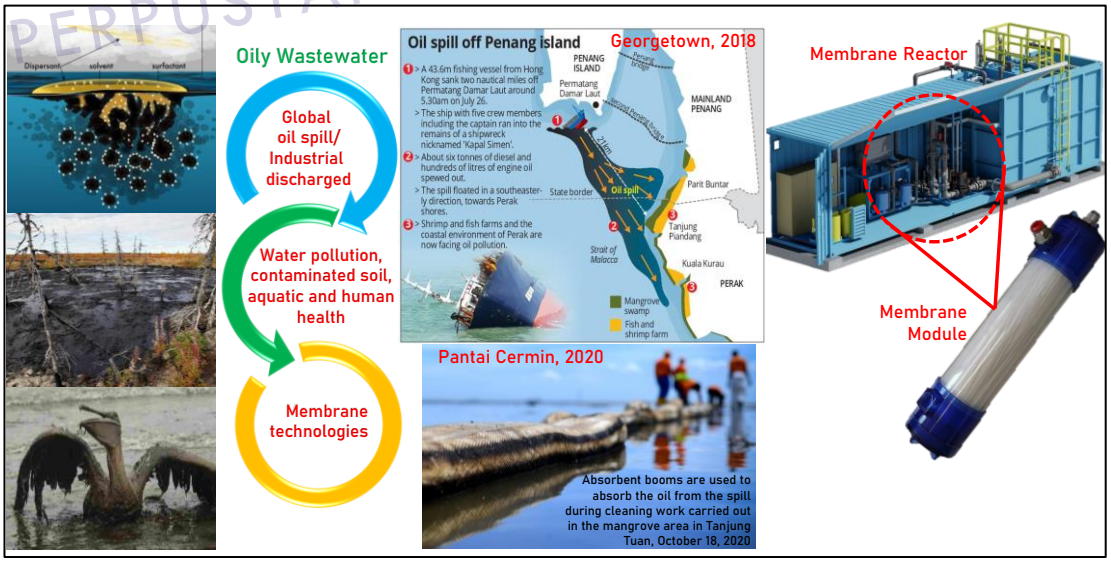

Figure 1.1: Oil–water separation using membrane technologies

The produced water treatment is significant from an environmental view to minimize waste disposal and for economic perspective, the water management

expenses can account for drilling costs about 5–15%. In recent times, membrane based separation processes ascertained to be effective for the oil separation from wastewater, although there are few discrepancies for the same process [3–5]. For example, reverse osmosis is restricted by the application of high pressure and greater fouling problem, resulted in a low permeability [6]. Likewise, ultrafiltration [7] and nanofiltration [8] also induces a slightly low permeate flux. Microfiltration is most favours in water treatment and oil–water separation from wastewater streams due to higher water permeability and low pressure requirements [9].

Nowadays, application of ceramic membranes has been increasing and circumvent the polymeric membranes utilisation. In 1989, Finnigan and Skudder [10] pioneering ceramic membrane for application of beer and recovery of extract through microfiltration process. At the same time, the treatment of oily wastewater using ceramic hollow fibre membrane (CHFM) via separation process is develop for more economical fabrication of ceramic membrane that offer many advantages, combined phase inversion and sintering technique. Dramatic reduction in the cost makes membranes economically competitive with traditional processes. In preparing of a good membrane, the properties such as high flux rate (permeability), high selectivity, ideal pore size, high surface area, low manufacturing cost, small thickness, and mechanically stable must be achieved.

In addition, ceramic membrane microfiltration has been widely studied as a membrane support for various applications such as gas separation, photocatalytic activities [11], hybrid membrane for combine separation–adsorption application [12], membrane contactor [13], and membrane distillation [14]. For example, Huang et al. [15] applied a coating method to prepare superhydrophobic layers on ceramic membrane from alumina for membrane distillation application. Also, previous work reported on preparation of tubular porous ceramic membrane from alumina through a phase inversion casting as membrane support for gas separation application [16]. However, ceramic membrane from high–purity alumina is expensive with high sintering temperature up to 1500 °C, making this ceramic membrane preparation is extremely pricey [17–19] (as shown in Figure 1.2).

Over the last decade, the exchanging trends of the economical ceramic raw sources usage have been studied extensively for various technological applications. Therefore, the ceramics manufacturing is also in search of developing waste material in its outcome productions. Numerous works of related area have been conducted to

reach this ceramics industry goal [20, 21]. As a result, waste by-products have been recognised as prospective materials for the ceramics production; i.e., fly ash [22, 23], bottom ash [24, 25], blast furnace slag [26, 27], glass waste [28, 29], petroleum waste [30, 31], water treatment sludge [32, 33] etc.

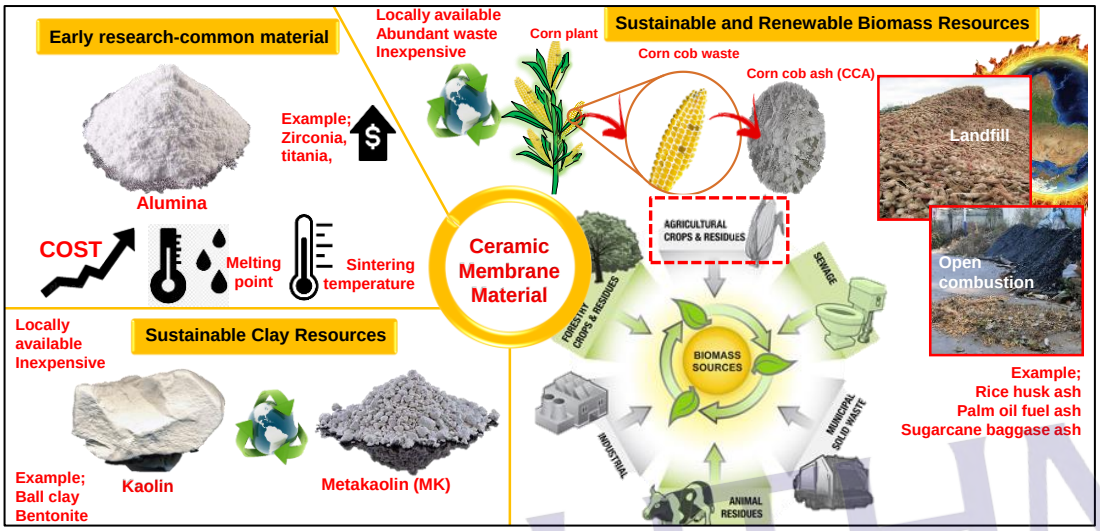

Figure 1.2: Advancement of ceramic membrane materials

Conventionally, low-cost ceramic membranes are prepared from clay and starch as the main raw material and pore agent (porogen), respectively, as investigated by Lorente-Ayza et al. [34]. Also, Hubadillah et al. [35] prepared low-cost ceramic membrane from kaolin clay through phase inversion and sintering technique to investigate the effects of kaolin content and particle size.

It should be mentioned that the world generates millions of tons of agricultural waste every day, e.g., rice husks, empty palm oil bunches, sugar cane bagasse, corn cobs etc. These abundant unused agro-waste are burned in boilers to generate energy, which at the same time produced ash with health hazard, cause lung disease if the ashes are disposed into open fields [18]. Considering its abundancy, rice husk ash, which highly consists of silica has been utilised as adsorbent–separator CHFM for efficient heavy metal removal [12]. In another application, activated carbon derived from agricultural sugarcane bagasse waste was applied for adsorbing lead ions from wastewate [36].

The preparation of low-cost ceramic membranes from clay and ash-based agricultural wastes have attracted more attention, as these natural materials highly consist of potential minerals to be developed as cheap alternative ceramic materials.

For example, metakaolin (MK) is classified as calcined clay type which produced from the calcination of kaolin [37].

Despite the already high applicability, the interests in the use of MK is more often reported in these recent years with lesser cost, mainly as substitution of cementitious materials in replacing fly ash [38]. MK is an alternative pozzolanic materials that are requisite for concrete properties improvement corresponding to durability and reducing the cement amount that commonly known as most expensive ingredients in concrete production. This is due to the fact that, in some country, the coal-fired power industry that producing fly ash will stop the production and thus, the applications of fly ash in the cementious will come to end in the next upcoming years [39].

Corn cobs also considered as a major agricultural by-product from the corn production which is typically burned after kernel removal from corn or maize [40]. Factories sort out corn seeds for further processing, leaving corn cobs as industrial waste. The biodegradation of corn cobs by microorganisms is a very long process, and incineration can be the simplest and fastest method to dispose corn cobs. However, incineration releases greenhouse gases (CO_2) into the atmosphere and causes global warming. Moreover, the smoke from the process affects public health. Thus, it is crucial for the corn industry to be concerned on environmental pollutions and to convert corn cob waste into highly valuable products.

From the literature, corn cob ash (CCA) has been widely used as a pozzolan in blended cement [41, 42]. CCA's pozzolanicity has been proven to meet the demand requirement on the strength activities [43]. It is also has been used as a supplementary replacement material that is able to improve the compressive strength and workability of cementitious samples [44]. The suitability of CCA as an alternative ceramic membrane material for CHFM fabrication, and as a reinforced material and pore-performing agent was investigated in this study via combined phase inversion and sintering techniques. The use of CCA not only provide an alternative ceramic membrane material but also a sustainable and greener production technology that minimize the contamination to the environment.

1.2 Problems statement

Alumina is mostly used as the base support of membrane material which is expensive and applied high sintering temperature towards various separation technologies. In the attempts to produce ceramic membranes from cheap materials, a number of researchers have utilised naturally abundant green materials. These materials can be easily extracted at low sintering temperature and known with their renewable nature, environmental friendliness and superior as compared to synthetic materials. MK meets the requirements as a new development of CHFM material, such as hydrophilic and porous properties with varying range of pore size distribution that benefit in permeate flux. It also offers a cheap abundance product. However, MK hollow fibre membrane offers low strength with a porous structure which requires high sintering temperature to achieve complete consolidation even for minimal mechanical strength.

Despite of CCAs complexity behaviour, they are useful during the phase inversion due to their naturally solubility in solvent and non-solvent medium. Interestingly, this has not been addressed in previous literature, and this benefits the flexibility control of thermodynamic dope suspension system and membrane spinnability process. Moreover, water leaching treatment has been studied to improve the properties of CCA.

Many works reported on potassium oxide (K_2O) high percentage in CCA, which induced a low melting temperature. The KCl originated from CCA seemly benefit in reducing MK-based CHFM sintering temperature with greater strength via vitrification process. The liquid phase sintering by CCA urged for high-diffusion rates, giving fast sintering and rapid complete consolidation at lower sintering temperature. Also, due to the high degree of mixing of finer powder particles, precursors often can be sintered to the final form at significantly lower temperatures, thus avoiding the higher sintering operation to accomplish adequate mechanical strength. Therefore, green CCA can be successfully implemented as an alternative supplementary material for ceramic membrane strengthening applications at low-sintering temperatures and powder loadings.

Moreover, the large amounts of oily wastewater discharge that could affected the groundwater resources, aquatic and human health is seems has a strong beneficial in implementing this membrane based-CCA structure. This is due to fluxing of CCA at lower sintering temperature that able to minimise and control the pore size with the

integration of hydrophilic properties, which strongly influenced the attraction of water towards membrane surface and rejected the oil molecule. This mechanism will generate the oil waste separation without any pressure or energy consumption, which indirectly introducing green approach in reclaiming water from oily wastewater discharge. For the above reasons, the aim of this study is to produce an alternative green MK–CCA CHFM which are environmentally friendly for water filtration and oily water treatment.

1.3 Research objectives

The main objective of this study is to develop an alternative ceramic hollow fibre membrane using phase inversion/sintering technique from green MK and CCA waste for oil–water treatment applications. This objective was achieved by accomplishing the following objectives:

1. To investigate the characteristics and properties of raw MK and CCA waste as the main ceramic materials for the CHFM fabrication.
2. To determine the optimum formulation of different proposed mixture (ceramic powder ratio and ceramic powder loading) and different controlled variables (bore fluid flow rate and sintering temperature) in CHFM fabrication via phase inversion/sintering technique.
3. To investigate the effects of different mixtures of membrane composition towards sintering kinetics mechanism and phase changes in the final CHFM sintered body.
4. To measure and analyse the CHFM performance and efficiency towards oil–water separation.

1.4 Research scopes

A series of experiments were performed to ascertain the effects of key process parameters and material factors on the preparation of CHFMs. In addition, a series of tests were conducted to characterise the raw materials and final products as a baseline for this study and for future studies. Hence, the scope of the study is summarised as follows:

1. Preparing and characterising waste environmental sources of MK and CCA as the ceramic materials for producing CHFM:
   i. Kaolin powder was fired at 650 °C for 4 hours at 10 °C/min to get MK powder.
   ii. Prior to use, corn cob waste was sun dried and then crushed to get 5mm of corn cob particles.
   iii. The ashing temperature of corn cob were set at 600, 700, and 800 °C, respectively, and kept for 8 hours at heating rate of 10 °C/min using a kiln furnace. However, CCA produced at 700 °C was selected for further processing and divided into two sample: 1) non-treated corn cob ash (NCA) was used as prepared, and 2) treated corn cob ash (TCA) was initially subjected to water leaching treatment and then oven dried. Both of powders were subjected to dry milling and sieved to get uniform powder (below than 25 µm).
   iv. The morphology and size of the ceramic powders were analysed using a scanning electron microscope (SEM)/elemental diffraction analyser (EDS).
   v. The chemical and physical properties of the ceramic powders were investigated using x-ray fluorescence (XRF), x-ray diffraction (XRD), thermogravimetric–differential scanning calorimetry (TGA–DSC), and particle size analyser (PSA).
2. Fabricating and characterising CHFMs by using phase inversion/sintering technique:
   i. CHFM was prepared into three samples: NCA-based CHFM (H-NCA), MK–NCA-based CHFM (H-MNCA), and MK–TCA-based CHFM (H-MTCA).
   ii. Ceramic suspension containing the ceramic powders (MK and CCA) as the main material (35–50 wt%), with N-methyl-2-pyrrolidone (NMP) as the solvent (44–59 wt%), Arlacel P135 as the dispersant (1 wt%), and polyethersulfone (PESf) as the binder (5 wt%).
   iii. Ceramic suspension viscosity at different ceramic powder ratios (MK:NCA; 100:0, 75:25, 50:50, 25:75, and 0:100) and loading (35, 40, 45, and 50 wt%) were measured and investigated on their rheological

effects towards membrane shape configurations and structure formation.

iv. Ceramic suspension was shaped into a CHFM precursor through a single-orifice spinneret using the phase inversion technique.

v. The effect of the spinning parameter (i.e., bore fluids flow rate of 5, 10, 15, and 20 mL/min) was investigated to find the perfect configuration shape and properties of the hollow fibres.

vi. The thermal temperature started from room temperature to 600 °C at heating rate of 2 °C/min for 2 hours, which then increased to selected temperature based of CHFM types (800, 900, 1000, and 1100 °C for H-NCA fibre membrane; 1100, 1200, 1300 and 1400 °C for H-MNCA fibre membrane; 1050, 1100, 1150 and 1200 °C for H-MTCA fibre membrane) at 5 °C/min and held for 3 hours.

vii. Cross sectional and surface morphological characteristics were analysed using scanning electron microscopy (SEM).

viii. The details of MK impact are not captured in this study, as the effect of newly CCA is more highlight towards the membrane fabrication.

ix. The mechanical strength of CHFM was measured at different ceramic powder ratios/loadings, bore fluid flow rates, and sintering temperatures using the 3-point bending test.

x. The porosity and pore size distribution of the sintered CHFMs were analysed using Archimedes principle and mercury intrusion porosimetry (MIP).

xi. The pure water flux (PWF) of CHFMs were investigated using cross-flow filtration.

3. Investigating and characterising the phase changes in CHFM:

i. The phase transformation of CHFMs prepared at different compositions and sintering temperatures were determined using x-ray diffraction analyser.

ii. The relationship of phase changes in CHFM were referred to the K_2O-SiO_2-Al_2O_3 ternary phase system and SiO_2-Al_2O_3 binary phase system.

4. Evaluating the performance test of the selected CHFMs towards oil–water separation using the fabricated membrane system:

i. Oil–water flux towards oil–water separation was measured.

ii. The separation efficiency of oil–water separation was investigated.

1.5 Significance of the study and novelty of work

1. The application of ceramic material is hindered as it has a high capital cost as well as the brittleness behaviour. Knowing that, MK and CCA waste have been transformed into valuable and useful material, especially in membrane applications, which definitely with lesser cost of production. The suitability of MK and CCA that possessed high hydrophilicity property able to attract and purify the contaminated wastewater efficiently.
2. The development of these new materials also naturally consisted of low burning materials KCl that can act as pore-forming agent of the porous structure in allowing better permeate flow.
3. Futher observed property with high strength phase and as viscosity enhancer that limit the inclusion of usage raw composition for the dope preparation has lead to the formation of low-cost materials with better performance at economical price.
4. The present development not only limit to the solution of reduce cost of production and environmental problems but also able to produce a high-value CHFM for highly efficient of oil–water separation application with potential in purifiying water production and oil resource recovery with better performance. This development is very crucial for our country which is known as one of the main contibuter in palm oil production industry. Thus, the interest in utilisation of these waste environmental sources is not only offered the development of newly low-cost ceramic materials and membrane but also able to enhance membrane properties and performance.
5. In fact, further benefit that can be obtained from the development of CCA also expected to guide an effective way and sharing knowledge in recycling abundant agriculture waste of corn cob. Further development and improvement towards new materials/product from our local sources are expected to drive the economy of the country as well as to benefit local people.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

According to World Population Prospects: The 2017 Revision, the population in Malaysia is expected to increase to more than 40 million in 2050 [45, 46]. Therefore, it is believed that urbanisation will cause a tremendous increase in produced water being released into the environment due to local usage demands and growth consumption. Therefore, the need for more efficient wastewater management arose for water cycle resolution. Being located in a rainfall-blessed region, Malaysia is not warranted to face water shortages in the years to come. Therefore, proper strategies need to be put in place to ensure water safety and security.

For example, for sewerage services, Indah Water Konsortium (IWK) Sdn. Bhd. is responsible in participating sewage treatment plants prior to discharge the sewage into rivers, which was done with high and strict compliance to preserve the environment. However, the cost of cleaning the environment far exceeds the costs involved in preserving it, as the cost of treating wastewater is increasing. Therefore, due to the impact of wastewater on the environment, the utilisation of membranes with lesser cost and with sustainable technologies in wastewater treatment has attracted into foremost attentions.

At some point, many membrane research modifications have gained the attention of research for controlling the material composition of membrane, the type of materials, the use or influence of additives (such as inorganic precursors on the morphology), the stability and the porous texture of the inorganic matrix, and even controlling the processing variables. All these factors are always interlinked and

associated with each other to bring the establishment of quality and the targeted membrane properties and performance.

2.2 Overview of oily wastewater industry

Worldwide oil spills and industrial oily wastewater introduce various toxic compounds into clean water systems which subsequently threaten the global ecosystem [47, 48]. Water pollution caused by oily wastewater effluents is largely produced from the rapid industrial growth of oil and gas, pharmaceutical, metallurgical, petrochemical, and food industry sectors. Indeed, the necessity to treat wastewater is quite challenging and inevitable. In addition, the increased demand for clean water, particularly in water-stressed areas is the result of the rapid growth in economy and population, as well as the need of future generations [49].

From ecological safety perspective, the oil–water emulsions contribute to a bigger destructive impact to the ecosystems and the environment towards aquatic and human life, which necessitates the oil separation from oily wastewater [50]. Padaki et al. [51] described the organic compounds in oily discharge water containing the amount of nitrogen (N) content less than 3% sulfur content molecule 0.3–10%. While, oxygen content molecule is usually less than 4.8% [52]. Other contaminants at wide range are also being discharged together with oil. It is important to know that high amount of dissolved oxygen in discharge wastewater affects the algae productivity as it is very important link in the food chain. Furthermore, the required amount of oxygen is 2 mg/L in order to sustain a safe and normal life for aquatic environment [51].

High level of organic matter in oily wastewater discharge into water bodies can caused the fatal condition to microorganism due to oxygen excess consumption [51]. The out coming of this matter will leads to maintenance of higher life forms. This oily sludge water is not only contributes a side effects in aquatic environment, but also in agriculture sectors, i.e., soil pollution. This is due to the physical and chemical properties changes as it enters into the soil and consequently effects the soil morphologies. As a result, plants grow with a fewer nutrients with seed germination obstruction and plants restriction growth [53]. Additionally, wastewater pollution also weakening the crop production and natural landscape devastation [54].

Oily water constituents are categorised based on oil droplets size, namely, free oil (>150 mm), dispersed oil (20 to 150 µm), and stable emulsified oil (<20 µm) [55]. In this manner, government agencies and pollution control boards have prepare a strict control guidelines for the maximum oil/grease concentration in discharged industrial effluents. Therefore, many academic researchers and industrial experts have put high efforts in finding solutions for oily wastewater treatment with treated water released concentrations below the standard discharge limit (5–10 mg/L) [56]. Table 2.1 listed on the produced oily water discharge from several industrial sectors.

Table 2.1: Produced oily wastewater from industries

Types of oily water	Oily water concentration (mg/L)	Ref.
Refractory oily wastewater	100–200	[57]
Vegetable oil refinery industry wastewater	2648–3880	[58]
Palm oil mill effluent	250,000	[57]
Crude oil	500	[59]
Synthetic oil (lubricant oil (REPSOL ELITE TDI 15 W40) + soluble oil (SOL 1000)	1500–6000	[60]
Synthetic oil (paraffin oil + Oleic acid + Triethanolamine)	200	[61]
Restaurant oil water in Hong Kong	100–2100	[59]
Drilling wastewater from Barekuri, India	35	[62]
Refinery plant	26	[9]
Oil and gas	50–1000	[63]
Fish meal plant	1210	[64]
Petrochemical	250	[65]

Necessary actions for wastewater treatment are not only for reusing and recovering the oil for economic gains but also for oil phase environmentally friendly disposal [55]. A preferable solutions in separating oily wastewater are more effective compared to oil burning, which address to the global environmental issues. It is considerably benefit in reducing environmental pollution, as well as resource recycling approach and energy saving [66]. In point of sustainability, oily wastewater management has executed a considerable costs and solving challenges so that a cost-effective and energy-efficient separation techniques should be implemented seriously. Among the contaminants found in oily wastewater, oil and grease are the most challenging task for wastewater separation.

2.3 Conventional oily wastewater treatment

In the last two decades, there are several methods used for the separation of oil from oily wastewater, including air flotation [67, 68], coagulation [69, 70], gravity separation [71], de-emulsifications process [72] and flocculation [55] (Figure 2.1). Most of these methods are reported as not efficient in following pollution control agencies standards, especially when involving low oil concentration. Additionally, streams/products of secondary waste are also produced in large quantities, and oil emulsions droplet size must less than 20 µm for efficient separation [73]. Crucially, these conventional methods have some drawbacks, such as using toxic compounds, requiring a large space for installation, having expensive operations and generating secondary pollutants [51].

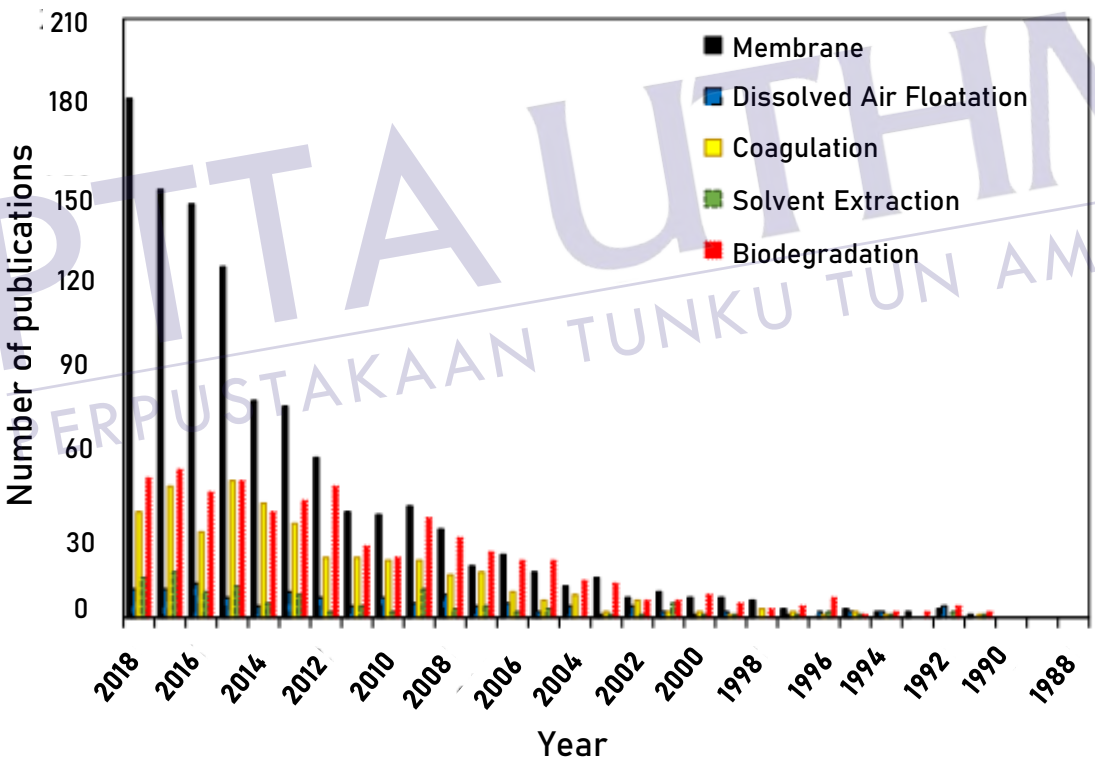

Figure 2.1: Reported of oil–water treatment technology from 1988 to 2018 from Web of Science [55]

Prior to the discharge of produced water into the environment, region-specific regulations have been initiated to enforce the oily wastewater treatment applications [55]. Table 2.2 is summarize of the discharge oily wastewater regulations. Hence, there is an urgent needs to develop technologies that reach treatment goals cost-effective

according to the regulations. The common technologies innovation for treating oily waste water are discussed in the next section.

Table 2.2: Regulations for discharging oily wastewater

Country/Sea	Regulations	Upper limit of discharged oil concentration	Ref.
North East Atlantic	Oslo Paris Convention (OSPAR)	30 mg/ L of oil and grease	[74]
Paris	-	40 mg/L for the offshore fields 5 mg/L for the on-land fields (discharged to the sea)	[75]
United States	Environmental Protection Agency (EPA)	72 mg/L for any 24 h period 45 mg/L over a 30 days period	[76]
		40 mg/L and typical range around 10-15 mg/L	[77]
Norway	-	30 mg/L	[78]
China	-	10–15 mg/L	[78]
Malaysia	Malaysia Environmental Quality (Sewage and Industrial Effluent) Regulation 1979	5 mg/L inland waters within listed catchment areas (standard A) 10 mg/L for water discharge into any other inland or Malaysian waters for standard B	[79]

2.3.1 Flotation

A floating mechanism significantly reduces the overall density of oil/solid particles by aggregating them with gas bubbles [80]. The tiny air bubbles adhere to the oil particles suspended in water, and because the floating density of oil is less than that of water, the formation of a scum layer separates from the water [81] (Figure 2.2). These aggregates will rise rapidly and eventually skim off. The mechanism is also suitable for the removal of oil droplets with particle sizes of more than 20 µm and with an average effluent concentration of 10–40 mg/L [80].

In flotation technology, induced and dissolved gas flotation systems are the most regularly applied. These are very effective in floating small suspended particles (organic matter, oil and grease). Moreover, fine particles as small as 3 µm can be removed using this coagulation technology [82]. Typically, waste influent with oil concentration lower than 1000 mg/L can be treated using the system. However, the disadvantages of this method are that the dissolved oil is difficult to be removed and chemical pre-treatment is usually required to remove the emulsified oil. It is also not cost-effective to produce large volumes of microbubbles that are smaller than oil droplets [80]. Painmanakul et al. [83] studied the modified induced air flotation process

for oily wastewater containing anionic surfactants. The study presented that pH value, alum dosage, and gas flow rate were related to removal efficiency as considered in terms of chemical oxygen demand (COD).

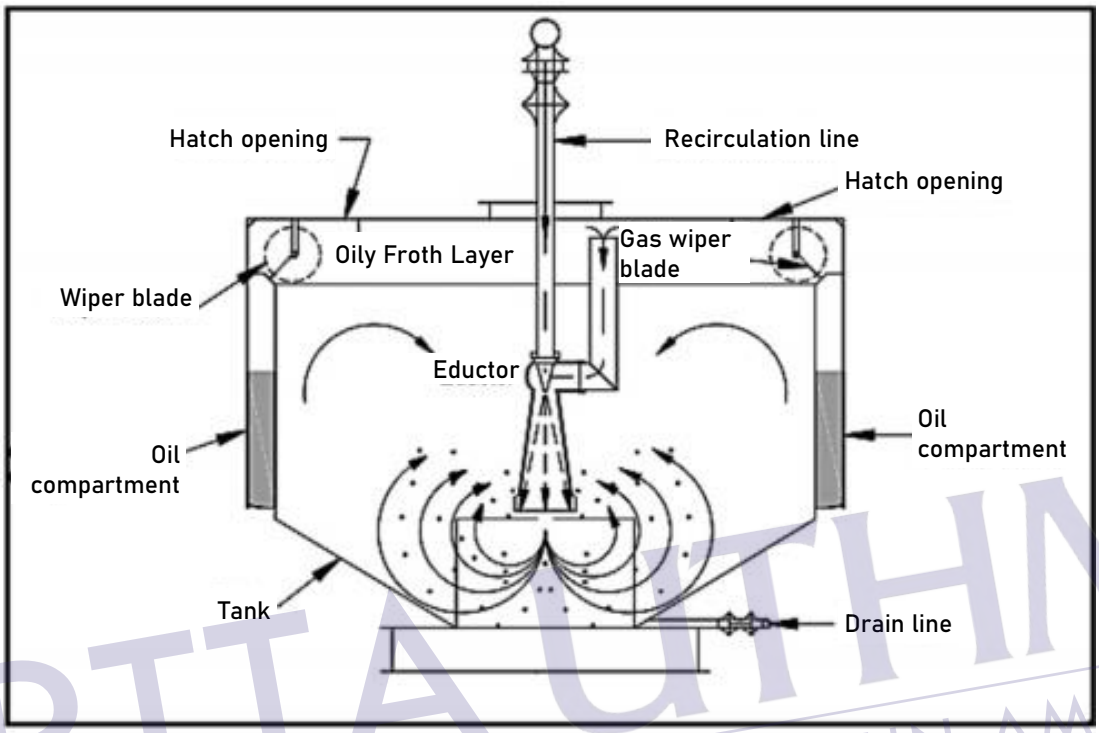

Figure 2.2: Induced gas flotation system [81]

2.3.2 Biological treatment

The use of microorganisms in treating oily wastewater has yielded some impressive results [84]. The experimental setup sytem can be illustrated in Figure 2.3 via single-phase system [85]. A consortium of microbes is usually used in oily wastewater treatment process in order to eliminate hazardous pollutants. Even though this biological treatment is not so popular or well developed as other techniques due to the unstable nature of the microbes, there are findings that showed notable contaminant removal percentages from oily wastewater [54].

Nopcharoenkul, Netsakulnee & Pinyakong [86] developed a bio-treatment using Pseudixanthomonas sp. RN402 to degrade crude oil, diesel oil, n-hexadecane and n-tetradecane, respectively with efficiency of 83, 89, 65 and 92%. Meanwhile, Chanthamalee, Wongchitphimon & Luepromchai [87] studied the treatment of bilge

water using polyurethane foam (PUF)-immobilized Gordonia sp. JC11. They found that PUF–immobilized bacteria performed was efficiently in removing oil and able to remove boat lubricant at 40–50%. Hidalgo, Martin-Marroquin & Sastre [85] demonstrated the co-digestion of residues and pig manure in the oily vegetable waste treatment without chemicals addition by co-digestion of substrates.

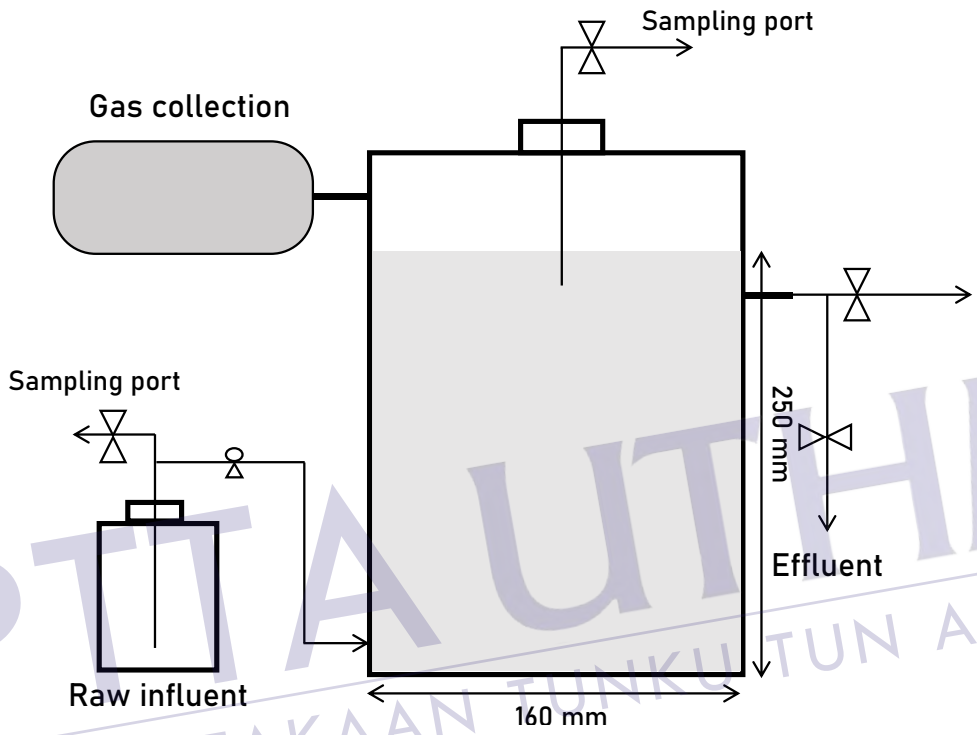

Figure 2.3: Experimental set-up in lab-scale single-phase system [85]

2.3.3 Coagulation

Coagulation is commonly applied in industrial wastewater treatment by using coagulants. Chemical coagulation with certain chemicals can bring non-settleable particles together to become a larger heavier mass of floc or solid material, which then is removed [88]. The working mechanism of coagulation can be simply explained by referring Figure 2.4, as coagulant is added into wastewater tank, it will precipitate and trap the impurities which then settle to the bottom tank for further treatment processing. Unfortunately, the presence of the precipitatation of impurities in the treated water needs another procedure for clean water separation, which presents the most challenging task and becomes a drawback for this method as it is time-consuming and costly.

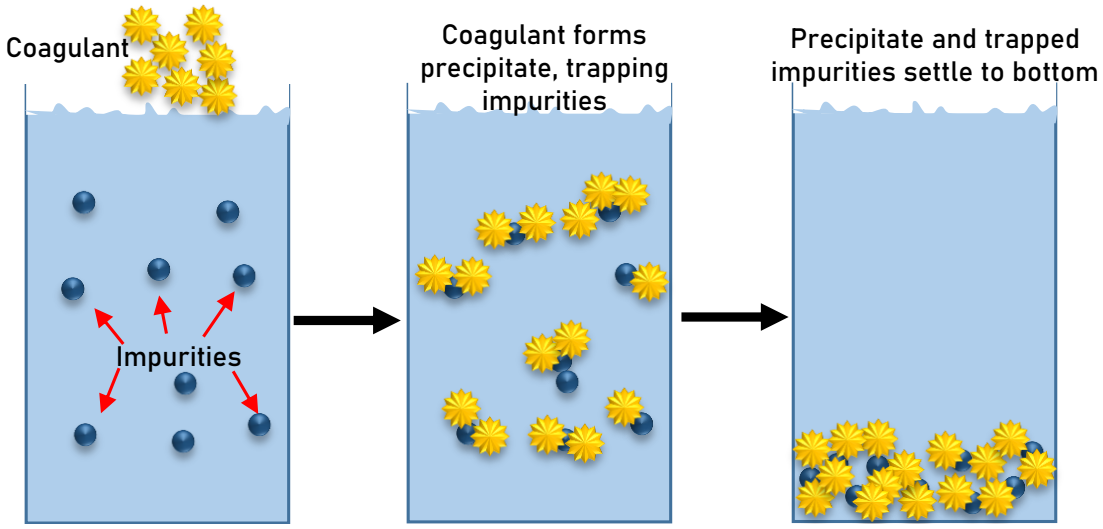

Figure 2.4: Coagulation process employed water treatment [88]

Cong, Liu & Hao [89] used poly-aluminum zinc silicate chloride for the treatment of oily wastewater. Turbidity, chromaticity, and COD were respectively given as 98.9, 91.3, and 71.8%. This conventional method is gradually being replaced by advanced hybrid techniques of coagulation/membrane, particularly in oily wastewater treatment. Coagulants are added earlier into the feed-water before it is enters the membrane module, as at this point sedimentation is omitted. The organic matters that accumulate on the membrane surface can be aggregated and escaped the fouling cake layer that easily remove by hydraulic cleaning [90]. Hence, membrane fouling can be controlled with enhanced filtration performance [91].

Rasouli, Abbasi & Hashemifard [88] applied coagulation–MF hybrid process using mullite–alumina-zeolite and mullite–zeolite ceramic microfiltration membranes for synthetic oily wastewater treatment. Results disclosed that iron (II) sulfate ($FeSO_4$) with concentration of 200 mg/L is the best coagulation with excellent performance for both membranes. In particular, a hybrid coagulation as pre-treatment use coagulates for oil removal such as poly aluminium chloride (PAC1) and casein [92], aluminum and iron salts [88] and PACl, aluminum chlorohydrate and iron (III) chloride ($FeCl_3$) [93] and aluminium and ferric sulfates [94].

2.3.4 Membrane

Noteworthy separation systems that can separate most oil particles from water are regularly used for initiating oil–water separation. For that reason, the remaining oil in

water must be fully eliminated with not even in a small amount at an acceptable limit, before the water resources are discharged into rivers or the sea or reinjected for water flooding [95]. The research on membrane-based separation has started since 1973, and it is has been recognised as the most cost-effective technology for removing oil droplets with sizes smaller than ~10 µm [96]. Membrane separation processes achieve great advantages of no chemical addition and the absence of oily sludge [97]. Many works have reported on the ability of membrane separation techniques that successfully meet these specification.

These days, the use of membrane separation processes have outstandingly grown in many industrial applications, owing to the properties and efficiency in many separation processes, including industrial effluent water treatment [98]. Being able in solving an environmental problems in related area with low-cost management has made membrane technology become more acknowledged [99]. Due to its high selectivity, good productivity, fouling resistance, stability, efficient performance, and reliable separations, membrane separation is the best system to protect water resources apart from being economical [79]. Ever since the membrane technology was introduced, it already demonstrated efficiency in separation applications and competitiveness in water purification, as compared to conventional techniques. However, it should be noted that efficiency and compatibility are entirely determined by the membrane properties itself.

Membranes separation can be defined as a very simple concept as illustrated in Figure 2.5. They acts as a selective partition of semi-permeable layer between two phases which then regulates the transportation of those two phases under the effect of a driving force. In a simple way, feed will permit and transports through the membrane and at the some point, the suspended solids and other substances will be restricted and not able to pass through it [51]. A transfer force can be generated by a gradient of pressure or applied of electrical potential to induce the membrane permeation. Permeate is referred to the fluid part that passes through the membrane, while retentate is the liquid with retained elements [99].

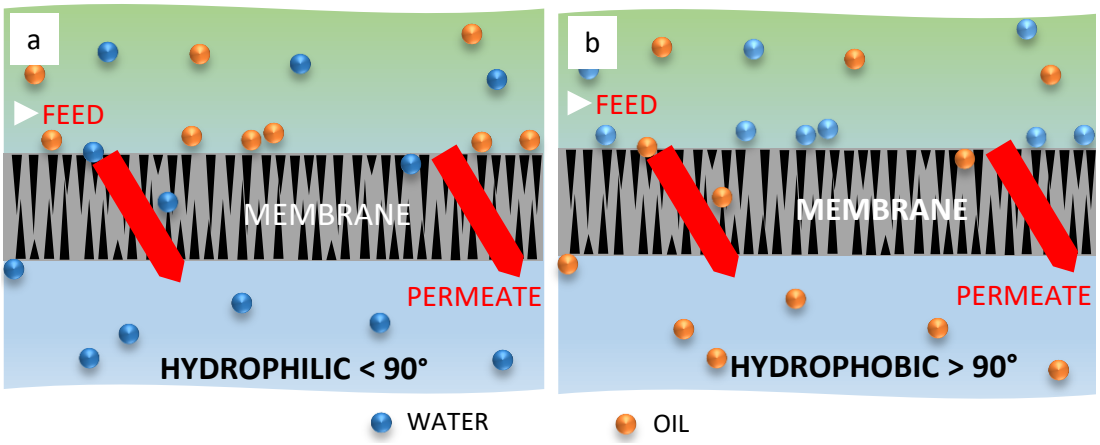

Figure 2.5: Membrane separation mechanism

Suresh, Pugazhenthi & Uppaluri [100], in comparing membrane filtration techniques, microfiltration membranes (MF) is suggested more favoured for oil–water separation rather than nanofiltration (NF) and ultrafiltration (UF) membrane. This was due to the wide range of pore size and also the narrow pore size distribution in the MF membrane, which can contribute to high flux with better rejection [101]. During the filtration process, the NF and UF membranes needed to be regularly stopped for membrane cleaning in order to restore membrane permeability due to pore blockage by the cake layer of oil or the fouling mechanism. Normally, concentration polarisation and adsorption of oil foulants on the membrane surface were major reasons for the fouling issue [102].

Oil rejection efficiency or the separation performance of oil–water emulsion depends on several factors, such as membrane properties, operating parameters (cross flow rate and applied pressure), and feed oil properties (initial feed concentration and feed temperature) [100]. Membrane pore size has an interaction with oil droplet size. According to Almojjly, Johnson & Hilal [103], membrane pore size is likely determined oil–water emulsion filtration properties, which depended on oil droplet size related to oil concentration. Zanatta et al. [104] found that a higher oil rejection performance of oil separation was due to the larger particles of oil instead of the pores of the microfiltration ceramic membrane.

Tai et al. [105] suggested that the membrane flux of oil–water emulsion was significantly low as compared to water flux due to the higher viscosity of the oil emulsion. Theoretically, membrane flux enhances with increasing driving force during filtration. On the other side, the flux reduces with increasing feed oil concentration. A concentrated oil refers to greater oil particles in emulsion, which increase the pore-

blocking rate of the membrane surface, causing fouling. Most reported studies focused on oil concentration equal to or higher than 1000 ppm for treating oily wastewater [7, 63, 106].

At high pressure, oil droplet wetting is enhanced and then coalesces to permit greater transportation of oil droplets through the membrane porous structure. As a consequence, a reduced rejection rate performance is obtained. On the other hand, membrane rejection improves with increasing feed concentration, owing to increase oil droplet size and oil emulsion density. Fouling control or membrane cleaning definitely will increase operation cost, as well as process complexity, thus making membrane separation processes less competitive in many circumstances [107].

Therefore, enhanced hydrophilicity of membranes are more often reported for treated oil–in–water emulsions to acquire clean water rather than membranes with hydrophobicity characteristics [108, 109]. This is because, a hydrophilic material is less sensitive to adsorption and fouling properties as compared to a hydrophobic material [110]. This membrane acquires a surface contact angle value of less than 90° (Figure 2.5 (a)). Aside from hydrophilic membrane fabrication with high water flux for oil–water emulsion, oleophobic characteristic also should be considered for reducing oil adhesion onto the membrane surface. This is due to the higher surface energy of the hydrophilic surface as compared to the surface energy of the biological and organic foulants in water. As a result, pore blockage occurs because oil surface tension is lower than that of the membrane surface [79].

On the contrary, a hydrophobic membrane (contact angle>90°) has been used for oil adsorption (Figure 2.5 (b)) [111, 112] and at the same time prevents water molecules from pass through it [113]. Moreover, to adsorb oil droplets, a hydrophobic membrane ideally attains a high value of water contact angle. At the same time, the membrane must possess an oleophilic characteristic due to the low oil contact angle.

Above all, special wettability-oriented materials for oil–water separation are identified as superhydrophobic/superoleophilic, which refers to oil–removing materials, or superhydrophilic/superoleophobic for water–removing materials. Membranes wettability-regulated separation methods demonstrate numerous advantages over conventional separation methods due to their good reusability and high separation efficiency. Prior to this, membrane has been a promising for oil–water treatment, as well as oil–water emulsion [114].

2.4 Recent advances in membrane technology towards oily wastewater separation

Nowadays, the membrane technology is known to be a "worldwide technology", owing to its cost-effectiveness, sustainable technology, simple operation, and high energy efficiency. Typically, membranes are made from polymeric materials and inorganic (ceramic) materials. They are produced in diverse of configurations, such as disk, spiral, tubular, and hollow fibre structures. Each type of membrane configurations possesses a varying degree of separation. In terms of compatibility and competency, hollow fibre membrane configuration usually offers compact modules with a high effective surface area [51]. For years, the applications of polymeric and ceramic membranes are emerging and effective in separating oil particles from produced water.

2.4.1 Polymeric membrane

Numerous studies have conducted on treating oily wastewater using polymeric membranes. The most typically used polymer materials for MF/UF membranes preparation are polyacrylonitrile (PAN) [115], polysulfone (PSf) [116], cellulose acetate (CA) [117], polyvinylidene fluoride (PVDF) [118], polyethersulfone (PESf) [119], and polytetrafluoroethylene (PTFE) [120]. The common structure of polymer membrane can be demonstrated by the PESf membrane, which mainly consists of a dense top layer and a porous support of finger-like voids (Figure 2.6) [119]. The advantages of the polymeric membrane technology are offering high-efficiency of particles removal, low energy requirements, and most significantly are inexpensive to that of ceramic-based membranes [51].

However, these membrane types possess some drawback in their utilisation as membrane separation, such as ineffectiveness in volatile-compound separation. They also tends to suffer from deterioration in flux and rejection during oily wastewater treatment, as severe fouling easily occurs for polymeric membranes [121]. Therefore, to overcome this phenomenon, a significant number of polymer formulations and alterations have been developed to enhance the hydrophilicity, antifouling properties and membrane performance, for examples, polymer-blending [122], membrane surface modification [118] and nanoparticle-incorporation in the membrane matrix [119].

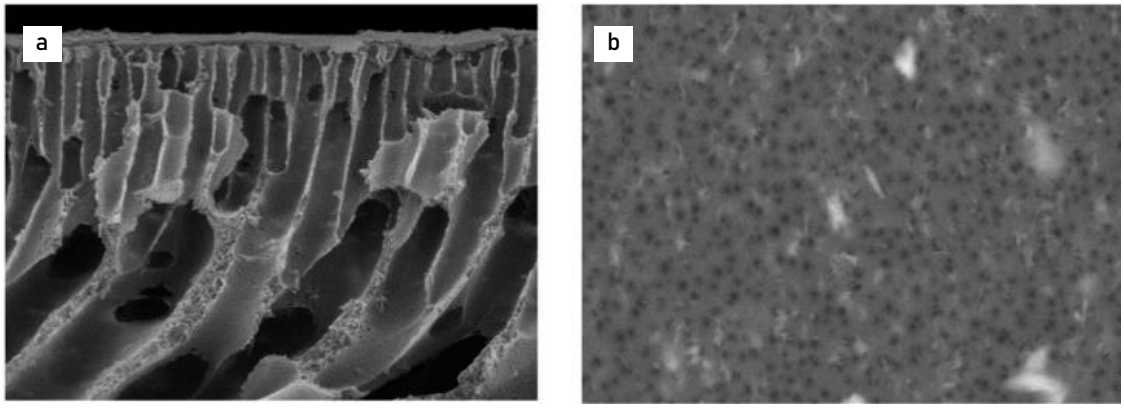

Figure 2.6: Morphological of PESf membrane; a) cross sectional and b) outer surface membrane [119]

Despite prior usage of hydrophobic neat polymers in treating oily wastewater, the hydrophilicity characteristic of the membrane is more promising, owing to easy water molecule transportation and reduction of oil droplets at the end of the produced treated water. Moreover, oil adhesion on the hydrophilic membrane surface could be lessened, subsequently resulting in membrane fouling reduction and water productivity improvement [123]. Unfortunately, the implementation of hydrophilic polymeric membrane is not an easy task. This is because of the accumulated oil formation on the membrane surface due to emulsion flow force during oil emulsion filtration. As a consequence, fouling is formed by oil accumulation that penetrates into the membrane pores. This fouling layer will block the membrane pores and reduced the flow ability of water, as accumulated oil molecules or foulants restricted water transportation [79].

Polymer blending is produced by mixing a dope solution that consist of two or more polymer types to improve membrane morphology, hydrophilicity wettability, and fouling resistance. A research by Zhu, Lu & Bai [124] found that blending or immobilized a hydrophilic substances (additive polymer (AP)) onto hydrophilic membrane resulted in the hydrophilicity/oleophobicity and thus minimized the membrane fouling. Meanwhile, PVDF/sulfonated polycarbonate (SPC) membranes have been successfully developed by Masuelli [125] via the same method. The blended membrane established a high oil rejection efficiency of 97.8% and permeate flux of 28.59 L/m^{2}h at 20 wt% of PVDF/PC blending. Zhu et al. [126] have fabricated a superhydrophilic zwitterionic polyelectrolyte/PVDF–blend membrane with superoleophobicity characteristics owed to zwitterionic sulfonate group presence on the membrane layer.

In recent years, the mixed matrix membrane (MMM) fabrication method is reported as a compromise to polymer blending due to low fouling and the ability to remove specific contaminants. MMM aims for the enhancement of overall efficacy by combining advantageous characteristics of two types of material [79]. For example, Chen et al. [127] incorporated iron (II) phthalocyanine (FePc) into a PVDF membrane. Membrane hydrophilicity increased, as well as mean pore size and membrane porosity with enhanced membrane antifouling. This was due to the charged surfaces of Fe repulsing similarly charged foulants more strongly which is excellent for fouling resistance.

In another study, a low-cost bentonite nanoclay was mixed into a polyvinyl chloride (PVC) membrane to prepare a high hydrophilicity membrane, giving superior permeate flux (94 L/m^2h) and oil rejection (92.5 %). Evidently, bentonite enhances the membrane properties of surface roughness, pore density, and porosity. However, long-term membrane operation is limited due to the occurrence of fouling behaviour by the bentonite nanoclay [128].

On the other hand, PAN is a promising candidate for oil–water separation due to its high water flux and separation efficiency. Zhang et al. [129] prepared a UF PAN membrane via the sodium hydroxide (NaOH)–induced phase inversion process. Membrane wettability improved by the surface hydrophilicity, as well as membrane surface roughness with ultrahigh water permeation (2270 L/m^2h) and oil separation efficiency (85%). In order to replace the expensive PAN material, Zuo et al. [118] proposed MF polydopamine–coated PVDF membranes for hydrophilicity surface modifications with high permeation flux of > 2600 L/m^2h and oil rejection rates of 98.5%.

On the other hand, hydrophobic membranes is literally block the passage of water and are commonly used for oil adsorptions. Sadrzadeh, Gorouhi & Mohammadi [130] observed on separation of oil from oily wastewater using PTFE hydrophobic membrane at different feed concentration, operating temperature and pressure, and feed flow rate towards permeate flux and water rejection efficiency. In contrast, Karimnezhad [131] studied water flux of hydrophobic membrane after the membrane was continuously filtered for almost 1.4 hour. The results showed about 20% reduction of water flux from its initial value and ultimately became constant. This result obeyed the hydrophobic membrane working purpose in reducing the water passing.

The fabrication of a porous substrate of an electrospun polyurethane (PU) membrane by stacking nano or micro fibres to form a hydrophobic surface has been deliberately explored in these past years [132, 133]. The hydrophilic epidermis is imparted onto the membrane chemically modified with hydrophobic agents, i.e., polydimethylsiloxane [134], fluorolkylsilane [135], and dimethyldiethoxysilane [135]. Gu et al. [136] indicated that a membrane water permeation could be easier due to the polar groups in the polyurethane soft segment. So that, permeate water could be blocked by membrane grafting by hydrophobic silica particles to produce a low-energy epidermis and a rough surface. The resultant membrane exhibited hydrophobicity with high water contact angles, durable water repellency, superoleophilic and had high permeation fluxes for various oils separation testings.

Liu et al. [137] fabricated a coated polypropylene (PP) membrane with TiO_2 and 1H,1H,2H,2H-perflluorooctyltrir thoxysilane (POTS) by multi-layer deposition. The modified membrane displayed a superhydrophobic/superoleophilic characteristics and good surface stability in the acid environment. The membrane was applied for water–in–oil emulsion separation with rejection efficiency of higher than 80 % solely using gravity.

2.4.2 Ceramic membrane

Inorganic–ceramic membranes have been developed for several process applications. For example, a well-known research group led by Li adopted a phase inversion technique to produce ceramic membrane for multiple applications, namely ultrafiltration, nanofiltration, microfiltration and gas separation, and also serves as porous support for dense membrane formation [138, 139].

Ceramic membrane is more preferably applied compared to the polymeric membrane due to their better resistance over chemical attack, thermal stability, long life span [140], microbial degradation resistance [141], mechanical stability [142], easy clean regeneration, and high separation efficiency [143]. Hence, they are gradually applied in a number of industries, particularly for water purification of sectors from municipal, textile, domestic, printing and dyeing, chemical, and oily water treatment [144].

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