SUPERHYDROPHOBIC-SUPEROLEOPHILIC KAOLIN BASED MICROFILTRATION MEMBRANE FOR OIL RECOVERY FROM OILFIELD PRODUCED WATER

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ABSTRACT

The discharge of oilfield produced water (OPW) causes disruption of the ecosystem and environmental degradation. Herein, novel hybrid membrane coupled absorption-filtration technology is proposed for the recovery of oil from OPW. The present study aims to develop a superhydrophobic-superoleophilic kaolin-based hollow fibre ceramic membrane using phase inversion and sintering technique for the recovery of oil from synthetic OPW. To achieve the superhydrophobic-superoleophilic modification, organosilanes sol-gel coating was performed on kaolin-based hollow fibre ceramic membranes. Membrane morphology and surface roughness was analysed using field emission scanning electron microscopy (FESEM) and atomic force microscopy. The membrane surface functionality was studied using Fourier transform infrared, X-ray photoemission spectroscopy (XPS) and X-ray diffraction analysis. The membrane filtration performance was evaluated using cross flow module. In the first stage of the work, feasibility studies of Malaysian kaolin (MK) and Nigerian kaolin (NK) were studied on fabrication of kaolin-based hollow fibre membrane by varying the loading composition (34 to 37 wt.%) and sintering temperature (1200 to 1500˚C). Experimental results show that increase of kaolin concentration and sintering temperatures decreases the flux rate. The physiochemical and performance analysis showed that 34 wt.% MK ceramic membrane exhibits better water flux (565.06 L/m^{2}h) with desired pore size and stability than 34 wt.% NK membrane. It owes to the MK which hold higher degree of crystallinity and smaller particle size. In the second stage, for effective oil absorption-filtration, organosilane agents such as methyltriethoxysilane (MTES), fluoroalkylsilane, octadecyltrimethoxysilane, chlorotrimethylsilane chlorotrimethylsilane, and trichloro(octadecyl)silane were used for the modification of superhydrophobic-superoleophilic kaolin hollow fibre membrane. XPS and FESEM analysis clearly indicated that the organosilanes are bound firmly on the surface of kaolin membranes. The effect of coating cycle and oil concentration were also studied. Among the coated membranes, MTES coated kaolin membrane showed the maximum water contact angle of 161.3° and lowest oil contact angle of 0^o. Resultantly, this depicts that the superhydrophobic-superoleophilic property were attained. In the third stage of the study, the oil recovery performance of the kaolin membranes with different organosilane agents were evaluated and compared. MTES-coated membranes showed maximum oil absorption capacity of 10 g/g, oil flux of 80 L/m^{2}h, and oil separation efficiency 90%. The optimized MTES coated membranes were adopted to further optimization of process condition (oil concentration, feed flow and feed pH) in cross flow module for the effective oil flux and separation efficiency using response surface methodology (RSM). From the central composite design, maximum oil flux of 97.67 L/m^{2}h and separation efficiency 98.41% were observed at oil concentration of 50 mg/L, feed flow of 300 mL/min, and feed pH of 4. The RSM model was good coherent with experimental data. Overall, this study portrays the development of economically viable superhydrophobic-superoleophilic kaolin hollow fibre membrane for the absorption combined filtration process for the separation of oil from produced water. This study would pave the way for researchers to eliminate the pollutants using hybrid absorption-filtration process.

ABSTRAK

Pembuangan air keluaran dari medan minyak (OPW) telah menyebabkan berlakunya gangguan terhadap ekosistem dan kemerosotan alam sekitar. Oleh itu membran hibrid baharu yang digabungkan dengan teknologi penyerapan-penapisan telah dicadangkan untuk perolehan minyak daripada OPW. Kajian ini bertujuan untuk menghasilkan membran seramik gentian berongga berasaskan kaolin superhidrofobik-superoleofilik menerusi penggunaan teknik penyongsangan fasa dan pensinteran bagi perolehan minyak daripada OPW sintetik. Bagi melaksanakan pengubahsuaian superhidrofobik-superoleofilik, penyalutan sol-gel organosilana telah dilakukan terhadap membran seramik gentian berongga berasaskan kaolin. Morfologi dan kekasaran permukaan membran telah dianalisis menggunakan mikroskopi elektron imbasan pancaran medan (FESEM) dan mikroskopi daya atom. Kefungsian permukaan membran dikaji menggunakan analisis daripada inframerah jelmaan Fourier, spektroskopi pelepasan cahaya sinar-X (XPS), dan belauan sinar-X. Prestasi penapisan membran telah dinilai menggunakan modul aliran silang. Pada peringkat pertama, kajiankebolehlaksanaan telah dilaksanakan terhadap kaolin Malaysia (MK) dan kaolin Nigeria (NK) bagi penghasilan membran gentian berongga berasaskan kaolin, dengan mempelbagaikan komposisi bahan (34 sehingga 37 wt.%) dan suhu pensinteran (1200 sehingga 1500˚C). Hasil kajian telah menunjukkan bahawa peningkatan kepekatan kaolin dan suhu pensinteran mengurangkan kadar fluks. Hasil analisis terhadap fiziokimia dan prestasi menunjukkan bahawa membran seramik MK dengan 34 wt.% menghasilkan fluks air yang lebih baik (565.06 L/m^{2}h) pada saiz liang dan kestabilan sasaran berbanding membran NK dengan 34 wt.%. Keputusan ini berpunca daripada MK yana mempunyai darjah kehabluran yang lebih tinggi dan saiz zarah yang lebih kecil. Pada peringkat kedua, bagi mencapai keberkesanan penyerapan minyak-penapisan, agen organosilana seperti metiltritoksisilana (MTES), fluoroalkilsilana, oktadesiltrimetoksisilana, klorotrimetilsilana, dan trikloro(oktadesil)silana telah digunakan untuk pengubahsuaian membran gentian berongga kaolin superhidrofobik-superoleofilik. Hasil analisis daripada XPS dan FESEM jelas menunjukkan bahawa organosilana telah melekap dengan kuat pada permukaan membran kaolin. Kesan kitaran penyalutan dan kepekatan minyak turut dikaji. Dalam kalangan membran bersalut, membran kaolin bersalut MTES menunjukkan sudut sentuh air maksimum yang bernilai 161.3° dan sudut sentuh minyak paling rendah, iaitu 0°. Ia menunjukkan bahawa sifat superhidrofobik-superoleofilik berjaya diperoleh. Pada peringkat ketiga kajian, prestasi perolehan minyak bagi membran kaolin dengan agen organosilana yang berbeza turut dinilai dan dibanding. Membran bersalut MTES menunjukkan keupayaan serapan minyak yang maksimum iaitu 10 g/g, fluks minyak sebanyak 80 L/m^{2}h dan kecekapan pemisahan minyak pada tahap 90%. Membran bersalut MTES teroptimum telah digunakan untuk pengoptimuman keadaan proses yang seterusnya (kepekatan minyak, aliran suapan dan pH suapan) dalam modul aliran silang untuk meningkatkan fluks minyak dan kecekapan pemisahan menggunakan kaedah sambutan permukaan (RSM). Berdasarkan reka bentuk komposit pusat, fluks maksimum minyak dan kecekapan maksimum pemisahan masing-masing bernilai 97.67 L/m^{2}h dan 98.41% telah dicapan pada kepekatan minyak bernilai 50 mg/L, aliran suapan bernilai 300 mL/min, dan pH suapan bernilai 4. Model RSM yang diperoleh adalah setanding dengan data uji kaji. Secara keseluruhan, kajian ini menunjukkan pembangunan membrane gentian berlubang kaolin superhidrofobik-superoleofilik yang berdaya maju secara ekonomik untuk proses gabungan penyerapan dan penapisan bagi pemisahan minyak daripada air keluaran. Kajian ini mampu membantu para penyelidik dalam menyingkir bahan cemar menggunakan proses hibrid penyerapan-penapisan.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
ANOVA	-	Analysis of variance
CCD	-	Central composite design
CTMS	-	Chlorotrimethylsilane
DOE	-	Design of experiments
DTA	-	Differential thermal analysis
EDS	-	Electron dispersive spectroscopy
FAS	-	Flouroalkylsilane
FESEM	-	Field emission scanning electron microscopy
FTIR	-	Fourier transform infrared spectroscopy
MK	-	Malaysian kaolin
MKM	-	Malaysian kaolin membrane
MTES	-	Methyltriethoxysilane
NK	-	Nigerian kaolin
NKM	-	Nigerian kaolin membrane
OCA	-	Oil contact angle
OTMS	-	Octadecyltrimethoxysilane
RSM	-	Response surface methodology
TCOS	-	Trichloro(octadecyl)silane
TEOS	-	Tetraethylorthoxysilicate
TGA	-	Thermogravimetry analysis
WCA	-	Water contact angle
XPS	-	X-ray photoelectron spectroscopy
XRD	-	X-ray diffraction
XRF	-	X-ray fluorescence
OPW	-	Oilfield produced water

LIST OF SYMBOLS

Δt - Operation time
A - Area of membrane
f - Force
v - Velocity
p - Pressure
g - Gram
Jo - Oil flux
Q - Oil absorption capacity
m_f - Weight of membrane after coating
mL - Millilitre
V - Volume
$V_{d,final}$ - Final volume of water
$V_{d,initial}$ - Initial volume of water
σ_F - Bending strength
wt % - Weight percentage
mg/L - Milgram per liter
P_a - Pressure
t - Time
L - Permeate flux
m_i - Weight of membrane before coating
$Error\ (\%)$ - Percentage error
Y - Response
% - Percentage
µm - Micrometre
^oC - Degree Celsius

CHAPTER 1

INTRODUCTION

1.1 Background of Research

A large volume of oilfield produced water (OPW) is generated as refinery by-product during crude oil production, enhanced oil recovery and exploitation processes which are carried out in both offshore and onshore. The constituents are carcinogenic, toxic and persistent due to the presence of oils, hydrocarbons, dissolved formation minerals, suspended solids, dissolved gases, and injected chemical such as biocides, corrosion inhibitors, emulsion and reverse emulsion breakers [1–3]. Discharge of OPW to the environment can contaminates the quality of drinking water, groundwater, degrade soil, and deplete oxygen [4–6]. Furthermore, the discharge also unavoidably resulted to a great loss of valuable energy resources due to the presence of vast amount of unexplored crude oil in OPW.

Therefore, recovery of oil from OPW is a prerequisite to cost saving method to oil and gas industry as well as the protection of environment. Conventional methods of oil removal are gravity separation, coagulation and air flotation, electrocoagulation and electrostatic separation, microwave and heat treatment methods, oil absorbing, biodegradation, sonication, cyclones filters, sand filter, oxidation, photocatalytic treatment, ozone treatment, electrochemical process, Fenton process, flocculation, adsorption and ion exchange [7–14]. However, the aforementioned techniques have drawback such as less energy efficient, low product quality and creation of huge amount of sludge, high energy cost, requirement of large space for installation, complex separation equipment, use of toxic compounds, and difficult to clean up or recycle. To overcome this limitation, membrane technology has gained interests in treatment of water as well as recovery of oil in oil and gas industries.

Membrane is a selective barrier to separate the solute molecule based on size and charge based interaction. Globally, membrane technology has been implemented in various industrial wastewater treatment and public sewage as well as municipal waste management. The main advantage of membrane technology is low cost, high emulsion separation efficiency, no phase change, no addition of chemicals and simplicity of operation [15–17]. Polymer and ceramic are the common membrane material for industrial scale applications. Polymeric membranes are widely adopted in wide range of versatile applications such as desalination, wastewater treatment and bio-product purification [18, 19]. Ceramic membranes are not well explored but it has the unique characteristics of chemical and thermal stability, excellent resistance to fouling, pressure resistance, long lifetime, ease of cleaning and mechanical stability. Nonetheless, its shortcoming is in its limitation for large scale operation due to high cost. In this regards, recently low-cost raw materials such as kaolin [15–17], natural clay [20–22], waste material such as rice husk ash [23] and fly ash [24–26] corn cob ash [27] and palm oil fuel ash (POFA) [28, 29] are attempted. Among all the ceramic materials used for fabrication of ceramic membrane, kaolin is one of the most used and applied due to its distinct features like abundant availability, cost-effectiveness, low plasticity, ease of processing and high refractory properties to the membrane [30, 31].

Superhydrophobic-superoleophilic materials are preferred in the absorption and recovery of the low surface tension oil molecules. However, ceramic membranes are rich in hydrophilic groups thus tend to absorb water molecules. For the oil molecules absorption/recovery, hydrophobic-oleophilic modification is necessary to the ceramic membranes. Silica sol coating is a single step sol-gel technique which provides a cost effective and simple approach to improve the hydrophobic property of membrane through the decrease of surface free energy. There are several reports on various chemicals used in lowering the surface energy of a substrate which include; organophosphonic acids, steric acids, fatty acids, alkanethiols and organosilanes [32–37]. Organosilanes are the most widely and frequently used chemicals in both research and technology for alignment of proper surface topology and lowering of substrate surface energy through the hydrolytic condensation of tetraethylorthoxysilicate (TEOS) and organosilanes. It is preferred due to its simplicity, creation of superhydrophobic-superoleophilic substrate, cost-effective and less toxic [38].

Most of the organosilane agents used in superhydrophobic-superoleophilic modifications of substrates are methyltriethoxysilane (MTES), fluoroalkylsilane (FAS), octadecyltrimethoxysilane (OTMS), chlorotrimethylsilane (CTMS), and Trichloro(octadecyl)silane (TCOS). And also, these organosilanes are mostly used in surface coating of fabric, filter paper, porous glass, polymeric membranes, nanofibre mats, sponge substrates. Wen *et al.* [39] produced a superhydrophobic surface with water contact angle of 156^o on a glass substrate via a sol-gel derived organic-inorganic hybrid emulsion. The hybrid sol-gel was prepared by co-hydrolysis and co-polycondensation reactions of TEOS, MTES and tri(isopropoxy)vinylsilane (TIPVS). Wang *et al.* [40] prepared a silica sol by co-hydrolysis and co-condensation of TEOS and FAS for superhydrophobic fabric, nanofibre mat, filter paper, glass slide and silicon wafer coatings. The coated substrates were reported to have a high degree of water contact angle above 170^o. Another group of researchers [41] fabricated a robust superhydrophobic fabric bag for oil absorption and collection of oily water. The fabric bags were immersed into TEOS and OTMS, and ammonia solution, respectively. While SiO_2 nanoparticles functionalized with OTMS on the fabric surfaces. The substrate was reported to possess water contact angle above 150^o. Meng and co-workers [42] also fabricated a hydrophobic nano-structure porous glass membrane by deposition of SiO_2 nanoparticles followed by grafting with CTMS on the membrane surface. Zhang and Seeger [43] used chemical vapour deposition of trichloro(octadecyl)silane (TCOS) to produce superhydrophobic-superoleophilic polyester textile material for selective oil absorption application. The resultant material showed a high-water contact angle above 150^o demonstrating the superhydrophobic features.

For oil-water separation, ultrafiltration (UF) and microfiltration (MF) are widely deployed for the treatment of oil-water mixture. However, clogging of oil on the membrane pore and surface is a critical issue in filtration of oily wastewater feed, which leads to decline of the flux rate and membrane lifecycle. Hence, membrane modification gained more interest in tailoring of ceramic membranes for versatile applications. Adsorptive membranes are showing promising effect on the removal of pollutant such as metal ions, hydrocarbons and dyes from the contaminated sources.

In ceramic membrane, there are three types of modification process, which are chemical vapour deposition (CVD), immersion, and sol gel method. Immersion method is the simplest method but do not possessed any oil absorption capabilities. Meanwhile, CVD is dangerous method due to the thermal process involved. From literatures, it was reported that modification via sol gel method is always applied for the application of oily wastewater separation. As stated by Pierre (2013), there are many definitions of sol gel process exist. For instance, sol gel process takes into account multicomponent oxides that are homogeneous at the atomic level. In fact, the term “sol gel” is restricted to the gels synthesized from alkoxides in which from colloidal dispersion or from metal alkoxides. In other word, grafting process through sol gel method can be defined as a colloidal route used to synthesize ceramics with an intermediate stage including a sol and/or gel state. Literatures on the absorption of oil using ceramic membranes are less studied. Therefore, this study aimed to recover the oil from produced water using silane functionalized ceramic membranes.

The Jabatan Mineral dan Geosains Malaysia (JMG)) minerals reported that, Peninsular Malaysia produces up to 112 million tons of raw kaolin with three kaolin processing industries all located in Perak state [44]. However, most of the kaolin produced by these three industries are mainly used in production of paper as such less emphasis is given other research related application such as membrane fabrication. On the other hand, the raw material research and development council of Nigeria reported that, Nigeria have about 3 billion metric tons of kaolin deposit [44, 45]. The kaolin clay is mainly used in producing household, office utensils and for pharmaceutical used in the country. Also, they are used in fabricating ceramic utensils, paper, porcelain, white incandescent light bulbs, and paint, among others. The kaolin mineral from Nigeria are used for other purposes but its application for the fabrication of membrane is elusive in literature. This research both used Nigerian kaolin and Malaysian in fabrication of ceramic membrane for recovery of oil from produced water.

1.2 Problem Statement

Membrane fouling is a critical issue in filtration of oil-water emulsion and it causes the decline of the membrane performance and life span. To overcome this limitation, the advent of hybrid absorption combined membrane filtration technology has lured in specific removal of target pollutants. It is based on the mechanism of absorption of feed components with respect to surface functionality of membranes. Thereby, it prevents the blocking of solutes on membrane surface which endured for longer duration filtration. Surface modification are prevalently used to tailor the membranes for better oil-water separation. Literatures on various pollutants such as metal ions, ammonia, arsenic etc. are reported using ceramic membranes [46–48]. However, ceramic membranes are quite expensive and low-cost ceramic materials such as clay and biomass based green silica materials are paid more attention in development of membranes. Hence, this study aimed to develop low cost kaolin based hollow fibre ceramic membrane for the recovery of oil from produced water. Malaysian kaolin based ceramic membranes are used in versatile application for liquid [49] and gas separation [50, 51], owes to chemical and thermal stability. Similarly, Nigerian kaolin has a rich of silica groups required for ceramic membrane and used extensive as additives in cement industries and pharmaceutical applications [52–54]. Hence, the feasibility study of each kaolin was performed to aid comparative analysis for the development of low cost ceramic membranes. The main advantage of utilization of kaolin is low cost, abundant in nature and rich of hydroxyl silica groups. Yet, the conventional ceramic ultrafiltration membrane retains the oil molecules on surface and cause adsorption of oil. Thus, cost effective hybrid kaolin-based absorption membrane was proposed for the recovery of oil from produced water.

Surface hydrophobic modification are prevalently used to tailor the hybrid absorption-based membranes for better oil-water separation. Silane functionalization is the common method to enhance the super hydrophobic (anti-wetting properties) and superoleophilic (wetting properties) properties in ceramic membrane. Meng and co-workers [42] fabricated a hydrophobic nano-structure porous glass membrane by deposition of SiO_2 nanoparticles followed by grafting with chlorotrimethylsilane (CTMS) on the membrane surface for oil absorption. Similarly, Hubadillah et al [55] used methyltriethoxysilane (MTES) to develop superhydrophobic-superoleophilic ceramic membrane for efficient oil separation. Another group of researchers used 1H,1H,2H,2H-perfluorooctyltriethoxysilane (FAS) to fabricate superhydrophobic ceramic membrane for carbon dioxide capture [50]. Based on the literature, different organosilane agents have significant influence in control of membrane hydrophobic properties. However, mechanistic study of different silane agents on superhydrophobic-superoleophilic ceramic membrane modification is limited for the oil-absorption. Hence, methyltriethoxysilane (MTES), fluoroalkylsilane (FAS), octadecyltrimethoxysilane (OTMS), chlorotrimethylsilane (CTMS) and trichloro(octadecyl)silane (TCOS) were chosen as organosilane agents for the modification of kaolin ceramic membrane to absorb the oil molecules such as hexane, kerosene and crude oil. Moreover, membrane filtration involves hydrodynamic conditions such as oil concentration, cross flow velocity and feed pH. Therefore, it is necessary to optimize the condition for enhanced filtration for longer durations. Response surface methodology is widely recognized tool to optimize the parameter and evaluation of interaction of parameters with minimal experiments. RSM was also used in this study to design the experiments and evaluation of parameters on oil absorption.

To the best of our knowledge, literatures on absorption and filtration of oil using ceramic membranes are limited, owes to ceramic membranes are expensive. Based on the literature, the study is organized into three section and it includes (i) Fabrication of kaolin hollow fibre ceramic membrane via phase inversion/sintering method using Malaysian and Nigerian Kaolin, (ii) Comparison of different organosilane agents (MTES, FAS, OTMS, CTMS and TCOS) on super hydrophobic-oleophilic modification of kaolin ceramic membrane, (iii) Performance evaluation and optimization of process variables on optimized super hydrophobic-oleophilic kaolin hollow fibre ceramic membrane in cross flow filtration set up.

1.3 Research Objectives

i. To investigate the feasibility of Malaysian and Nigerian kaolin as the main ceramic materials for the fabrication of kaolin-based hollow fibre ceramic membrane at different ceramic loadings and sintering temperatures.

ii. To examine the effects of different organosilane agents on the superhydrophobic-superoleophilic modification of kaolin-based hollow fibre membrane regarding surface morphology, phase transformation, wettability, surface topology, pore size and porosity distribution.

iii. To evaluate the filtration performance of the superhydrophobic-superoleophilic kaolin-based hollow fibre membranes and the optimization process variables using response surface methodology (RSM) for the recovery of oil from synthetic OPW.

1.4 Scope of Study

The scope of this research is devised as follows;

i. Screening and characterization of Nigerian and Malaysian kaolin precursor material for the fabrication of the kaolin-based hollow fibre ceramic membrane. Physiochemical properties of each kaolin were analyzed using particle size analyzer (PSA), thermogravimetry/differential thermal analysis (TG/DTA) and X-ray fluorescence (XRF), X-ray diffraction (XRD) and Brunauer-Emmett-Teller (BET) (to accomplish objective 1)

ii. Fabrication of Nigerian and Malaysian kaolin based hollow fibre ceramic membrane based using kaolin loading composition of 34 to 37 wt.% and sintering temperature of 1200 to 1500 ^{o}C. Comparison of physio-chemical characterization and pure water flux performance of the Nigerian and Malaysian kaolin based hollow fibre ceramic membrane for water filtration studies. Physio-chemical characterization involves field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), atomic force microscopy (AFM), mercury intrusion porosimetry (MIP), contact angle measurements (CA (to accomplish objective 1)

iii. Superhydrophobic-superoleophilic modification of optimum kaolin based hollow fibre ceramic membrane using different organosilane agents such as methyltriethoxysilane (MTES), fluoroalkylsilane (FAS), octadecyltrimethoxysilane (OTMS), chlorotrimethylsilane chlorotrimethylsilane (CTMS) and trichloro(octadecyl)silane (TCOS) through surface coating technique. Also, evaluation of 1 to 4 coating cycles of the organosilane agents on the kaolin-based hollow fibre ceramic membrane for the superhydrophobic-superoleophilic modification (to accomplish objective 2)

iv. Confirmation of individual silane agent modification of kaolin-based hollow fibre ceramic membrane using (FESEM), X-ray diffraction (XRD), energy dispersion spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), Fourier transform infrared spectroscopy (FTIR), mercury intrusion porosimetry (MIP), contact angle measurements (CA) (to accomplish objective 2)

v. Optimum oil recovery with respect to different silane agent modified kaolin-based hollow fibre ceramic membrane for oil absorption capacity and filtration efficiency using synthetic produced water of oil concentration of 1000 mg/L, 1500 mg/L and 2000 mg/L (to accomplish objective 3)

vi. Studying the optimum parameters and conditions of the superhydrophobic-superoleophilic kaolin-based hollow fibre ceramic membrane towards optimization of the oil recovery from synthetic oilfield produced water by set of independent parameters (oil concentration of 50-10000 mg/L, feed pH 4-10 and feed flux of 150-300) with responses of oil flux and oil separation efficiency) (to accomplish objective 3)

1.5 Significant of the Study

Water crisis is a serious issue across the world in the present era. The produced water constitutes of various organic and inorganic substances, which are toxic to environment. The permissible limit of discharge of produced water is 10-15 ppm [56]. Membrane technology has acquired greater interest in production sustainable of quality potable water from industrial wastewater and to meet the strict environment regulation policy. This study contributes to development of cost effective functionalized superhydrophobic-superoleophilic kaolin hollow fibre ceramic membrane for the recovering oil from produced water. It also aids in novel scheme of hybrid absorption combined membrane filtration methodology for the recovery of oil from oily wastewater effluents. Apart, organosilanes functionalization effects also extend the frontier of knowledge on novel approach for modification of kaolin membrane for the effective filtration of low surface tension solutions. The developed prototype may also serve as a low-cost filtration module for the industrial wastewater treatment.

1.6 Thesis Organization

This section describes the organization of the different chapters of this thesis for the fabrication of superhydrophobic-superoleophilic kaolin-based hollow fibre ceramic membrane for the recovery of oil from oilfield produce water. The flow of the entire thesis is depicted in Figure 1.1 bellow.

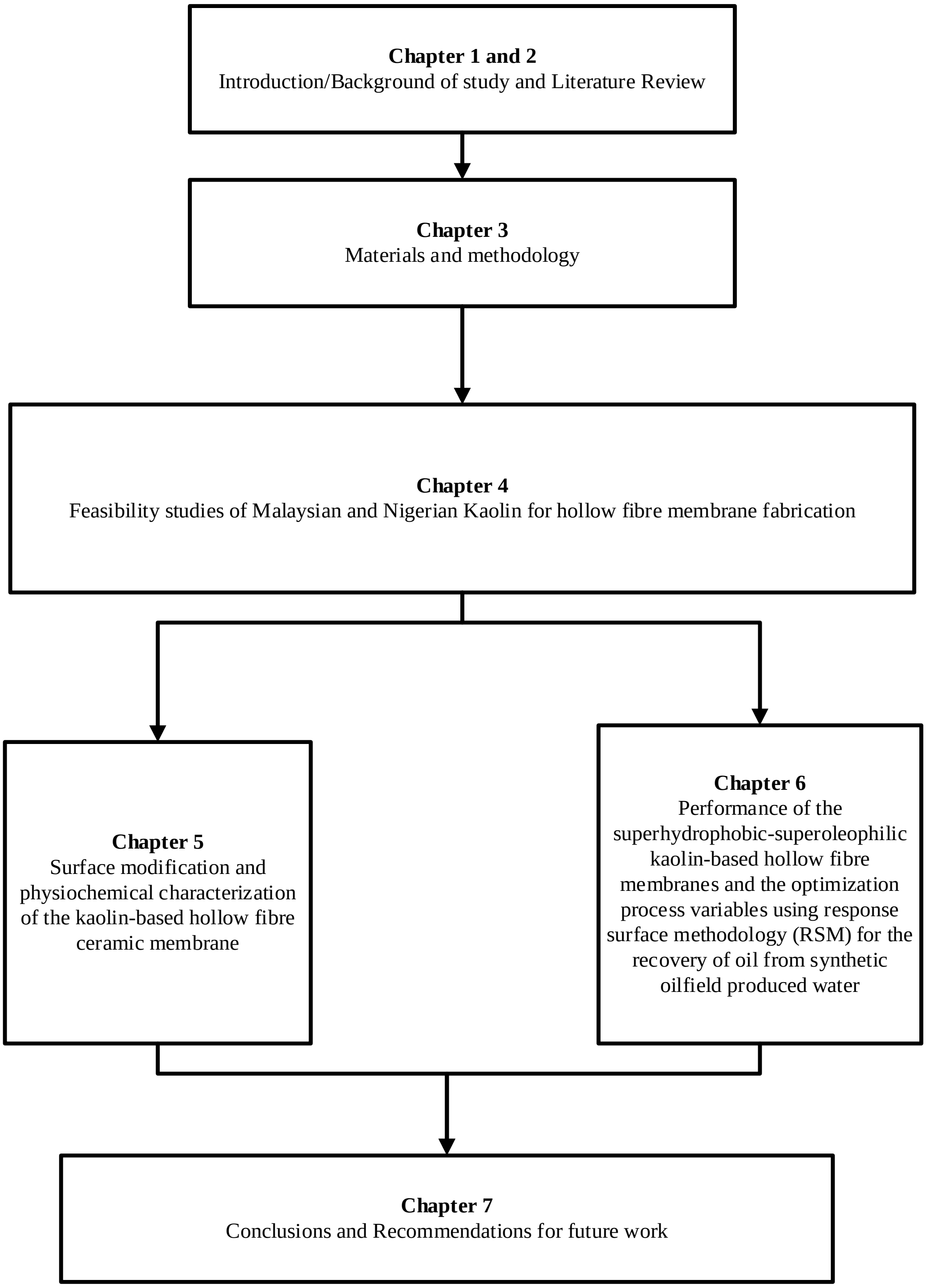

Figure 1.1 Summary of overall thesis structure

Chapter 1 briefly introduce the oilfield produce water contamination in the ecosystem and the current technologies used for the treatment which mainly devoted to this study. It followed by detail problem statement, objectives, scopes and the significance of the study.

Chapter 2 describes the scientific literature review on the produce water, the composition, available methods for the treatment were comprehensively discussed, with much focus on the membrane technology separation techniques for oily wastewater treatments. The adverse effects and the toxicity of oilfield produced water to the ecosystem were also discussed. The chapter also discussed the current available methods of surface modification on different substrate materials as well as their advantages and limitations.

Chapter 3 of the thesis describes the techniques, materials, working principles, modification process, characterization approaches used, membrane setup for oil recovery from produced water and complete operation framework.

Chapter 4 discusses the characteristics features of two different kaolin powders as an alternative material to fabricate kaolin hollow fibre ceramic membrane for the modification and subsequent oil recovery purpose. The characterizations include evaluation of chemical composition of the both kaolin, particle size distribution, crystallinity, morphology and surface area analysis. Afterwards the fabrication and characterization of defect-free high-performance kaolin-based hollow fibre ceramic membrane from the selected kaolin material using various loading composition and sintering temperature. The effect of loading composition, sintering temperature and also the thermal stability, phase transformation as well as pure water flux were evaluated. This chapter also describe the preliminary ceramic membrane fabrication and pure water flux evaluation of the fabricated membrane.

Chapter 5 discusses the processes of surface modification of the kaolin-based hollow fibre ceramic membrane. The effects of various surface coating agents on the membrane surface with respect to its concentration, coating cycle and coating time on the wettability, surface porosity and surface chemical composition.

Chapter 6 presents the potential of the oil recovery by the fabricated superhydrophobic-superoleophilic kaolin-based hollow fibre ceramic membrane was evaluated using the cross-flow filtration system. Also, the factors that influenced the separation efficiency were studied in detail. And it also discussed the optimization study of the superhydrophobic-superoleophilic kaolin-based hollow fibre ceramic membrane performance for 3 significant factors viz oil concentration, feed pH and feed flow rate via response surface research methodology approach. The desirability test was also performed to verify the adequacy of the developed model.

Chapter 7 finally presents the conclusions from present study, suggestions and recommendations for future researcher.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Jamilu Usman**; Mohd Hafiz Dzarfan Othman*; Ahmad Fauzi Ismail; Muklish A Rahman; Juhana Jaafar; Yusuf Olabode Raji; Tijjani Hassan El-badawy; Gbadamosi Afeez, Impact of Organosilanes Modified Superhydrophobic-Superoleophilic Kaolin Ceramic Membrane for Efficient Oil Recovery from Produced Water J*ournal of Chemical Technology & Biotechnology*, 2020, Wiley (**Q1, IF: 2.75**)

Indexed Journal

1. **Jamilu Usman**; Mohd Hafiz Dzarfan Othman*; Ahmad Fauzi Ismail; Muklish A Rahman; Juhana Jaafar; Tijjani Abudllahi, Comparative study of Malaysian and Nigerian Kaoli-based ceramic hollow fibre membranes for filtration application, *Malaysian Journal of Fundamental and Applied Sciences*, 16:2(2020) 182-185 **(Indexed by ISI)**

2. **Jamilu Usman;** Mohd Hafiz Dzarfan Othman*; Ahmad Fauzi Ismail; Yusuf Olabode Raji; Tijjani Hassan El-badawy; Tijjani Abdullahi; Jamila Baba Ali, Facile Approach in Development of Superhydrophobic-Superoleophilic Kaolin-Based Hollow Fibre Ceramic Membrane, *International Journal of Advance Science and Technology***,** 29:108(2020), 3451-3459 **(Indexed by Scopus)**

Non-Indexed Conference Proceedings

1. **Jamilu Usman**; Mohd Hafiz Dzarfan Othman*; Ahmad Fauzi Ismail; Muklish A Rahman; Juhana Jaafar; Tijjani Abudllahi, Influence of kaolin on fabrication of ceramic hollow fibre membrane for oily wastewater treatment, National Congress on Membrane Technology, 2017, Pulai Springs Resort, Skudai, Johor, Malaysia

2. **Jamilu Usman**; Mohd Hafiz Dzarfan Othman*; Ahmad Fauz Ismail; Mukhlis A Rahman; Juhana Jaafar; Khairul Anwar Mohamad Said; Yusuf Olabode Raji, Superhydrophobic-Superoleophilic Modification of Kaolin-Based Ceramic Membrane Via Silanization Technique, International Conference on Sustainable Environment and Technology, 2019, Double Tree Hilton Hotel, Johor, Malaysia

3. **Jamilu Usman**; Mohd Hafiz Dzarfan Othman*; Ahmad Fauzi Ismail; Mukhlis A. Rahman; Juhana Jafar; Yusuf Olabode Raji; Tijjani Hassan El Badawy, Superhydrophobic-Superoleophilic Modification of Ceramic Membrane Via Nano-Silica Based Silane Sol, Malaysian-Japan International Conference on Nanoscience, Nanotechnology and nanoengineering 2020, Universiti Teknologi Mara (UiTM), Shah Alam, Malaysia