BAUXITE-BASED HOLLOW FIBER MEMBRANE FOR OILFIELD PRODUCED WATER TREATMENT

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This thesis is dedicated to my late father Mr. Mohamad Esham Bin Awang, my beloved mother, Mrs. Fauziah Binti Shamsudin, my lovely wife, Mrs Intan Norshafika Binti Annuar, my cute child, Nur Eryna Inara Binti Mohamad Izrin and my siblings and friends, who taught me that the best kind of knowledge to have is that which is learned for its own sake and also for their support and love through this completion of the thesis.

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ABSTRACT

An advanced treatment technology of oilfield produced water (PW) is required because of the incapability of physical separation technologies to produce high quality treated water. Recently, hollow fiber membranes (HFMs) have been widely utilized since it is recognized as one of the most effective PW treatment technologies. However, membrane fouling is the main drawback of commercial polymeric membranes while commercial ceramic membranes are extremely expensive. Therefore, the aim of this research is to develop HFM from natural low cost ceramic material which is raw bauxite to counter these problems. The bauxite hollow fiber membranes (BHFMs) were developed using Malaysian raw bauxite with different bauxite loadings (45-60 wt.%) and sintering temperature (1250-1450 °C) via phase inversion and sintering technique. From the results obtained, BHFM with 50 wt.% of bauxite loading and sintered at 1350 °C showed the best morphological structure with sufficient mechanical strength of 135 MPa, suitable porosity value of 15.54 % and the medium average pore size of 0.78 µm. The BHFM also showed excellent water permeation of 195 L/m^2.h.bar and 99% of oil rejection. The results of the contact angle revealed that the membranes are super-hydrophilic and super-oleophobic membrane. The fouled BHFM was further evaluated for its self-cleaning ability via ultraviolet (UV) driven photocatalytic test. The results showed the water permeation of the UV radiated BHFM increased by 30% compared to non-radiated BHFM. Then, the fouled BHFM was tested again for another two runs with the long term photocatalytic test to observe the efficiency of the self-cleaning properties. The result showed that the long-term study does affect the water permeability of the BHFM, which decreased to 169 L/m^2.h.bar in the 3rd run of the self-cleaning test. The results of the oil rejection do not affect by the long-term photocatalytic test which stay in the range of 99%. The BHFM need some modification in the pore size of the membrane to maintain the self-cleaning properties. In conclusion, this study would provide a new insight on the application of BHFM as the cost-effective, self-cleaning membrane for the PW treatment.

ABSTRAK

Teknologi rawatan termaju untuk air sisa campuran minyak (PW) diperlukan kerana teknologi pemisah fizikal tidak mampu menghasilkan air terawat berkualiti tinggi. Baru-baru ini, membran gentian berongga (HFMs) telah banyak digunakan sejak ia dikenali sebagai salah satu teknologi rawatan PW yang paling berkesan. Bagaimanapun, kotoran membran adalah kelemahan utama bagi membran polimer komersial manakala membran seramik komersial adalah sangat mahal. Oleh itu, tujuan penyelidikan ini adalah untuk membangunkan HFM dari bahan seramik kos rendah dan semula jadi iaitu bauksit mentah bagi mengatasi masalah tersebut. Membran gentian berongga bauksit (BHFMs) dibangunkan dari bauksit mentah Malaysia dengan muatan bauksit berbeza (45 % berat -60 % berat) dan suhu pensinteran berbeza (1250 °C -1450 °C) melalui teknik penyongsangan fasa dan pensinteran. Dari hasil ujian yang diperoleh, BHFM dengan 50 % berat muatan bauksit dan sinteran pada 1350 °C menunjukkan struktur morfologi terbaik dengan kekuatan mekanikal yang mencukupi iaitu 135 MPa, nilai keliangan yang sesuai iaitu 15.54% serta dengan purata saiz liang yang optimum iaitu 0.78 µm. BHFM juga menunjukkan kebolehtelapan air yang baik iaitu 195 L/m^2.h.bar dan 99% penolakan minyak. Hasil kajian dari sudut sentuhan menunjukkan bahawa membran adalah membran super-hidrofilik dan super-oleofobik. BHFM tercemar seterusnya dinilai untuk keupayaan pembersihan-diri melalui ujian fotobermangkin menggunakan ultra-ungu (UV). Hasilnya menunjukkan kebolehtelapan air BHFM diradiasi UV meningkat sebanyak 30% berbanding dengan BHFM tanpa diradiasi dengan UV. Seterusnya, BHFM yang tercemar diuji untuk dua kali ujian fotobermangkin dengan jangka masa yang lebih panjang untuk melihat kecekapan sifat pembersihan-diri. Hasil ujian menunjukkan bahawa kajian pembersihan-diri fotobermangkin dengan masa yang lebih panjang ini mempengaruhi kebolehtelapan air BHFM, yang menurun kepada 169 L/m^2.h.bar pada ujian kali ke-3 pembersihan-diri. Keputusan penolakan minyak pula tidak dipengaruhi oleh ujian fotobermangkin pada jangka panjang kerana masih berada di sekitar 99%. BHFM memerlukan beberapa pengubahsuaian dalam saiz liang membran untuk mengekalkan sifat pembersihan-diri. Kesimpulannya, hasil kajian ini akan memberikan satu wawasan yang baharu mengenai penggunaan BHFM sebagai membran berkos efektif dan keupayaan pembersihan-diri untuk rawatan PW.

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LIST OF ABBREVIATIONS

PW	-	Produced Water
UF	-	Ultrafiltration
RO	-	Reverse Osmosis
NF	-	Nanofiltration
MF	-	Microfiltration
HFM	-	Hollow Fiber Membrane
BHFM	-	Bauxite Hollow Fiber Membrane
UV	-	Ultraviolet
EOR	-	Enhanced Oil Recovery
API	-	American Petroleum Institute
BTEX	-	Benzene, Toluene, Ethyl Benzene, Xylene
PAH	-	Polycyclic Aromatic Hydrocarbon
TOC	-	Total Organic Carbon
MW	-	Molecular Weight
TDS	-	Total Dissolved Solid
XRD	-	X-ray Diffraction
XRF	-	X-ray Fluorescence
NOM	-	Natural Organic Matter
SEM	-	Scanning Electron Microscopy

LIST OF SYMBOLS

ºC	degree celcius
%	percentage
mm	millimetre
µm	micrometre
g	gram
wt.%	weight percentage
N	Newton
D_o	outer diameter
D_i	inner diameter
min	minute
rpm	rotation per minute
mol	Molar
cm	centimetre
P	pressure
MPa	Mega Pascal
psi	pound per square inch
mL	millilitre
L	litre
$L/h.m^2.bar$	water permeability
RM/m^2	Ringgit per meter square
kWh/bbl	kiloWatt hour per barrel

CHAPTER 1

INTRODUCTION

1.1 Research Background

Huge amount of produced water (PW) generated throughout the oil and gas extraction constituted the majority of waste effluent in the petroleum industry (Hussain *et al.*, 2014). PW is a natural water layer which located under the layer of hydrocarbon in the reservoir which composed of dispersed oil, aromatic hydrocarbon, a broad variation of salts, fine silts of both clay and silicon composition, and also contain active biological materials. Hussain *et al.* (2014) stated that, in 1995, The American Petroleum Institute (API) estimated 18 billion barrels of PW was generated in the US region which is only from onshore platform. They also stated that values keep increasing in 2007 and 2011 which is in between 70 to 100 billion barrels of PW, with only a small part treated for the beneficial reuse. For the purpose of disposal and other beneficial reuse, PW needed to be treated first using water treatment technologies until to appropriate extent with the required quality which in accordance to permissible discharge level. Conventionally, PW treatment were limited to remove large suspended particles and free oil before discharge in the sea or injection into the disposal wells (Dores *et al.*, 2012).

Current technologies such as gravity separation, hydrocyclone, centrifuging, gas floatation and filtration cannot stand alone without using the expensive chemicals to achieve the desired cleanliness standard. New and unconventional technologies should be established to discard dissolved oil and other smaller component without the use of chemicals. Igunnu & Chen (2012) stated that the general permissible discharge level for the disposal of PW into the sea is 40 ppm Oil-in-Water (OIW). However, they also stated that many countries had changed the standards into more stringent value. As example, Paris agreed the maximum discharge value reduced to 30 ppm OIW, US region also set the daily maximum discharge value at 42 ppm of oil. Lower value also was set for Australia and China which are 30 ppm and 10 ppm of oil and grease content. In 2000, some of the region was aimed to ‘zero discharge’ in order to protect the aquatic environment. Since that, most oil and gas operators are now searching for a new and better technology to implement the zero environmental harmful discharge which apparently unable to achieve by the current technologies.

Hence, oil and gas operators have looked forward toward membrane filtration technology due to the potential to counter the disposal issues associated with the conventional purification technologies. Membranes technology have become one of the most effective methods in some separation and purification applications especially for onshore and offshore oilfield PW treatment (He & Jiang, 2008). These membranes play on important roles because of their ability of breaking off the emulsion without using high cost chemical, high oil removal efficiency, high removal of chemical oxygen demand (COD) and total organic carbon (TOC) and their facilities being more compact rather than the current technologies. Membrane filtration processes, such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO), have some potential in removing the smallest particle which is less than 10 μm and most stable oil droplets, suspensions and dissolved components. These membranes are effective for obtaining sufficiently high quality of water for other beneficial use and disposal.

However, previous field and laboratory tests showed some of the membranes have faced problem regarding to the membrane fouling (Li & Lee, 2009). Even though most membrane can be cleaned, however this process requires extra chemical, energy and downtime of the treatment instalment, resulting in increasing operating cost of the treatment. Many researches have tried to overcome the fouling problem, which involving of pre-treatment of feed solution, adjustment of membrane properties, operation conditions improvement and module arrangement optimization. Most of the mentioned method had successfully reduced the fouling problems to acceptable level, but they still need the cleaning method to be employed as always. The cleaning method can be categorized into four types, which are mechanical cleaning, hydraulic cleaning, electrical cleaning and chemical cleaning (Abadi *et al.*, 2011). Among of these cleaning methods, the most extensively used is chemical cleaning. There are many types of chemicals have been used for the chemical cleaning of the membrane such as NaOCl, Na-OH based formula and surfactants (Madaeni & Mansourpanah, 2004). In membrane treatment plant, membrane filtration process must be shutdown regularly for membrane cleaning to maintain the membrane performance. Frequent cleaning affects the increment of the labour cost also complexity of the membrane filtration process. The worst part is, chemical cleaning of the membranes results on the problem of chemical waste disposal and yet affects the membrane durability.

Recently, the ceramic membranes have been used in field run test for the PW treatment as a full-scale facility (Igunnu & Chen, 2012). The results indicated that the treated effluent produced after the treatment was free from suspended solids and almost all of the non-dissolved organic carbon has been removed. Both ceramic MF and UF membrane can be conducted using cross-flow and dead-end filtration modes and they acquire a longer lifetime (more than 10 years) which can be considered longer compared to polymeric MF and UF membranes (Li & Lee, 2009). Ceramic membranes also offer more advantages compared to polymeric membranes in term of ability to resist such harsh physical and chemical surroundings like high contain of organic solvent, acid and base solution, oxidative and reductive condition also high thermal and pressure condition. Some ceramic membranes also resistance toward microorganisms and organic matter which can cause membrane fouling (Abadi *et al.*, 2011). Thus, this study is basically focused on the application of ceramic hollow fiber membrane for the PW treatment with the ability to counter the fouling problem.

1.2 Problem Statement

The ceramic membranes are commonly fabricated using micron-sized pure alumina powder due to their magnificent stabilities in term of structure, chemical and temperature. But, the pure alumina needs a high sintering temperature which is usually greater than 1500℃ to achieve desired porosity and mechanical strength (Mestre *et al.*, 2019). Due to high sintering temperature and pure alumina which is relatively expensive, a rising number of studies have been focused on the ceramic membranes preparation using inexpensive materials like natural ore and other inorganic industrial wastes such as cordierite (Zhang *et al.*, 2009), sepiolite (Zhou *et al.*, 2011), clays, industrial coal fly ash wastes (Dong *et al.*, 2008a) and bauxite.

However, the studies on the fabrication by utilizing raw bauxite minerals to replace the pure alumina are less reported (Li *et al.*, 2016). In general, bauxite is an aluminum ore that compose high quantity of alumina and low content of silicon dioxide. Bauxite also contain some impurities such as titanium dioxide and iron oxide. The amount of silicon dioxide in the bauxite not only helps in decreasing the desired sintering temperature but also they can act as a reactant to form chemically stabilize mullite through a secondary mullitization reaction with the inherent alumina. It also offered better porosity, lower average pore size, strong mechanical strength and excellence in chemical resistance. In addition, bauxite also hydrophilic characteristic which can counter the fouling problems.

Fouling has become main drawback in the practical of membrane technology in PW treatment. Membrane fouling not only affect the performance of the membranes, but also shorten the lifetime of the membranes. To solve the problems, a recently study merged the ceramic membrane with photocatalyst like TiO_2 to degrade the pollutants on membrane surface with the UV radiation (Mohtor *et al.*, 2018). Few researchers have published an anti-fouling properties of the membranes with TiO_2 by coating the composite membrane with TiO_2 particles and photodegradation under UV light. But, this coating method might have some drawbacks when applied on daily PW treatment which are the detachment of the coating particles from the membrane surface due to repetitive, which eventually can block pores of the membrane.

Bauxite possess great potential to overcome most of the problem related to the membrane technology for the PW treatment since the impurities like TiO_2 of the bauxite can incorporate with the aluminum source to form a better membrane including the anti-fouling properties (Bai *et al.*, 2015). Several studies have showed that membranes with TiO_2 were great in degrading the foulants, thus improving the membrane filtration performance. Damodar *et al.* (2009) have successfully fabricated modified PVDF membrane by adding different amount of TiO_2 particle into the casting solution, and the membranes showed their photocatalytic and anti-fouling properties under the UV light exposure.

In this research, the ceramic hollow fiber membrane was fabricated using Malaysia raw bauxite by phase inversion and sintering technique. The characterization of the prepared membrane was employed to find the optimum mechanical strength of the membrane, hydrophilicity, average pore size and the suitability of the membrane for the PW treatment. Then, the BHFM was further studied for the self-cleaning property. The BHFM was radiated with UV radiation before the oil rejection test and the result was compared with the system without UV exposure.

1.3 Research Objectives

The main objective of this study is to develop a bauxite hollow fiber membrane (BHFM) with self-cleaning property using Malaysian raw bauxite for the oilfield PW treatment. The specific objectives of the study are:

1. To establish the relationship of bauxite loading and sintering temperature on the structural, physical and chemical properties of the BHFM.
2. To evaluate the rejection of oil and permeate flux performance of BHFM in the PW treatment.
3. To assess the potential of BHFM as a self-cleaning membrane.

1.4 Scopes of Research

In order to achieve all of the objectives, the specified scopes have been organized for this research. The scopes are:

1. Preparing and characterizing raw bauxite obtained from Pahang, Malaysia as the alternative ceramic material:

    a. Drying the raw bauxite in oven before used. Grinding the dried raw bauxite into powder form. Lastly, sieving the bauxite powder to get the bauxite powder with particle size less than 36 μm through sieving process.

    b. Investigating the chemical composition and crystalline phase of the raw bauxite powder and crushed BHFM using x-ray fluorescence spectrometry (XRF) and x-ray diffraction (XRD).

2. Fabricating the BHFM by using the phase inversion and sintering technique:

    a. Preparing the ceramic suspension containing bauxite powder as the chosen ceramic material at different composition of suspension (45 wt.% - 60 wt.% of bauxite loading), *N*-methyl pyrrolidone (NMP) as solvent, Arlacel P135 as dispersant and polyethersulfone (PES) as binder, in order to find the most suitable formulation.

    b. Analyzing the viscosity of ceramic suspension prepared at different bauxite loading (45 wt.% - 60 wt.%) using viscometer.

    c. Fabricating the prepared ceramic suspension into bauxite hollow fiber precursor through tube-and-orifice spinneret using phase inversion technique.

    d. Forming the BHFM through sintering process at target temperatures ranging from 1250°C - 1450°C.

3. Characterizing the properties of BHFMs.

   a. Measuring the internal diameter (ID), outer diameter (OD) and thickness of cross-section morphology of BHFMs using scanning electron microscopy (SEM) analysis.

   b. Measuring the hydrophilicity and oleophobicity of the BHFMs using contact angle measurement.

   c. Investigating the mechanical strength of BHFMs using three-point bending test analysis.

   d. Measuring the average pore size and porosity of the membrane using mercury porosity test.

4. Performing the performance test of the selected BHFMs towards oil rejection in PW treatment using synthetic PW through cross-flow filtration mode in term of permeate flux and oil rejection:

   a. Preparing the synthetic PW with 1000 ppm using commercial heavy crude oil collected from PETRONAS Melaka Refinery Complex and then mixed with distilled water. The cross-flow system for the oil rejection was tested under the operation conditions of 30°C and 3 bar.

   b. Performing the water permeation test initially before running the rejection test.

   c. Investigating the effect of bauxite loading on the performance of the BHFMs by measuring the permeate flux and the oil rejection by using ultraviolet–visible (UV-vis) analysis.

   d. Investigating the effect of sintering temperature on the performance of the BHFMs by measuring the permeate flux and the oil rejection by using ultraviolet–visible (UV-vis) analysis.

   e. Comparing the results between the BHFMs to find which one showed the best performance.

5. Evaluating the bauxite membranes self-cleaning performance:

   a. Conducting the self-cleaning test of the BHFM using UV radiation. The BHFMs were radiated by UV light in the distilled water for 3 hours then the radiated BHFM were tested for oil rejection test.

   b. Comparing the results of the non-radiated membrane with radiated membrane by using ultraviolet–visible (UV-vis) analysis, to compare the rejection of oil.

   c. Conducting the long-term self-cleaning test to the BHFM to compare the condition of the BHFM after several run.

   d. Investigating the condition of BHFM after radiated again with UV and the result were compared with the first time UV radiation.

1.5 Significance of Research

Even though the study regarding to the treatment of PW by membrane technology are well known in the past few years, but the focus of previous studies was more on the polymeric and polymeric-based composite membranes compared to the ceramic membrane. But, when it comes to the application of ceramic membranes for the PW treatment, ceramic membranes also have some drawback which include the cost of the fabrication. So, recent studies on the ceramic membrane have been focused on the low cost ceramic materials with high chance to replace the commercial ceramic materials. However, fabrication of inexpensive ceramic hollow fiber membrane using raw bauxite especially Malaysia bauxite is less reported.

The other problem faced by ceramic membrane is membrane fouling that probably occur in the long time period of utilization. Cleaning method like chemical cleaning should handle the problem but this method could damage the membrane and lower its rejection performance. The needs of inexpensive ceramic hollow fiber with fouling resistance is crucial especially when applying to the oily PW treatment. PW contain finer particle and dissolved oil to be remove for better quality of water. Current technologies have some problems in rejecting the smaller particle and dissolved oil. So, new advanced membrane technology like ceramic hollow fiber membrane is needed to fulfill oil and gas industry strict rules for water disposal into the sea and water reinjection to the hydrocarbon reservoir. In this study, the fabrication of bauxite hollow fiber membrane is more focused on the ability of the membrane to reject the dissolved oil while maintaining its rejection performance. This study can become the pioneer of research regarding to the utilization of bauxite hollow fiber membrane (BHFM) with self-cleaning ability for the PW treatment since there is no reported yet.

1.6 Organization of the Thesis

This thesis is divided into five chapters, which are introduction, literature review, research methodology, results and discussion and conclusion and recommendations.

Chapter 1 describes the brief introduction on the PW includes its problems and the current technology that were used to treat PW. Then, the details of the problem statement, objectives and scopes of the study, also the significant of the study have clearly stated.

Chapter 2 displays the literature reviews of the topic of interest in this thesis. In this first part of the chapter, the PW are discussed thoroughly in terms on its compositions, problems and the conventional technologies of the PW treatment. Then, the next part of the chapter is more focused on the membrane technology which includes the application of conventional polymeric and ceramic membranes and the ceramic membrane materials used for the PW treatment. This part of chapter deliberates the advantageous and the limitation of the current membrane technology. Next part of the chapter is involved the low cost materials for the ceramic membrane which go thoroughly until the explanation of the bauxite material. After that, the chapter continue with the explanation of the fabrication BHFM and the characterization of BHFM. Before the summarization of the chapter, the last part of the chapter is discussed on the self-cleaning membrane and the mechanism of the self-cleaning properties.

Chapter 3 emphases on the materials, fabrication and sintering techniques, characterization approaches, water permeation setup for the oil removal in PW and the self-cleaning setup.

Chapter 4 discusses the characterization of bauxite powder and BHFM including the chemical properties, crystalline phase, rheological study and also hydrophilicity and oleophobicity of the membrane. The next part discussed on the relationship of the bauxite loading and sintering temperature on the properties of the BHFM. In this part, the most acceptable properties of BHFM were chosen in term of its morphology, mechanical strength, porosity and average pore size. The self-cleaning properties discusses on the last part of the chapter in term of water permeation of the BHFM.

Finally, **Chapter 5** stated the general conclusion for each objective that has been mentioned in the thesis. The suggestion and recommendation for the future work have also discussed in this chapter.

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