CERAMIC HOLLOW FIBRE MEMBRANES DERIVED FROM NATURAL RESOURCES FOR TREATMENT OF ARSENIC-CONTAMINATED WATER VIA MEMBRANE DISTILLATION

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ABSTRACT

Arsenic is regarded as one of the most toxic heavy metals and the largest mass poisoning material in the world. Recently, membrane distillation (MD) using hydrophobic membranes has been a promising technology for arsenic removal in water. While polymeric membranes are known to show drawbacks such as low thermal and chemical resistivity, similarly, commercial ceramic membrane from alumina that is extremely expensive. Therefore, the development of cost effective ceramic membranes from natural materials have grown inexorably to solve some of the underlying issues. In this work, hydrophobic ceramic hollow fibre membranes (CHFM) derived from natural resources (kaolin, rice husk waste and cow bone waste) were developed via phase inversion and sintering technique and modified through fluoroalkylsilane grafting. At the beginning of the study, characterization on chosen natural resources (kaolin, silica based rice husk ash and hydroxyapatite based cow bone) were performed. The prepared membranes were characterized and modified with 1H, 1H, 2H, 2H-perfluorodecyltriethoxysilane and ethanol solution for 24 hours with respect to their morphological structure, surface roughness, wettability behaviour, pore size distribution and porosity. The results revealed that the modification process successfully turned the CHFM from hydrophilic to hydrophobic with contact angle value of 145°, 157°, 161° and 170° for membranes prepared from kaolin, amorphous silica, crystalline silica and hydroxyapatite, respectively. Afterwards, the prepared CHFM were tested towards synthetic arsenic wastewater by varying direct contact membrane distillation (DCMD) parameters such as arsenic pH, arsenic concentration, and arsenic-feed temperature. It was found that CHFM prepared from kaolin (KHFM) prepared at kaolin content of 37.5 wt.% and sintered at 1300°C showed the best performance with 100% rejection of arsenite [As(III)] and arsenate [As(V)]) towards arsenic removal via DCMD system. Nevertheless, the last part of the study is treating the arsenic-contaminated water collected from Sungai Pengorak, Malaysia using the best membrane that induced 100% arsenic removal via DCMD system. When comparing the performance of the prepared membrane in this study with nanofiltration and reverse osmosis membranes, it was found that the newly-developed KHFM showed excellence performance in treating arsenic-contaminated water with 100% arsenic rejection and stable flux of 23kg/m^{2}h. It is worth mentioning that no membrane fouling was observed in the prepared KHFM for 72 hours of operation in this study compared to polymeric membranes.

ABSTRAK

Arsenik dianggap sebagai salah satu logam berat yang paling toksik dan beracun di dunia. Terkini, penyulingan membran (MD) menggunakan membran hidrofobik ditemui sebagai teknologi yang efektif untuk penyingkiran arsenik di dalam air. Sementara itu, membran polimer menunjukkan kelemahan seperti ketahanan kimia dan suhu yang rendah dan begitu juga seramik membran komersial diperbuat daripada alumina adalah sangat mahal. Oleh itu, pembangunan membran seramik yang berkos efektif daripada bahan semula jadi telah berkembang dengan pesat. Dalam kajian ini, membran gentian geronggang seramik semulajadi hidrofobik (CHFM) telah dibangunkan dari bahan seramik alternatif yang dipilih (kaolin, sisa sekam padi dan sisa tulang lembu) melalui penyongsangan fasa dan teknik persinteran dan diubah suai menerusi teknik cantuman *fluoroalkylsilane*. Pada awal kajian, pencirian pada bahan alternatif yang dipilih (kaolin, silika berasaskan abu sekam padi dan hidroksiapatit berasaskan tulang lembu) telah dilakukan. Membran terhasil dicirikan dan diubahsuai dengan larutan *1H,1H,2H,2H-perfluorodecyltriethoxysilane* dan etanol selama 24 jam terhadap struktur morfologi, kekasaran permukaan, kelakuan kebolehbasahan, taburan saiz liang dan keliangan. Keputusan yang diperoleh menunjukkan bahawa proses pengubahsuaian berjaya mengubah membran seramik dari bersifat hidrofilik ke hidrofobik dengan nilai sudut sentuh 145°, 157°, 161° dan 170° untuk membran yang disediakan daripada kaolin, silika amorfus, silika kristal dan hidroksiapatit. Seterusnya, semua CHFM diuji ke atas air sisa arsenik sintetik dengan pelbagai parameter penyulingan membran sentuhan langsung (DCMD) seperti pH arsenik, kepekatan arsenik dan suhu suapan arsenik. Keputusan menunjukkan CHFM yang disediakan daripada kaolin (KHFM) pada kandungan kaolin 37.5 % berat dan disinter pada 1300°C menunjukkan prestasi terbaik dengan penolakan 100% arsenit [As (III)] dan arsenat [As (V)]) terhadap penyingkiran arsenik melalui sistem DCMD. Pada akhir kajian, air tercemar arsenik yang yang di ambil daripada Sungai Pengorak, Malaysia dirawat menggunakan membran terbaik dan berjaya menyingkirkan arsenik 100% melalui sistem DCMD. Apabila membandingkan prestasi membran yang disediakan dalam kajian ini dengan nano-penurasan membran dan osmasis balik membran, didapati bahawa KHFM yang baharu dihasilkan menunjukkan kecemerlangan dalam merawat air tercemar dengan memberi penyingkiran arsenik 100% dan 23kg/m^{2}h fluks. Selain itu, tiada kotoran membran diperhatikan dalam KHFM sepanjang 72 jam operasi berbanding dengan membran polimer.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
AGMD	-	Air gap membrane distillation
AS	-	Amorphous silica
As (III)	-	Arsenite
As (V)	-	Arsenate
ASHFM	-	Ceramic hollow fibre membrane from amorphous silica
BOD	-	Biological oxygen demand
CA	-	Contact angle
CHFMs	-	Ceramic hollow fibre membranes derived from natural resources
CS	-	Crystalline silica
CSHFM	-	Ceramic hollow fibre membrane from crystalline silica
DCMD	-	Direct contact membrane distillation
FTIR	-	Fourier-transform infrared Spectroscopy
h-ASHFM	-	Hydrophobic ceramic hollow fibre membrane from amorphous silica
h-CSHFM	-	Hydrophobic ceramic hollow fibre membrane from crystalline silica
HHFM	-	Ceramic hollow fibre membrane from hydroxyapatite
h-HHFM	-	Hydrophobic ceramic hollow fibre membrane from hydroxyapatite
h-KHFM	-	Hydrophobic ceramic hollow fibre membrane from kaolin
KHFM	-	Ceramic hollow fibre membrane from kaolin
MCL	-	Maximum contaminant level
MD	-	Membrane Distillation
NF	-	Nanofiltration
PP	-	Polypropylene
PTFE	-	Polytetrafluoroethylene
PVDF	-	Polyvinylidene fluoride
RO	-	Reverse osmosis

SEM - Scanning electron microscopy
SGMD - Sweeping gas membrane distillation
TEM - Transmission electron microscopy
TGA - Thermogravimetric analyzer
VMD - Vapor membrane distillation
WHO - World Health Organization
XRD - X-ray diffraction

LIST OF SYMBOLS

A - Effective membrane area (m^2)
R - Rejection (%)
J - Permeate flux (kg/m^{2}h)
σ - Mechanical strength (MPa)
R_a - Surface roughness (µm)
t - Time (min)
L - Effective membrane length
P^o - Vapor pressure of water (Pa)
$£_s$ - Concentration polarization
$C_{m,f}$ - Solute concentration at feed solution (ppm)
$C_{b,f}$ - Solute concentration at bulk solution (ppm)
B_w - DCMD coefficient
α_w - Membrane activity
T - Temperature

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CHAPTER 1

INTRODUCTION

1.1 Research Background

“Fresh water is the world’s first and foremost medicine”. The world is poised at the brink of a severe global crisis especially lack of fresh water. As the population increases, water scarcity is becoming more of an issue. Water covers 70% of the world, and it is easy to think that it will always be plentiful. However, fresh water, in which referring to the precious thing that we drink, bathe in, and irrigate our farm field, only 3% of the world’s water is fresh water and 1.1 billion people lack access to clean and safe drinking water. The remaining percentage is tucked away in frozen glaciers or otherwise unavailable for our use.

Access to safe drinking water is now one of the most challenging issue to mankind due to the ever-rising water demand (Vorosmarty *et al.*, 2010). Inadequate sanitation is also a problem for 2.4 billion people. They are exposed to diseases such as cholera, typhoid fever, and other water-borne illness (i.e., diarrhoea, gastrointestinal illness). According to World Health Organization (WHO), 3.4 million people, mostly children, die each year from diarrheal disease alone (Pandey *et al.*, 2014). Among the main pollutant found in water is the family of heavy metals such as lead, arsenic, cadmium, fluoride, and mercury. Comparing to other pollutants, heavy metals are categorised to be harmful and toxic for ecosystem and human due to their acute behaviour that cannot be destroyed (Yurekli, 2016).

Arsenic is regarded as one of the most toxic heavy metal and largest mass poisoning in the world, with atomic number 33, located in group 15 of the periodic table and widely present in the environment in rocks, soils and groundwater (Bissen and Frimmel, 2003; Smedley and Kinniburgh, 2002). In fact, it has been classified as Group 1 human carcinogen by the International Agency for Research on Cancer (IARC) (Fan *et al.*, 2016). Arsenic is the 20th most abundant element in the earth's crust, 14th in seawater and the 12th most abundant element in the human body (Pal, 2015d). Consequently, there are two types of arsenic which are arsenite [As(III)] and arsenate [As(V)]. In general, arsenic can be traced in both surface water and groundwater, but higher concentration level for groundwater, as summarised in Table 1.1. Groundwater is one of the main drinking water sources, recently, to overcome shortages of clean water caused by chronic climate change for most developing countries (Basu *et al.*, 2014). Bangladesh, India, Argentina, Taiwan, China and Mongolia have been reported as among the countries that face major arsenic contamination.

Table 1.1 : Arsenic Concentration in both surface water and groundwater (Source: US-Environmental protection Agency 2000)

Sources of water	Arsenic concentration range
Air, ng/m^3	1.5-53
Rain from unpolluted ocean air, µg/L (ppb)	0.019
Rain from terrestrial air, µg/L	0.46
Rivers, µg/L	0.20-264
Lakes, µg/L	0.38-1000
Ground (well) water, µg/L	< 1.0 and > 1000
Seawater, µg/L	0.15-6.0
Soil, mg/kg (ppm)	0.1-1000
Stream/river sediment, mg/kg	5.0-4000
Lake sediment, mg/kg	2.0-300
Sedimentary rock, mg/kg	0.1-490
Biota: green algae, mg/kg	0.5-5.0
Biota: brown algae, mg/kg	30

In view of this issue, literatures have revealed that arsenic contamination can cause serious human health problem such as long-term cancer (Basu *et al.*, 2014). Most recently, in Malaysia (Sungai Pengorak, Pahang), it was reported that a very high concentration arsenic of 101.5 mg/kg (101,500 ppb) was found t in fish body where its habitat has been contaminated by bauxite. Generally, bauxite contains mainly 40-50% aluminium oxide, 20% ferric oxide and 3–5% combined silica (Valeton, 1972). However, Rajah stated that bauxite in Kuantan is characterised by high ferric oxide content ranging from 14.4 to 40.6% depending on the area (Rajah, 1984). Because of its composition, aluminium and iron are the main contaminants that pollute the water resources but depending on the geological characteristics of the land and surrounding land use activities, other toxic metals such as arsenic, mercury, cadmium, lead, nickel and manganese may also contaminate drinking water resources when the natural ecosystem is aggressively removed and excavated (Abdullah *et al.*, 2016).

For more than 100 years, many technologies have been introduced for arsenic removal from water including precipitation, coagulation, electrocoagulation, reverse osmosis, electrodialysis, adsorption, ion exchange, and membrane filtration. Conventionally, coagulation and flocculation are among the most common methods for arsenic removal. The term coagulation and flocculation are often used in single term “flocculation” that describe both process (Bratby, 2016). Consequently, hydroxide-based coagulant is the most commonly employed in flocculation process due to its eco-friendly and simplicity. However, this material does not ensure total compliance for various metals especially arsenic, since hydroxide do not completely precipitate at a single pH.

Adsorption evolved as the most promising and well-known method that can effectively remove As(III) and As(V) from water (Mohan and Pittman Jr, 2007). More than 100 papers and patents reported on arsenic removal by adsorption in literature. There are many types of adsorbents used to remove arsenic through adsorption system, such as ferrous material, surfactants, biomass waste and activated carbon. A recent review on removal arsenic from water using nano adsorbents and challenges have also been studied (Lata and Samadder, 2016). Unfortunately, in the review, it was reported that adsorption also shows some drawbacks that need urgent modification. Some of that are (i) limitation to further the technology into market due to the lack of excellent adsorbent with high adsorption capacity and unavailability for commercial scale column; and (ii) adsorption capability of different types of water pollutants.

Membrane separation has been known to be a “worldwide technology” especially towards water treatment due to its cost effective, simple operation, long-life term and need less energy (Mulder, 1996). According to some source (Strathmann, 1981), the demand of pure water flux have driven the market for crossflow membrane equipment and membranes worldwide from $ 6.8 billion in 2005 to $ 9 billion in 2008. Early investigation towards this technologies was developed from animals such as bladders of pigs, cattle or fish, and sausage casings made of animal gut (Baker, 2012). By the early 1930s, microporous collodion membranes were ready and commercially available in market. During the next 30 years, this early microfiltration membrane technology was expanded to other polymers, notably cellulose acetate, in which fabricated using phase inversion technique by Loeb and Sourirajan (Loeb and Sourirajan, 1963) that could produce membrane in asymmetric structure. Nowadays, these technologies have been divided into four types, which are microfiltration, MF (< 100 nm), ultrafiltration, UF (4-100 nm), nanofiltration, NF (1.2-12nm) and reverse osmosis, RO (< 0.5 nm) (Schäfer *et al.*, 2005).

Microfiltration (MF) membrane have the largest pore size ranging from 0.1 to 10 µm. Subsequently, arsenic can be existing in water in any form such as particulate (> 0.45 µm), colloidal (between 0.45 µm to 3000 Da) or dissolve state (< 3000 Da). Hence, by applying MF membrane alone can only remove less than 10% of arsenic, in which still falls short of target reduction below the WHO-prescribed limit of 10 µg/L. It is obviously shown that NF and RO have high potential arsenic removal through membrane separation. Figoli *et al.* (2010) applied NF membrane and rejected more than 91% of As(V) with initial feed in the range of 100-600 ppb while Yu *et al.* (2013) obtained a high As(V) removal of 97.8% through commercial NF membrane made from aromatic polyamide with existence of 40 mg/L of humic acid. In literature, it was hard to find studies that successfully done As(III) removal through NF membrane. This is due to As(III) is very small and can diffuse easily through NF membrane’s pore. Similar to NF, RO membrane also have high rejection for As(V) but very low for As(III) at neutral pH (Waypa *et al.*, 1997). In fact, water treated through RO may not consist of precious minerals such as calcium and magnesium in which concerned by human being through drinking water (Verma and Kushwaha, 2014).

Membrane distillation (MD) is a recent technology that received the most remarkable attention towards water purification including desalination and heavy metals removal. In 2008, MD was first innovated into arsenic removal and obtained 100% rejection for both As (III) and As (V) (Macedonio and Drioli, 2008). In membrane distillation system, only water vapour is allowing to pass through a microporous hydrophobic membrane. The water vapour refers to thermally driven transport of vapour pressure difference between the two sides of the membrane's pores (Khayet and Matsuura, 2011a). Unlike other methods such as RO membrane, MD rewards many unique features like low operating pressure. In fact, MD pore size is relatively larger than those membrane separations. Most importantly, MD need a hydrophobic membrane. In fact, due to this, MD possess antifouling behaviour.

Among all types of MD configuration, direct contact membrane distillation (DCMD) seems to become the first-line choice over others configuration. This is according to DCMD does not need an external condenser and very suitable for water-based application (Khayet, 2011). Furthermore, it is interesting to note here that the DCMD has the simplest MD configuration to set up. In DCMD operation system, the hot feed solution is in direct contact with hot membrane side surface, thus, evaporation takes place at the feed membrane surface. Due to evaporation, vapour formed and moved by the pressure difference across the membrane to permeate side and condense inside the membrane module. The feed solution cannot permeate into membrane pores due to the membrane hydrophobicity, which means only the gas phase exists inside the membrane pores. Qu et *al.* (2009) used polyvinylidene fluoride (PVDF) hydrophobic polymeric membrane and obtained a high rejection of > 99.95% for both As (III) and As (V) using DCMD. In fact, a high feed arsenic concentration at average of 1000 to 2000 mg/L have been tested. It is worth to mentioned that MD process in the work have been tested for more than 10 days with excellent arsenic removal.

1.2 Problem Statement

To date, membrane distillation (MD) especially direct contact membrane distillation (DCMD) is attracting widespread attention (Ashoor *et al.*, 2016) for treating wastewater containing high toxicity of heavy metals removal like arsenic. Hydrophobic polymeric membrane such as polyvinylidenefluoride (PVDF), polytetrafluoroethylene (PTFE) and polypropylene (PP) are commonly employed for MD because of their low surface energy and high hydrophobicity (El-Bourawi *et al.*, 2006; Wang *et al.*, 1999). However, polymers have disadvantages at which they have the inability to act in harsh condition such as high temperature and high chemical resistance, in which are of crucial membrane's properties for MD.

To tackle this problem, ceramic membrane with superior characteristics is able to withstand harsh conditions due to its excellent mechanical, chemical stability and thermal resistance (Li *et al.*, 2016). In general, alumina is the common ceramic material in fabrication of ceramic membrane (Norfazliana *et al.*, 2016; Ren *et al.*, 2015; Shi *et al.*, 2015). Unfortunately, ceramic membrane from alumina shows some drawbacks and dramatic alteration due to high sintering temperature up to 1500ºC to reach a compromise between mechanical strength and porosity using micron-sized alumina powder (Li *et al.*, 2016). At this high sintering temperature, in addition to the alumina powder itself that known to be a high cost material, thus making the ceramic membrane extremely expensive. In addition, when high sintering temperature is used, the fabrication process will be prolonged.

Realising the huge potential that is offered by ceramic membrane, therefore, alternative ceramic material from natural resources such as clays, ashes from agricultural wastes and animal bone wastes were recently used as new material for the fabrication of alternative ceramic membrane (Eom *et al.*, 2015; Saffaj *et al.*, 2013; Tolba *et al.*, 2016). Generally, there are three types of clays that are commonly used in industrial which are kaolin, ball clay and bentonite. In this regard, kaolin is a white ceramic powder that are used widely in ceramic filling and coating applications. Among all clays, kaolin is the most popular alternative ceramic material towards fabrication of ceramic membrane (Bouzerara *et al.*, 2006; Harabi *et al.*, 2015; Hedfi *et al.*, 2016). To be noted, kaolin provides low plasticity, high refractory and hydrophilic properties to the membrane, in which extremely desired for membrane characteristic especially towards water filtration (Mgbemena *et al.*, 2013; Mittal *et al.*, 2011). Whereas, the issue of utilizing abundantly agricultural waste such as rice husk, sugarcane bagasse and bamboo leaves is remained unsolved. Interestingly, these wastes could be simply converted into precious ceramic material, which is silica in the form of ashes, through calcination process. In literatures, it was found that rice husk is one of the most silica rich raw materials containing about 90-99% silica, compared to other waste (Alyosef *et al.*, 2018). In fact, it can be turned into amorphous and crystalline silica depending on the calcination temperature. Meanwhile, bio-ceramic based material called as hydroxyapatite (HAp) can be produced from animal bone wastes like cow bones, fish bones and pig bones through calcination at temperature of 800-1000°C (Brzezińska-Miecznik *et al.*, 2015). To produce large amount of HAp powder, cow bone wastes are commonly used due to its size and abundantly available as wastes.

Another remarkable problem is that, most of these ceramic membranes are hydrophilic due to their nature of surface hydroxyl (Ren *et al.*, 2015). Consequently, a literature search revealed that this problem can be solved by simple surface modification with low surface energy materials before used for MD. Ceramic membrane grafted with silane agents like fluoroalkylsilanes (FAS) have been receiving most attention in turning hydrophilic properties of ceramic membrane into hydrophobic. The pioneer for modification from hydrophilic to hydrophobic ceramic membrane was first reported by Larbot in 2004 (Larbot *et al.*, 2004). In the work, hydrophobic ceramic was obtained with contact value at the range of 150° for desalination application through MD. Almost 100% rejection of salt rejection was obtained in the study, proving that ceramic membrane can be used as promising membrane, replacing the polymeric membrane in MD.

Based on the above mentioned problems, this study focused on preparation and characterization of ceramic hollow fibre membrane derived from natural resources (CHFM) which are kaolin, amorphous and crystalline silica (AS an CS) and cow bone waste that obtained from natural resources of clays, agricultural waste, and animal bones waste, respectively, through a phase inversion and sintering technique. Afterwards, the prepared CHFMs were subjected towards hydrophobization process to modify the surface of CHFMs from hydrophilic to hydrophobic. Consequently, the modified CHFMs were tested on arsenic synthetic wastewater removal via DCMD system at various parameters such as arsenic concentration, arsenic pH, and arsenic feed temperature. Finally, a arsenic-contaminated water will be treated at long term operation.

1.3 Objectives and Scopes

The main objective of this study is to develop ceramic hollow fibre membranes derived from natural resources (CHFMs) with hydrophobic properties via phase inversion/sintering technique for the use in membrane distillation system to remove arsenic from water. This objective has been achieved by accomplishing the following specific objectives:

a) To prepare and characterize alternative ceramic material obtained from natural resources (kaolin, amorphous silica, crystalline silica, and hydroxyapatite based cow bone waste).
b) To fabricate and characterize ceramic hollow fibre membranes (CHFMs) from kaolin, amorphous silica, crystalline silica, and hydroxyapatite based cow bone waste using phase inversion/sintering technique in term of their physical and chemical behaviours.
c) To graft and characterize hydrophobic layer onto selected ceramic hollow fibre membranes (CHFMs) using FAS silane agent and examine its physical and wettability properties.
d) To evaluate the separation performance of selected hydrophobic ceramic hollow fibre membranes (CHFMs) towards arsenic removal in water using direct contact membrane distillation.
e) To evaluate the performance of best hydrophobic ceramic hollow fibre membranes (CHFMs) towards arsenic-contaminated water for long term process.

In order to achieve the objectives, seven scopes have been identified in this research. The scopes are:

a) Preparing and characterizing alternative ceramic materials obtained from natural resources which are kaolin, amorphous silica, crystalline silica, and hydroxyapatite based cow bone wastes:
    i. Drying all the alternative ceramic materials in oven before used. Converting the rice husk and cow bone into ceramic powder through calcination process.

    ii. Measuring the morphology and size of all alternative ceramic materials using transmission electron microscopy (TEM) and Brunauer-Emmett-Teller (BET) theory.

    iii. Investigating the chemical and physical properties of all alternative ceramic materials using x-ray fluorescence (XRF), x-ray powder diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) and thermogravimetry/differential thermal analysis (TG/DTA).

b) Fabricating the ceramic hollow fibre membrane via phase inversion and sintering technique:
    i. Preparing the ceramic suspension containing ceramic powder of natural resources (kaolin, rice husk and cow bone) as main material at different content (35 to 50 wt.%), N-methyl pyrrolidone (NMP) as solvent, Arlacel P135 as dispersant and polyethersulfone (PESf) as binder, in order to find the most suitable formulation.
    ii. Analysing the viscosity of ceramic suspension prepared at different content using viscometer.
    iii. Shaping the ceramic suspension into ceramic hollow fibre precursor through tube-and-orifice spinneret using phase inversion technique.
    iv. Forming the final alternative ceramic hollow fibre membrane through sintering process at different temperatures from 900 to 1500ºC.

c) Characterizng the properties of ceramic hollow fibre membranes (CHFMs):
    i. Measuring the surface and cross section morphology of ceramic hollow fibre membranes using scanning electron microscopy (SEM) analysis.
    ii. Investigating the mechanical strength of ceramic hollow fibre membranes using three-point bending test analysis.
    iii. Identifying the porosity and pore size distribution using mercury intrusion porosimetry analysis.

d) Grafting and characterizing the selected ceramic hollow fibre membranes from each alternative material into hydrophobic ceramic membrane using FAS silane agent:
    i. Grafting the ceramic hollow fibre membranes through immersion process with mixture of FAS agent and ethanol for 24 hours.
    ii. Comparing the surface morphology and roughness of pristine and hydrophobic ceramic hollow fibre membranes using scanning electron microscopy (SEM) and atomic force microscopy (AFM).

    iii. Evaluate the changes in mechanical strength of pristine and hydrophobic natura ceramic hollow fibre membranes using 3-point bending test analysis

    iv. Measuring the wettability properties of pristine and hydrophobic ceramic hollow fibre membranes using liquid entry pressure of water measurement (LEPw) and contact angle test.

e) Performing the performance test of selected hydrophobic ceramic hollow fibre membranes towards arsenic removal using synthetic wastewaters through direct contact membrane distillation in term of permeate flux and arsenic rejection:
    i. Preparing the synthetic arsenic wastewater into two types which are arsenite [As(III)] and arsenate [As(V)].
    ii. Investigating the effect of membrane’s sintering temperature as function difference membrane’s pore size.

    iii. Investigating the effect of arsenic pH ranging from 3 to 11. It should be noted that the pH 7.48 is used instead of pH 7 as initial pH from the previous study on effect of membrane's sintering temperature.
    iv. Investigating the effect of arsenic concentration of 1, 50, 100, 500 and 1000 ppm.
    v. Investigating the effect of arsenic feed temperature range from 40 to 80°C.

f) Evaluating the performance of best hydrophobic ceramic hollow fibre membranes towards arsenic-contaminated water collected from Sungai Pengorak, Pahang, Malaysia through direct contact membrane distillation :
    i. Measuring the permeate flux and arsenic rejection for long term operation at 70 hours.
    ii. Comparing the permeate flux and arsenic rejection with pressure driven membrane (nanofiltration and reverse osmosis) from literatures.
    iii. Comparing the permeate flux and arsenic rejection with polymeric membrane in membrane distillation from literatures.

1.4 Rational and Significance of the Study

This study contributes to the development of ceramic hollow fibre membranes, at the same time, beneficial to the researchers in this area regarding to the knowledge on preparation of ceramic membrane using combined phase inversion and sintering technique. It is acknowledged that commercially available ceramic membrane is commonly made from alumina that has high cost and high melting point. Therefore, attempts are made to investigate the potential of natural resources of ceramic materials from clays, agricultural waste, and animal bones waste as main material in fabrication process of ceramic membrane and able to compete economically with commercial alumina membranes. Besides, other advantages of ceramic membranes such as hollow fibre configuration and modification of its hydrophilicity behaviour into hydrophobic are also interesting topic to study.

In addition, this study lead to direct implications towards industry, especially for mining industry, for treating contaminated water from heavy metals, such as, arsenic using membrane distillation technology. Recently, unregulated bauxite mining activity in the Malaysian state of Pahang has led to an alarmingly arsenic contamination. Accordingly, it was found that the high level of arsenic was measured in the contaminated fishes and water which is more than 100 times the legal amount of arsenic allowed by the Food Regulation 1985 and Water Quality Standard by Malaysian Health of Ministry. Thus, this study could be beneficial to the researchers in this area and support the government policies.

1.5 Organization of the Thesis

This thesis is organized into nine chapters addressing on fabrication of ceramic membrane prepared at different ceramic content and sintering temperature, then modified into hydrophobic membrane through simple FAS grafting method and application on arsenic removal through DCMD process. Figure 1.1 presents the overall thesis structure.

Chapter 1 outlines brief information on membrane separation technologies towards arsenic removal including MD process. Then, the detail of the problem statements, objectives and scopes of this study have also been stated in detail.

Chapter 2 presents literature reviews about the main topics of this thesis. In this chapter, background information on conventional and recent technologies towards arsenic removal are discussed. A comprehensive review is presented on the arsenic toxicity and its conventional treatment, preparation of hydrophobic ceramic membrane through surface modification process and various type of alternative ceramic membranes prepared from natural resources such as clays and wastes. The review also provides various fabrication steps available for ceramic membrane, factors that affect membrane structure and membrane configuration as well as advantages and disadvantages.

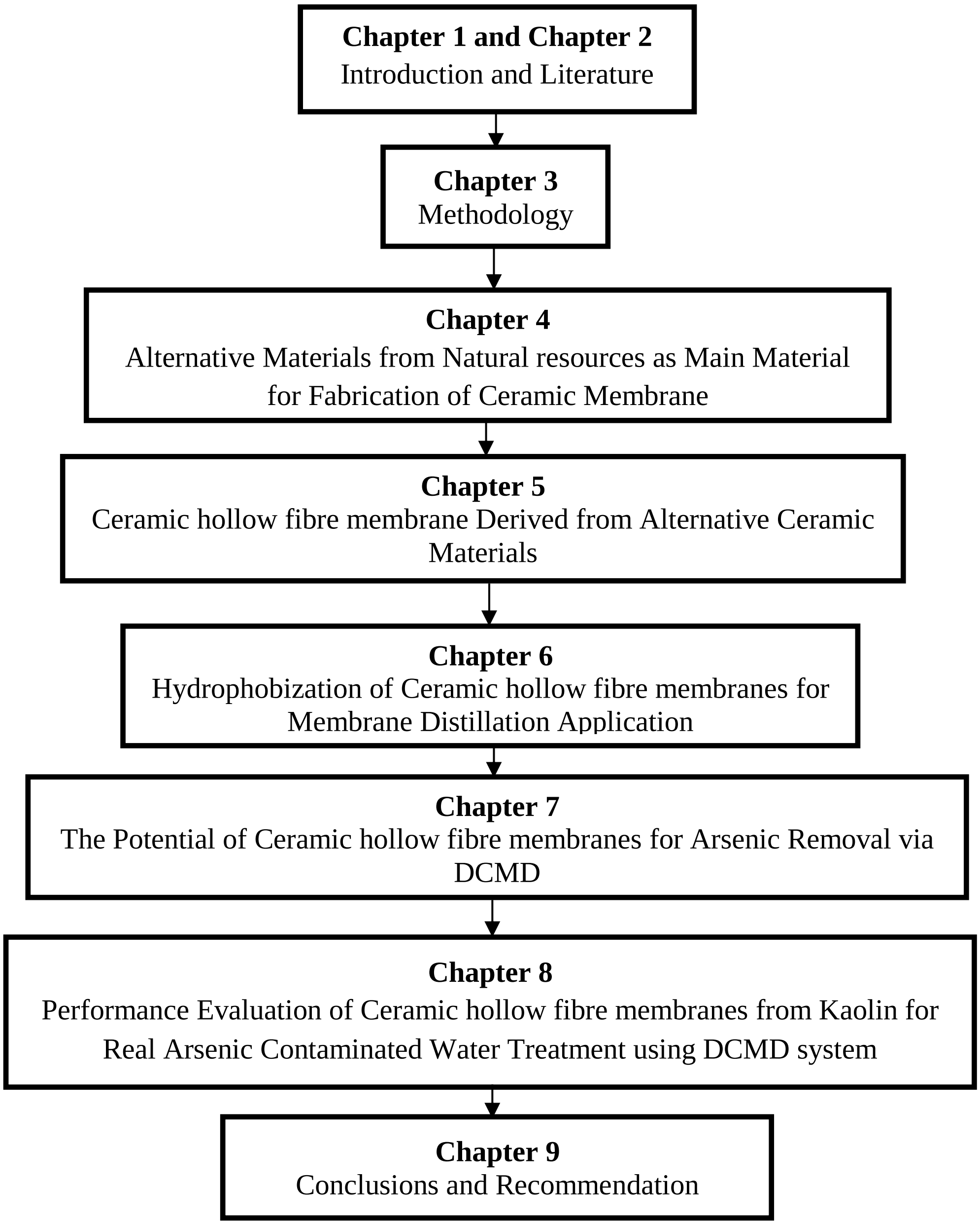

Figure 1.1 Overall thesis structure

Chapter 3 focuses on the materials, working procedures, characterization methods and DCMD experimental setup for arsenic removal.

Chapter 4 describes in detail the characterization of alternative materials from natural resources (kaolin clay, rice husk waste and cow bone waste) as main material prior to fabrication of ceramic hollow fibre membranes. The characterization includes particles morphology, the crystallinity behaviour, infrared spectrum, and adsorption-desorption analysis. Herein, rice husk waste is burned at 600°C and 1000°C to produce amorphous and crystalline silica, respectively. Meanwhile, cow bone was burned at 800°C to produce ceramic material in powder form.

In **Chapter 5**, preparation, and characterization of ceramic hollow fibre membranes from natural resources and prepared by combined phase inversion and sintering technique were studied in detail. The effect of ceramic content and sintering temperature towards membrane morphologies and mechanical strength were studied. Afterwards, membrane pore size analysis and porosity were further measured to investigate the effect of sintering temperature.

Meanwhile, **Chapter 6** discusses in detail the preparation and characterization of hydrophobic ceramic hollow fibre membranes through hydrophobization with FAS silane agent. The effectiveness of FAS grafting surface on ceramic hollow fibre membranes were investigated in term of surface morphologies and surface roughness, presence of F1 atom measured by XPS analysis, wettability behaviour includes contact angle and liquid entry pressure analysis, difference in mechanical strength as well as membrane pore size and porosity.

Consequently, **Chapter 7** presents the potential of hydrophobic ceramic hollow fibre membranes performance in DCMD process for arsenic removal. The effect of arsenic concentration, arsenic pH and feed temperature were also investigated in detail towards the permeate flux and arsenic rejection performance of each prepared hydrophobic ceramic hollow fibre membranes. Interestingly, hydrophobic ceramic hollow fibre membrane prepared from kaolin clay at 37.5 wt.% content and sintered at 1300°C recorded excellent performance.

In **Chapter 8**, the performance of excellence hydrophobic ceramic hollow fibre membranes from kaolin clay were investigated towards arsenic-contaminated wastewater taken from Sungai Pengorak, Kuantan. This chapter also evaluates the membrane stability of hydrophobic ceramic hollow fibre membranes from kaolin clay by testing for long term operation. Finally, the general conclusions and some recommendation are given in **Chapter 9**, outlining the directions for further research and optimization. A preliminary study on some recommendation were also tested and discussed in appendices.

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