ADSORPTIVE HOLLOW FIBRE CERAMIC MEMBRANE DERIVED FROM PALM OIL FUEL ASH FOR ARSENIC REMOVAL

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ABSTRACT

The use of ceramic membranes as sorbents has more advantages compared to polymer filtration systems. However, their production high costs with regards to raw materials has somewhat limited further applicability. Thus, the potential of palm oil fuel ash (POFA), an agricultural waste as a low cost adsorbent for the removal of arsenite (*As*(III)) and arsenate (*As*(V)) was explored. This study comprised of two stages: (1) POFA powder: characterization and adsorption mechanism and (2) POFA adsorptive hollow fibre ceramic membrane: properties and separation performance. In stage 1 of the study, the POFA powder were characterized using nitrogen adsorption-desorption, field emission scanning electron microscopy-energy-dispersive X-ray spectroscopy mapping, X-ray fluorescence, X-ray diffraction, Fourier transform infrared spectroscopy and thermogravimetric analyses while adsorptivity activity was examined by batch adsorption studies. The maximum adsorption capacities of 78.0 and 94.6 $mg \cdot g^{-1}$ for *As*(III) and *As*(V) were achieved when the smallest particle size of 30 µm POFA was used and increased from 18.75 to 99.4 $mg \cdot g^{-1}$ for both *As* species with increasing of calcination temperature from 900 to 1150 °C. Desorption test revealed that *As*-loaded POFA was stable in water. The equilibrium data was better described by the pseudo-second-order model for both *As*(III) and *As*(V) while in adsorption isotherm study, the data were better fitted to the Langmuir isotherm model. All the results were then optimized by response surface methodology which concluded that calcination temperature has a major significance in the adsorption proses. Further attempt of molecular modeling study using the density functional theory via Gaussian 09 software consequently identified optimized structure of SiO^- molecule and the energy for the proposed mechanism routes between the As^+ species. In stage 2, based on excellent properties and condition from stage 1 namely 30 µm particle size and calcination temperature, POFA hollow fibre ceramic membrane (PHFCM) was fabricated via phase inversion and sintering technique at three different sintering temperatures i.e. 1100, 1150 and 1200 °C, by which the samples were named as PHFCM-1100, PHFCM 1150 and PHFCM-1200 respectively. The characterization analyses clearly showed that the PHFCM was constructed of two concentric rings with rich composition of Si and Al. The highest mechanical strength of 52.84 MPa and permeation flux of 250.73 $L/m^2.h$ of PHFCM-1150 was in favor for adsorption of *As* species yielding maximum adsorption capacities corresponding to 95.62 and 98.34 $mg \cdot g^{-1}$ of *As*(III) and *As*(V) which then were selected for further exploration with ozonation study. The enhanced adsorption of *As*(III) and *As*(V) by the PHFCM-1150 was associated during pre-ozonation. For post-ozonation, 3 min exposure time used had permitted satisfactory cleaning of PHFCM-1150 to mitigate fouling problem while allowing repeated usages of the adsorbent for *As* removal. The performance of with and without ozonated PHFCM-1150 was evaluated with real wastewater samples and showed almost total rejection of arsenic contamination which signified the possible implementation in real wastewater system. Finally, this study has demonstrated that adsorptive PHFCM was effective and its respective *As* removal met the maximum discharge limit of 10 µg/L set by the world health organization and the national legislation in Malaysia.

ABSTRAK

Penggunaan membran seramik sebagai penjerap mempunyai banyak kebaikan berbanding sistem penapisan polimer. Walaubagaimanapun, kos pembuatan yang tinggi disebabkan oleh bahan mentah telah sedikit sebanyak mengehadkan penggunaannya. Justeru, keupayaan abu bahan bakar kelapa sawit (POFA), iaitu bahan buangan pertanian sebagai penjerap kos rendah bagi menyingkirkan arsenit (*As*(III)) dan arsenat (*As*(V)) telah diterokai. Kajian ini mempunya dua peringkat: (1) serbuk POFA: pencirian dan mekanisma penjerapan dan (2) membran seramik gentian berongga POFA: sifat dan prestasi pemisahan. Pada peringkat 1 kajian, serbuk POFA ini dicirikan dengan analisa fizijerapan nitrogen, pengimbas-elektron-pancaran-medan pemetaan spektroskopi imbasan-X, pendarfluor sinar-X, belauan sinar-X, spektroskopi inframerah jelmaan Fourier dan analisis termogravimetri manakala aktiviti penjerapan diuji dengan kajian-kajian penjerapan kelompok. Kapasiti penjerapan maksima sebanyak 78.0 and 94.6 $mg{\cdot}g^{-1}$ bagi *As*(III) dan *As*(V) dicapai apabila POFA partikel saiz terkecil pada 30 µm digunakan dan meningkat daripada 18.75 ke 99.4 $mg{\cdot}g^{-1}$ untuk kedua-dua spesis *As* dengan suhu kalsin dinaikkan dari 900 ke 1150 °C. Kajian penyahjerapan telah mendedahkan bahawa *As* terkandung POFA adalah stabil di dalam air. Data keseimbangan kedua-dua *As*(III) dan *As*(V) sesuai digambarkan oleh model tertib kedua pseudo, manakala dalam kajian penjerapan isoterma, data sesuai dipadankan dengan model isoterma Langmuir. Kesemua data kemudiannya dioptimumkan dengan kaedah sambutan permukaan yang merumuskan suhu kalsin ialah kesan utama dalam penjerapan. Percubaan lanjut pemodelan molekul menggunakan teori ketumpatan fungsian melalui perisian Gaussian 09 telah mengenalpasti struktur optimum molekul SiO^- dan tenaga bagi laluan mekanisma yang dicadangkan untuk tindakbalas antara spesis As^+. Pada peringkat 2, berdasarkan sifat cemerlang dan keadaan pada tahap 1 seperti saiz partikel 30 µm dan suhu kalsin, membran seramik gentian berongga POFA (PHFCM) disediakan melalui songsangan fasa dan teknik pensinteran pada 3 suhu kalsin berbeza i.e. 1100, 1150 dan 1200 °C, yang mana sampel masing-masing dinamakan PHFCM-1100, PHFCM-1150 dan PHFCM-1200. Analisa-analisa secara jelas menunjukkan bahawa PHFCM terdiri daripada dua cincin sepusat dengan komposisi Si dan Al yang tinggi. PHFCM-1150 yang mempunyai kekuatan mekanikal 52.84 MPa dan fluks penelapan tertinggi 250.73 L/m^3h telah menjadi pilihan untuk penjerapan spesies *As* dengan menghasilkan kapasiti penjerapan maksima 95.62 $mg.g^{-1}$ *As*(III) dan 98.34 mg.g-1 *As*(V) telah dipilih untuk kajian ozonisasi selanjutnya. Peningkatan penjerapan *As*(III) dan *As*(V) oleh PHFCM-1150 adalah dikaitkan semasa pra-ozonisasi. Untuk pasca-ozonisasi, tempoh pendedahan 3 min telah membenarkan pembersihan PHFCM-1150 yang memuaskan untuk mengurangkan masalah kotoran yang membenarkan pengunaan berulangkali bahan jerap untuk membuang *As*. Prestasi PHFCM dengan dan tanpa ozonasi telah diuji dengan sampel air sisa sebenar dan menunjukkan penolakan sepenuhnya pencemaran *As* yang menandakan pelaksanaan yang mustahil dalam sistem air sisa sebenar. Akhir sekali, kajian ini telah memperlihatkan bahawa PHFCM sangat berkesan dan penyingkiran *As* telah memenuhi piawaian 10 µg/L tahap maksimum pelepasan yang ditetapkan pertubuhan kesihatan sedunia (WHO) dan perundangan kebangsaan di Malaysia.

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LIST OF ABBREVIATIONS

B3LYP	-	Becke-3 and Lee Yang Par
BET	-	Brunauer-Emmett-Teller
BJH	-	Barrett-Joywen-Halenda
CCD	-	Central Composite Design
DFT	-	Density Functional Theory
DNA	-	Deoxyribonucleic acid
DOE	-	Design of Experiment
EPA	-	Environmental Protection Agency
FESEM	-	Field Emission Scanning Electron Microscope
FTIR	-	Fourier Transform Infrared Spectroscopy
ICP-OES	-	Inductively Coupled Plasma Atomic Emission Spectroscopy
MD	-	Membrane Distillation
MF	-	Microfiltration
MNPs	-	Magnetic Nanoparticles
NF	-	Nanofiltration
NLDFT	-	Non-Local Density Functional Theory
NOM	-	Natural Organic Matter
ORP	-	Oxygen Reduction Potential
O-PHFCM	-	Ozonated Palm Oil Fuel Ash Hollow Fibre Ceramic Membrane
PHFCM	-	Palm Oil Fuel Ash Hollow Fibre Ceramic Membrane
PHFCM-1150	-	Palm Oil Fuel Ash Hollow Fibre Ceramic Membrane at 1150 °C
POFA	-	Palm Oil Fuel Ash
POME	-	Palm Oil Mill Effluent
RSM	-	Response Surface Methodology
TGA	-	Thermogravimetric Analysis
UF	-	Ultrafiltration
UNICEF	-	United Nation Children’s Fund
UTM	-	Universiti Teknologi Malaysia
XRD	-	X-Ray Diffraction
XRF	-	X-Ray Fluoresces

LIST OF SYMBOLS

V	-	Volume
m	-	Weight
C_0	-	Initial Solution Concentration
C_e	-	Equilibrium Concentrations
$C_{As-desorp}$	-	Pressure
$C_{As-adsorbent\ loaded}$	-	Moment Of Inersia
q_m	-	Radius
n	-	Adsorption Intensity
K_F	-	Adsorption Capacity
K_L	-	Langmuir Constant
λ	-	Wavelength
θ	-	Angle
A	-	Area
t	-	Time
F	-	Maximum Load
L	-	Length
D_o	-	Outer Diameter
D	-	Inner Diameter
T	-	Temperature
β	-	Full width
Q	-	Adsorption capacity
q_t	-	Adsorption capacity at given time
q_e	-	Adsorption capacity at equilibrium

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Water is vital to life and there is limited amount of clean water on earth (Fenner, 2017). Evidently in 2025, half of the world's population is predicted to be facing serious water scarcity (Boretti and Rosa, 2019). This is alarming since water is basic necessity for human, also both in industry and agriculture consumption. The world dependence on water might be risky due to the water contamination affected by man-made rapid industrialization and deforestation (Ukaogo *et al*., 2020). Many literatures have reported that there are several contaminants found in industrial wastewater and surface water, which include heavy metals. Presence of heavy metals in water body is harmful to the environment and their consequences are well stated in many research study (Kulkarni *et al*., 2014).

Globally, the natural contamination of *As* has been reported in drinking water supplies in more than 70 countries and the majority of these nations belong to South Asian and Southeast Asian regions (Ravenscroft *et al*., 2009). The elevated levels of $As > 50$ µg/L in water have been reported in different countries of the world like Argentina, Bangladesh, China, Chile, Hungary, India, Pakistan, Mexico, Vietnam and as well as in many parts of the USA (Smedley and Kinniburgh, 2002). The presence of *As* in environment has gained considerable attention in the last decade because studies have reported alarming contamination levels of *As* in West Bengal of India and neighboring locations in Bangladesh (Nickson *et al*., 2000; Das *et al*., 2004). Similar to the other developing nations, Pakistan is also facing serious issues of water shortage and contamination in the available water resources. Previous reports showed that Pakistan has mostly exhausted its available freshwater resources due to low water storage capacity (Azizullah *et al*,. 2011). Now the country included in water stressed nations and if this scenario continues, it will likely to declare as water scarce nation (Hashmi *et al*., 2009). Worldwide, approximately 1.1 billion people have no access to clean water for drinking, about 2.5 billion people have lack of proper sanitation facilities, and waterborne disease-mediated annual death toll has been exceeded more than 5 million (Hinrichsen and Tacio, 2002). According to national survey, only 56% households in Pakistan have access to clean and safe drinking water (Farooq *et al*., 2008; Ullah *et al*., 2009). This situation is more worse in the eye of the international standards for safe and drinkable water, because as per international standards only 25.61% people in Pakistan have access to safe drinking water and this population includes 23.5% rural and 30% urban masses (Rosemann, 2005). Anthropogenic activities are mainly responsible for contamination of drinking water in densely populated areas of Pakistan, and authorities have declared groundwater as not drinkable.

The increasing reported incidences of arsenic (*As)* contamination in our food and water has seen *As* becoming a top priority toxicant for risk assessment and exposure reduction/mitigation (ASTDR, 2011). Prolonged consumption of *As*-contaminated food and drinking water can invoke a plethora of severe health complications, typically manifested as multiple organ disorders. Among other complications include different forms of cancers, skin problems, cardiovascular diseases, and conceivably respiratory, as well as kidney diseases (Steinmaus *et al*., 2014; Quddus *et al*., 2016; Kuo *et al*., 2017), due to variations in interindividual metabolism of the inorganic *As*. Most environmental problems associated with *As* contamination are related to the mobilization of two forms of ionized *As* in natural water environment, namely *As*(III) and *As*(V). In conjunction to being more toxic, *As*(III) is more mobile and its elimination from water sources is more challenging than *As*(V). Nonetheless, their removal ability remains limited, and there is still much to be done to improve current methods for removing *As* in water environments.

The past two decades have witnessed rapid advancements in membrane technology, which leads to the development of an array of membrane-based adsorbents for trapping arsenic from water. Such adsorbents have garnered considerable interests in the scientific and industrial community because of their low energy consumptions (Kumar *et al*., 2019) alongside with the high water quality of permeate (Wang *et al*., 2019). Similarly, ceramic membranes are increasingly being used in water treatment owing to their reliability, long life span, high hydrophilicity, and thermally, chemically, and mechanically stable, in addition to the high permeate flux at low operating pressures (Dong *et al*., 2018). Nonetheless, the introduction of ceramic membranes to a water treatment plant warrants a serious economic feasibility evaluation due to their relatively high initial investment cost compared to polymeric membranes (Hubadillah *et al*., 2018; Saja *et al*., 2018). Thus, there is a growing need to develop inexpensive and greener ceramic membranes for decontamination of heavy metals in water.

Despite the wide availability of natural polymers, many studies explore the feasibility of the silica-rich oil palm agricultural waste as adsorbent material for the aforementioned application remain relatively scarce. As a matter of fact, oil palm agricultural biomass is a promising renewable source of silica-based adsorbent material, especially for palm oil producing countries like Malaysia and Indonesia. In Malaysia alone, an enormous amount of oil palm biomass is generated all year round by the palm oil industry, but only a small fraction is converted into value-added products (Onoja *et al*., 2017) while the remaining is discarded. Approximately 4.5 million tons of oil palm solid waste, in particular, palm oil fuel ash (POFA) is produced per year (Yusof *et al*., 2018), thus implying the potential of POFA as a raw material for preparing adsorbents to clean up *As* contaminated water. The approach seems quite feasible as well as attractive since untreated POFA contains as much as 68% of SiO_2 (73). Moreover, waste material is cheap (Tai *et al*., 2018; Hubadillah *et al*., 2020), biodegradable and renewable source, and show low carbon dioxide release. The naturally abundant surface polar groups, i.e., silanols (Si–OH) and siloxanes (Si–O–Si) on POFA are easily tunable (Elias *et al*., 2017; Elias *et al*., 2018) in order to attract and adsorb *As* ions in water. In fact, POFA was successfully used as adsorbent to remove chromium (Cr^{6+}) in a batch distillation column study (Aziz *et al*., 2013).

While silica-based adsorbents have been extensively tested as adsorbents of cationic pollutants in water (Xiong *et al*., 2019; Yue *et al*., 2019), membrane fouling remains a bottleneck in wastewater treatment applications, as it decreases the flux permeability. Consequently, the operational cost has become costly due to an increasing energy consumption and higher quantity of chemicals required to regenerate the ceramic membranes (Gao *et al*., 2018; Shoener *et al*., 2016). To mitigate the impacts attributed to this bottleneck, suitable desorption techniques need to be developed in order to enhance membrane performance, so that the systems become affordable and reliable. The integration of adsorptive ceramic membrane and catalytic ozonation process has gained popularity in recent year due to its effectiveness to mitigate membrane fouling, as well as to degrade organic pollutants during water treatment (Song *et al*., 2018). The fouling control mechanisms of the integrated ozonation and ceramic membrane filtration process include direct molecular ozone oxidation and hydroxyl radical oxidation of feed and foulants deposited on the membrane surface and within the membrane pores (Wei *et al*., 2016).

Ozone is a powerful oxidant that preferentially oxidises electron-rich moieties containing carbon–carbon double bonds and aromatic alcohols. Therefore, the same process may adequately alleviate fouling in the ceramic membrane filtration system, considering that the method is effective for periodic cleaning of membranes in chemical processing (Kim *et al*., 1999). Envisioning possible synergistic interactions between ozonated Si–OH and Si–O–Si-rich surface on the POFA hollow fibre ceramic membrane (PHFCM), the treated material may produce a surplus of free radicals, i.e., reactive oxygen species that would interact with *As* and improve the sorption of *As*(III) and *As*(V) from water, as well as achieve rapid desorption of the trapped *As*. In view of the promising results seen in the previous study (Yusof *et al*., 2018), another seminal study, which investigates the technological values of PHFCM for removal of *As* in aqueous solutions becomes mandatory to further uncover its novelty for water treatment applications.

In spite of their promising potentials, reports on the use of ozonation on an inorganic ceramic membrane for improving adsorption efficacy and regenerability of the adsorbent, is sparse in the body of literature. Herein, the study proposed the use of a modified POFA suspension as adsorbent to recover *As*(III) and *As*(V) in water. The best optimum conditions were then being applied in further study of POFA suspension modified to hollow fibre configuration. To the best of our knowledge, a hollow fibre ceramic membrane from a POFA powder has yet to be tested for decontamination of *As* in water. The most stable route of adsorption mechanism by the developed POFA suspension was verified by a molecular modeling approach using the density functional theory. In this study also, we investigated the effect of pre- and post-ozonation on the modified POFA hollow fibre ceramic membrane (PHFCM) for removing two forms of arsenic, i.e., *As*(III) and *As*(V), from water. The best treatment conditions to maximize sorption/diffusion of *As* by the PHFCM were studied. In addition, the adsorption/desorption behaviour of this contaminant on pre- or post- ozonated Si-O in POFA was studied to determine its effect on the adsorption capacity of the adsorbent for the two *As* species. The World Health Organization (WHO) and the United States Environmental Protection Agency (EPA) have stipulated that 10 $\mu g \cdot L^{-1}$ is the maximum contaminant level (MCL) for *As* in safe drinking water. Pertinently, this work firmly supports the "Zero Waste" initiative by the Malaysian government for which to maximize the use of oil palm biomass. The study advantageously transforms the widely available but discarded POFA into a functional material to solve an environmental problem.

1.2 Problem Statement

The presence of arsenic in natural waters is critical at a global scale, and its removal is vital importance. While adsorption is the most adopted method for arsenic removal, coagulation, flocculation, precipitation, ion exchange, and membrane filtration are also used. Despite their simplicity, cost effectiveness and higher removal efficiency, adsorption however facing a serious adsorbent requirement. An ideal adsorbent should has high adsorption capacity, affinity for both the inorganic arsenic species (*As*(III) and *As*(V)) and should be effective under relevant environmental conditions.

For years, a porous structure polymeric membrane has been a promising technology for water treatment. While the role of polymer membranes as sorbents resides in their high porosity and ease of functionality compared to other membrane forms, adsorptive polymeric membrane such as PVDF and PTFE are vulnerable to chemical attack beside the extreme conditions of high temperature and pressure. Other drawbacks include the prohibitive cost due to constrains in sources, high energy consumption during manufacture, lower surface area due to covering of polymer on the adsorbent surface as well as prone to reduce membrane lifetime due to frequent regeneration/desorption process using chemicals. Thus ceramic membrane derived directly from adsorbent material like POFA is the way forward. The abundance alongwith easy availability and appropriate chemical composition suggest the POFA as the suitable low cost and greener source for preparing ceramic membrane. But mechanism properties and ideal formulation of ceramic membrane from POFA are still not reported in the literature.

Previously, commercialized silica adsorbent has been extensively showed a remarkable performance in adsorbing cationic arsenic species (As^{+}) in water because of the presence of SiO^{-}, however hindered by their need in a high volume scale and not in favourable immobilize compact module. Silica-rich POFA could be an alternative of silica sources due to less leaching problem and may contribute as a low cost arsenic adsorbent. Other than well-known high silica composition, a detailed characterization study is vital to be conducted in order to investigate the influence of other properties possessed by POFA that may contribute in their adsorption performance. Due to the novel silica source material, a research on POFA as an adsorbent is minimal and there is only scanty study proven their molecular interaction with contaminant *As*.

A hollow configuration could offer a high surface area to volume ratio of hollow fibre membrane, asymmetric structures with finger-like voids and sponge-like pores and a single step fabrication which delivers a higher adsorption capacity. Despite their excellent features, ceramic hollow fibre membrane showed severe membrane fouling cases because a thick and compressed cake layer, containing relatively large amounts of organic and inorganic matter was formed. It was very hard to remove, especially under high-turbidity conditions without any treatment. This phenomenon could hinder their performance and might shorten their life cycle. Cleaning of ceramic membrane with ozone treatment and study their behaviour towards before and after ozonation are still deficient. Previously, extensive backwash method has been utilized however facing implementation problem such as low efficiency and high chemical usage. Research focusing on the cleaning parameters by ozonation should be carried out in order to utilize the ceramic membrane performance. To overcome the above mentioned problems, silica-rich POFA fabricated as a low cost material for hollow fibre ceramic hybrid membrane (adsorption + filtration) can enhance the removal of arsenic in water by providing more active site due to the high surface area, a mullite structure and deliver a new insight of SiO-*As* molecular interaction as well as a fouling mitigation under an additional ozonation treatment which further enhance their performance as an adsorptive membrane.

1.3 Objectives of Study

Objective of this study is to produce a high performance adsorptive hollow fibre ceramic membrane from agricultural waste material to remove arsenic in water. The objective of this study could be specified as follows:

1. To investigate their arsenic removal performance under effects of particle size, adsorbent dosage, initial pH solution, powder calcination temperature, and competing anions.

2. To optimize the adsorption parameter towards the arsenic removal of POFA powder via Response Surface Methodology (RSM) and model the interaction and energy pathway of arsenic adsorption mechanism in a computational study.

3. To fabricate POFA hollow fibre ceramic membrane (PHFCM) and evaluate the effect of sintering temperature on their physicochemical properties, mechanical strength, water flux and arsenic adsorption performance.

4. To investigate the effect of pre and post-ozonation technique in different exposure time on the mitigation of the fouling problem and enhancement of the adsorption capacity of PHFCM.

1.4 Scopes of Study

The scope of this study consist of four parts namely; characterization and adsorption performance of POFA powder, optimization and interaction mechanism of POFA powder towards arsenic adsorption, fabrication and adsorption performance of POFA hollow fibre ceramic membrane and last but not least, the effect of pre- and post-ozonation treatment onto POFA hollow fibre membrane. In the first part, the characterization of POFA and their adsorption performance were conducted by drying at 24 hours followed by grinding and sieving to fine particles sizes based on varying scale (30, 50, 60, and 125 μm). The powder POFA using experimental set up under various parameters effect (initial pH solution (pH 3 to 13), adsorbent loading (0.2-1.2 g), calcination temperatures (500 -1150 °C) and particle sizes (30, 50, 60 and 125 μm)). Next, the optimization study and interaction mechanism between POFA-*As* contaminants was optimized via response surface methodology (RSM) using a central composite design (CCD) model which composed of dosage (A), solution pH (B), particle size (C) and calcination temperature (D) and as factors with 16 runs.The optimize model of SiO_2 was calculated under density functional theory (DFT) calculations using Gaussian09 suite of programs to study the interaction of SiO_2 and *As* ion species.

In the third stage, the fabrication of POFA hollow fibre membrane (PHFCM) was conducted by preparing the ceramic suspension dope containing 40wt % POFA powder, N-methyl pyrrolidone (NMP) as a solvent, Arlacel P-135 the dispersant and Polyethersulfone (PESf) to act as a binder. The ceramic suspension into a hollow fibre ceramic membrane was fabricated via phase inversion/sintering technique in a single spinneret by using tap water both as an internal and external coagulant. The effect of sintering temperature (1100, 1150 and 1200 °C) towards their physicochemical properties, mechanical strength, water flux permeation and arsenic removal performance was analysed. The mechanical strength of PHFCM was conducted via three-point bending analysis while water flux was tested through permeation system.

The excellent properties and removal performance of POFA hollow fibre ceramic membrane (PHFCM) was further treated under ozonation approach. The PHFCM was tested in post- and pre- ozonation setup with different exposure time (1, 3 and 5 min) under 600-800 mV of oxidation-reduction potential (ORP) values. Post-ozonation test performed in this study involved the ozonation of PHFCM-1150 after the batch adsorption study, where the *As*-loaded PHFCM was ozonated under different exposure time (1, 3 and 5 min) as a cleaning step of the fouling problem. While, pre-ozonation was done on PHFCM-1150 before the adsorption study to examine its effect on the adsorption capacity of the adsorbent for the two *As* species.

1.5 Significance of Study

In this study, low cost POFA is a new and emerging material in fabricating a hollow fibre configuration membrane for arsenic adsorption in water. As compared to conventional silica adsorbent, abundance waste POFA has major composition of silica which is among contributing factor in trapping arsenic onto the membrane surface. POFA hollow fibre configuration was constructed with two concentric rings providing more active sites for adsorption to occur. The pores of the inner ring showed pores that were finger-like voids, whereas the outer PHFCM ring was constructed of asymmetrical pores of three distinct voids (i.e. macro-, meso-, and microvoids). All these unique features were proven in contributing towards excellent adsorption performance. Detailed molecular study on the interaction between silica in POFA with *As* species in water were giving a new insight in optimizing the adsorption in molecular level. Besides, the proposed pathway energyfrom molecular study, promoted a selection of lowest energy consumption in the permeation system. Additionally, new approaches of ozonation technique onto POFA inorganic membrane delivering a promising solution on fouling problem and in same time increase the adsorption capacity.

1.6 Thesis Outline

This thesis begins with Chapter 1 describing the research background, problem statement, objectives, scope and significant of this research. Chapter 2 reviewed the literatures related to the POFA material, others low cost adsorbent, conventional silica adsorbent, the ozonation technique, fabrication of hollow fibre membrane configuration and molecular modelling approach. Chapter 3 described the experimental and characterization of the adsorbent. Chapter 4 discusses the findings from stage 1 of the study which involved POFA powder characterizations and adsorption performance, as well as molecular computational approach and optimization study. Chapter 5 is the stage 2 of this study, involved the deliberating on fabrication of POFA as hollow fibre membrane and their adsorption performance towards *As* species. The effect of pre and post-ozonation, regeneration and performance on waters sampling were further discussed throughout the chapter. The conclusions and recommendation for future studies were stated in Chapter 6.

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LIST OF PUBLICATIONS

1. **Yusof, M.S.M**., Othman, M.H.D., Wahab, R.A., Jumbri, K., Razak, F.I.A., Kurniawan, T.A., Samah, R.A., Mustafa, A., Rahman, M.A., Jaafar, J. and Ismail, A.F. (2020). Arsenic adsorption mechanism on palm oil fuel ash (POFA) powder suspension. *Journal of hazardous materials, 383*, p.121214. **(Q1, 9.038)**

2. **Yusof, M.S.M**., Othman, M.H.D., Wahab, R.A., Samah, R.A., Kurniawan, T.A., Mustafa, A., Rahman, M.A., Jaafar, J. and Ismail, A.F. (2020). Effects of pre and post-ozonation on POFA hollow fibre ceramic adsorptive membrane for arsenic removal in water. *Journal of the Taiwan Institute of Chemical Engineers*. **(Q1, 4.794)**

3. **Yusof, M.S.M**., Othman, M.H.D., Mustafa, A., Rahman, M.A., Jaafar, J. and Ismail, A.F. (2018). Feasibility study of cadmium adsorption by palm oil fuel ash (POFA)-based low-cost hollow fibre zeolitic membrane. *Environmental Science and Pollution Research*, *25*(22), pp.21644-21655. **(Q2, 3.306)**

4. **Yusof, M.S.M**., Othman, M.H.D., Wahab, R.A., and Ismail, A.F. (2020). Ceramic Membrane for Wastewater Treatment. *UTM Press*. **(Indexed by SCOPUS)**