PREPARATION, CHARACTERIZATION AND PERFORMANCE OF ZEOLITIC IMIDAZOLATE FRAMEWORK-8 MEMBRANE ON ALUMINA HOLLOW FIBER FOR LEAD(II) REMOVAL

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DEDICATION

This thesis is dedicated to my beloved husband for his tolerance and motivation throughout this study. Special appreciation to my mother for her unconditional love and care and for always being supportive. For my father, thank you very much for always taught me that perseverance and hard work are the pivotal part of success . To all my children, thank you very much for lending your time so that I can accomplish this journey. My siblings, really appreciate all the continuous trust and support. Last but not least, my friends, thank you for always motivating. Thank you all!

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ABSTRACT

Exposure to lead may cause severe health issues to human and life-beings. Therefore, the purpose of this study was to develop the zeolitic imidazolate framework-8 (ZIF-8) membrane on alumina hollow fiber for lead(II) removal in aqueous system. The first part of this study was to fabricate an alumina hollow fiber membrane as the support for the ZIF-8 membrane. The effects of alumina particle size on the morphology of alumina hollow fiber were determined by using several analyses including the porosity, surface roughness and mechanical strength. The performance of all the prepared membranes were determined using cross-flow filtration system. The results indicated that higher loading of fine particles produced alumina hollow fiber membrane with lower porosity and a higher ability to reject protein molecules. Three different preparation techniques were employed to determine the effects of the techniques on the growth of the ZIF-8 layer on the selected alumina support. The techniques include, in situ solvothermal method, combined in situ secondary growth, and layer-by-layer technique. The ZIF-8 membranes were characterized using several techniques including Fourier transform infrared spectrospcopy and X-ray diffractometer. A batch adsorption study was performed to evaluate the adsorption efficiency of the ZIF-8 membranes. Among all of the prepared membranes, the SC48 showed the highest performance for lead(II) adsorption with the ability to remove up to 67% of the lead(II) ions with the adsorption capacity of 115.38 mg/g. The cross-flow filtration test indicated that the SC48 exhibited the highest removal percentage, with the ability to remove up to 90% of lead(II) ions. Additionally, the regeneration study proved the reusability of the ZIF-8 membrane. The optimization study had shown that the optimum condition for removal of lead(II) ions using ZIF-8 membrane was at the initial concentration of 94.00 ppm, pH of 7.15, and operational pressure of 2.0 bar. Experimental test under optimized condition showed that lead(II) removal was at the average of 92.13% and the water permeation of 288.41 $Lm^{-2}h^{-1}bar^{-1}$. The outcome of this study suggested that the ZIF-8 membrane had shown excellent performance as an adsorptive membrane for lead(II) removal and deserves further in-depth development.

ABSTRAK

Pendedahan kepada plumbum boleh mengakibatkan kesan yang teruk kepada kesihatan manusia dan juga hidupan yang lain. Oleh yang demikian, tujuan kajian ini dijalankan adalah untuk menghasilkan membran kerangka imidazolat zeolitik-8 (ZIF-8) di atas permukaan gentian geronggang alumina bagi tujuan penyingkiran plumbum(II) di dalam air. Bahagian pertama kajian ini adalah untuk menghasilkan gentian geronggang alumina sebagai lapisan sokongan kepada membran ZIF-8. Kesan saiz partikel serbuk alumina terhadap morfologi gentian geronggang alumina ditentukan dengan beberapa analisis seperti penentuan keliangan, kekasaran permukaan dan kekuatan mekanikal. Keupayaan kesemua membran yang terhasil adalah ditentukan dengan menggunakan system penurasan aliran silang. Hasil daripada kajian ini, didapati bahawa dengan meningkatkan jumlah nisbah saiz partikel yang halus mampu menghasilkan gentian geronggang alumina yang mempunyai keporosan yang rendah dan berkeupayaan tinggi dalam menyingkirkan molekul protein. Tiga teknik penyediaan yang berbeza telah digunakan untuk menentukan kesan teknik-teknik tersebut terhadap pertumbuhan lapisan ZIF-8 di atas gentian geronggang alumina yang terpilih. Teknik yang digunakan adalah seperti teknik solvoterma in situ, kombinasi in situ dan pertumbuhan sekunder serta kaedah lapisan demi lapisan. Membran ZIF-8 dicirikan dengan menggunakan beberapa teknik pencirian termasuk spektroskopi infra-merah penjelmaan Fourier dan sistem pembelauan sinar X. Kaedah penjerapan kelompok digunakan untuk menilai kecekapan penjerapan membran ZIF-8. Antara kesemua membran yang dihasilkan, SC48 menunjukkan potensi yang paling tinggi dalam penyerapan ion pumbum(II) dengan kebolehan penyingkiran ion plumbum(II) sebanyak 67% dan kapasiti penjerapan sebanyak 115.38 mg/g. Kajian melalui sistem penurasan aliran silang pula membuktikan bahawa membran yang sama juga menunjukkan kebolehan menyingkirkan ion plumbum(II) sebanyak 90%. Kajian penjanaan semula membuktikan bahawa membran ZIF-8 menunjukkan keupayaan untuk diguna semula. Kajian pengoptimuman menunjukkan bahawa keadaan optimum untuk penyingkiran ion plumbum(II) menggunakan membran ZIF-8 adalah pada kepekatan awal sebanyak 94.00 ppm, pH 7.15 dan pada tekanan operasi sebanyak 2.0 bar. Eksperimen yang dijalankan pada keadaan optimum menunjukkan bahawa penyingkiran ion plumbum(II) secara purata adalah sebanyak 92.13% manakala kebolehtelapan air adalah sebanyak 288.41 $Lm^{-2}h^{-1}bar^{-1}$. Hasil kajian ini menunjukkan bahawa membran ZIF-8 menunjukkan potensi yang besar sebagai membran penjerap ion plumbum(II) dan kajian lanjutan boleh dijalankan untuk meningkatkan potensi membran ini.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

3D	-	three dimensional
AFM	-	Atomic Force micsroscopy
FTIR	-	Fourier Transform Infra-Red
NMP	-	N-methyl-2-pyrrolidone
PSF	-	polysulfone
UV-Vis	-	ultraviolet-visible spectrophotometry
WCA	-	water contact angle
XRD	-	X-ray diffraction spectroscopy
ZIF-8	-	Zeolitic Imidazolate Framework-8

LIST OF SYMBOLS

%	-	percentage
°C	-	degree celcius
μm	-	micrometer
Å	-	Angstrom
C_0	-	initial concentration
Ce	-	residual concentration
g	-	gram
h	-	hour
kDa	-	kilo Dalton
L	-	litre
m	-	mass
M	-	molar
pH	-	pH value
Ra	-	average plane roughness
rpm	-	rotary per minute
t	-	time
V	-	volume

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Problem Background

“Do not waste water even if you were at a running stream.” Prophet Muhammad ﷺ quoted those words more than 1400 years ago to signify the pivotal role of water and the importance of water conservation. Water is the major source of life. Each person on earth needs at least 20 to 50 litres of clean water for daily activities, including drinking, cooking, and cleaning. Consuming polluted water is a major concern and need to be taken care of because it might cause death to human and life-beings. Industrialization has grown at a very fast rate, contributed to the demand for natural resources. The mining activities have led to environmental pollution due to the release of inorganic ions, organic pollutants, organometallic compounds, radioactive isotopes, and nanoparticles (Briffa et al., 2020).

Heavy metal wastewater originated from urbanization and industrialization activities, leading to the accumulation of heavy metal in the water bodies. The high solubility of heavy metals in aquatic environments leads to the accumulation of heavy metal throughout the food chain. The accumulation might cause harmful effects to humans because the concentration of heavy metals will increase along the food chain. The common heavy metals present in industrial wastewater include nickel, zinc, silver, lead, iron, chromium, copper, arsenic, cadmium, and uranium. Among all, lead is known to be the most hazardous heavy metal, presence in 0 and +2 oxidation state in aqueous solutions, but mostly as +2 in the neutral condition.

The toxicity of lead might affect almost all of the human's organ system especially the nervous system (Wani et al., 2015). Other effects include anemia, an increase in blood pressure, and severe brain and kidney damage. The high exposure to lead may cause miscarriage and fertility issues. Therefore, there is an urgent need to treat the lead wastewater to avoid serious health issues to humans and other life-beings.

Numerous approaches have been taken to overcome the lead pollution in water. The common techniques include biological treatment (Jeong et al., 2019), chemical precipitation (Chen et al., 2018), ion exchange (Lalmi et al., 2018), electrochemical (Tran et al., 2017), adsorption (Zhou et al., 2020) and membrane technology. Among all of the approaches, adsorption is the most preferable technique due to the easy operation, low cost, and availability of a wide range of adsorbents. There are numerous types of adsorbents for instance activated carbons, zeolites, clay, silica beads, low-cost adsorbents from industrial by-products or agricultural wastes, and polymeric materials (Khulbe and Matsuura, 2018a). The emergence of Metal-Organic Framework (MOF) materials has become the current obsession among researchers due to their easy synthesis route and adjustable physicochemical properties for example their pore size (Lee et al., 2013; Deng et al., 2021) Therefore, the works on MOF adsorbent for heavy metal removal have shown an increasing trend year by year. However, the adsorption technique exhibited significant drawbacks, for instance, the generation of secondary pollution in the sludge after the adsorption process, the requirement for a large operation area, and also the complex process for regeneration of the adsorbents. Given this, researchers have come to a solution by synergizing the advantages offered by the adsorption technique and the membrane technology to further enhance the wastewater treatment process. In recent years, the number of study on adsorptive membrane has showed the increasing trends due to the straightforward separation process and the simple regeneration process (Huang and Cheng, 2020).

Several works have been reported on the fabrication and performance study of adsorptive membrane for lead(II) removal by incorporating different types of adsorbents into polymeric and ceramic membranes. The use of a ceramic membrane is more preferable for lead(II) removal applications due to the stability towards the harsh environment, ability to resist high temperature and pressure with lower fouling effect, and the easy cleaning process. Technically, in comparison with the polymeric membranes, the ceramic membranes possess high porosity, well-defined pore size distribution, high hydrophilicity, and high permeance. The high porosity of ceramic membrane can sometimes be the major problem for the rejection of molecules especially ions. Therefore, further modification on the ceramic membrane surface is a possible measure to develop a selective layer on top of the membrane.

The modification of ceramic membrane can be tailored via a few techniques for instance the *in situ* growth, seeding, and secondary growth method, layer-by-layer method, wet gel conversion method, and contra-diffusion method. Proper selection of synthesis method is very crucial to ensure that the ZIF-8 layer on top of the alumina support is not easily peeled-off and able to remain during the filtration process. Mostly, the common preparation method is *in situ* hydrothermal synthesis. The ceramic membrane is exposed to the ZIF-8 precursors under controlled conditions to enable the nucleation process to occur on the surface of the membrane. Another possible method for deposition of ZIF-8 layer on top of alumina membrane is via seeding and secondary growth approach. The approach involving the seeding process of ZIF-8 on top of the membrane, then subsequently exposed the seeded membrane to the ZIF-8's precursors. The method is considered one of the most effective and flexible methods to control the membrane thickness and uniformity of the ZIF-8 layer growth on the support membrane. The layer-by-layer synthesis approach is another attractive technique for the formation of a continuous layer of ZIF-8 on ceramic support. The method involves the deposition of ZIF-8 crystals without pre-seeding step or surface modification of the membrane. The support membrane is exposed to the precursors one at a time without mixing the precursors. The step-by-step approach enables the formation of a thin layer of ZIF-8. Whilst, the dry gel, and wet gel conversions involve the deposition of a ZIF-8 gel on the support then the crystallization in the presence of triethylamine, ethylenediamine, and water. This type of synthesis able to avoid homogenous nucleation and have advantages in minimizing waste generation. Therefore, it is a crucial step to select the best ZIF-8 membrane approach that may produce a good layer for lead(II) removal for this study.

1.2 Problem Statements

ZIF-8 is a type of MOF material, with promising potential in the adsorption of heavy metals(Zhao et al., 2015; Huang et al., 2018; K. Li et al., 2021). The ZIF-8 material has an aperture size of 0.4 nm, which is smaller than other MOF particles for example UiO-66 and MOF-5. The feature makes it to become a suitable candidate for the adsorption of lead(II) ions. However, most of the studies involving the adsorption of lead(II) ions in water utilized ZIF-8 are in the form of powder, thus the regeneration process of the adsorbents is rather a challenge. Therefore, fabricating the ZIF-8 in the form of a membrane is one of the simple approaches to enable the regeneration process to be possible.

However, embedding the ZIF-8 particles on top of the alumina hollow fiber membrane is another issue to be tackled. Generally, the phase inversion fabrication process produces membranes in the range of microfiltration (MF) or ultrafiltration (UF). In this study, the alumina hollow fiber membrane serves as the support layer for the fabrication of the ZIF-8 layer on the membrane. The support layer plays important role in the determination of the membrane's permeability and stability. Besides, the quality of the selective layer formed also depends on the surface pore size of the membrane. The previous study indicated that the large pore size of the support membrane had caused the one prominent issue, which is the penetration of the small particles of the precursors into the membrane that will later affect the selective layer formation and the permeance of the membrane.

Several kinds of researches had been done to overcome the penetration of the selective layer's precursors issue, includes coating the support membrane with sol-gel. However, the method had caused the peel-off problem of the selective layer. Thus, it is very important to have a support membrane with a smaller pore size to reduce the penetration of small particles into the support membrane. Although extensive studies have been reported on the preparation of alumina hollow fiber membrane since years ago, there is still limited study focusing on the preparation of the membrane specifically for reducing the pore size of the membrane without sacrificing its mechanical strength and permeability. The most applied particle size ratio for ceramic suspension preparation is 7:2:1 (1μm: 0.05 μm: 0.01 μm), developed by Kingsbury and Li. The ratio produced membrane pore size of around 5 μm for the macrovoid finger-like structure and 0.16 μm for the sponge-like structure. Thus, it is a great interest to study on the effect of different particle size loading to improve the morphology and performance of alumina ceramic membrane as support.

Apart from the morphology of membrane support, another important factor determining the morphology and performance of the ZIF-8 membrane is the deposition technique employed to grow the ZIF-8 layer. For water filtration, the ZIF-8 membrane has to be in a uniform and continuous layer, with a strong attachment to the support to avoid the peel-off problem that might affect the performance during the filtration process. The most common and simple deposition method is via *in situ* solvothermal method. However, the method usually causes the non-uniform and non-continuous growth of the ZIF-8 layer because the nucleation and crystal growth occur at the same time. Therefore, to reduce the defects problems that occurred via *in situ* solvothermal method, several researchers successfully grown the uniform layer of ZIF-8 membrane using the secondary seeded growth method. The seed of ZIF-8 was deposited on the membrane via various approaches, for instance, dip-coating hot-dip coating, reactive seeding, and contra-diffusion method. The seeding method via the abovementioned method involving physical interaction thus no strong attachment between support and the ZIF-8 layer. Strong chemical interaction is crucial especially for membrane applied for water treatment to avoid detachment of the ZIF-8 layer.

The application of ZIF-8 membrane for gas separation is very well established. However, the study on lead(II) removal using ZIF-8 membrane is still very not well discussed. Since the membrane acts both as an adsorbent and filter at the same time, it is worth defining the mechanism of action of the membrane.. Therefore, in this study, the adsorption mechanism and filtration performance of ZIF-8 membrane for lead(II) removal are determined and discussed.

Last but not least, the determination of the optimum condition for lead(II) ions removal is another important aspect to be focused on. Therefore, the optimization study using RSM is conducted in this study for this purpose.

1.3 Research Objectives

This study aims to develop a ZIF-8 membrane on an alumina hollow fiber membrane as support for the removal of lead(II) ions from an aqueous solution. The specific objectives of the study are as follows:

1) To study the effect of alumina particle size on the morphology and performance of the alumina hollow fiber membrane as the support layer.
2) To evaluate the effects of different synthesis approaches on the morphology and the performance of the ZIF-8 membrane.
3) To optimize the parameters for lead(II) ions removal by ZIF-8 membrane via Response Surface Methodology approach.

1.4 Research Scope

The scopes of study have been identified and are listed below:

Scope for objective 1: Fabricating the alumina hollow fiber membrane as support for ZIF-8 membrane.

a) Preparing the ceramic dope solution by using three different size of alumina powder (1 µm:0.5 µm: 0.01 µm) at five different ratio (9:1:0, 9:0:1, 8:1:1, 7:2:1 and 5:3:2).
b) Fabricating the alumina hollow fiber membrane by phase-inversion and sintering technique.

c) Characterizing the morphology and mechanical strength of alumina hollow fiber membrane by using a scanning electron microscope (SEM), mercury intrusion porosimeter (MIP), atomic force microscope (AFM), and three-point bending test.

d) Performing water permeation test and Bovine Serum Albumin (BSA) rejection test as screening for the selection of alumina hollow fiber membrane support

Scope for objective 2: Deposition of ZIF-8 particles on the alumina hollow fiber membrane via in situ hydrothermal methods, secondary growth method, and layer-by-layer method.

a) Pre-modifying the support membrane by depositing 3-(aminopropyl) triethoxysilane (APTES).

b) Synthesizing the ZIF-8 layer on the alumina support using the APTES modified and unmodified membrane. The composition of synthesis solution ratio is maintained for all the method used (0.65 HCOONa: 1.0 $Zn(NO_3)_2$: 1.5 Hmim: 450 DMF). The duration of synthesis for *in situ* hydrothermal method and secondary growth is varied from 24 hours to 72 hours. For the layer-by-layer method, the number of cycles is varied from one cycle to two cycles.

c) Characterizing physicochemical properties of ZIF-8 membranes using Fourier-Transform Infrared Spectroscopy (FTIR), X-ray Diffractometer (XRD), and Field Emission Scanning Electron Microscope (FESEM).

d) Studying the adsorption performance of ZIF-8 as adsorptive membrane by conducting the batch adsorption study and filtration test using a cross-flow filtration system. The concentration of lead(II) solution is determined using Atomic Absorption Spectroscopy (AAS). The mechanism of lead(II) removal by ZIF-8 is also be determined.

e) Evaluating the effect of factors that determine the performance of the membrane performance via a one-factor-at-a-time (OFAT) approach including the effect of pH (6-8), initial feed concentration (10-120 ppm), and operational pressure (1-2 bar).

f) Investigating the reusability of the ZIF-8 membrane after the filtration process.

Scope for objective 3: Optimization study for lead(II) removal using ZIF-8 membrane.

a) Studying the optimum parameters of ZIF-8 membrane to optimize the flux and removal of lead(II) removal. The parameters include pH of the feed solution (pH 6-8), initial concentration (80-120 ppm), and operational pressure (1-2 bar).

1.5 Significance of Study

The present study consists of the development of the ZIF-8 membrane, a type of adsorptive membrane for lead(II) removal in an aqueous system. The utilization of alumina hollow fiber membrane as the support membrane, instead of the polymeric membrane makes the membrane stable during the operation and not easily prone to fouling. The stability also contributes to the longer lifetime and reusability of the membrane. ZIF-8 was chosen as the selective layer due to the ultrahigh porosity, which benefits the coordination of lead(II) and molecular sieving capacity of the membrane. The fabrication of adsorptive membrane via the incorporation of ZIF-8 membrane on the alumina hollow fiber is a promising contribution to the wastewater treatment technology because the process includes the removal of contaminants includes adsorption and filtration of the lead(II) ions, with no requirement to filter out the adsorbent for recovery purpose.

1.6 Thesis Organization

This thesis is divided into five chapters as below:

Chapter 1: This chapter consists of the background of the study with a brief introduction about the lead(II) contamination issue and the technologies involved in the treatment of the contamination. This chapter also comprises the details of problem statements, research objectives, research scopes, and the significance of the study of the research.

Chapter 2: This chapter consists of the reviews on the previous studies related to the research interest. In this chapter, the lead effects to human and environments, and conventional methods for lead removal are reviewed. Besides, the recent approaches for lead removal are also being discussed. This chapter also deliberates the advantages and limitations of membrane technology and the advancement of the technology for better performance, specifically related to the modification of membrane Details on the synthesis technique for the ZIF-8 grown on porous support are also conveyed in this chapter. Apart from that, this chapter discusses on the adsorption study of lead(II) ions includes the adsorption isotherms and adsorption kinetic information from previously reported studies. The last part of this chapter describes a brief explanation of the optimization study using Response Surface Methodology.

Chapter 3: This chapter describes the materials, synthesis techniques, characterization, and methodology for performance studies of ZIF-8 membrane as an adsorptive membrane.

Chapter 4: This chapter discusses the data of fabricated alumina hollow fiber membranes includes their morphology and filtration performance. This chapter also includes the discussion on the effects of three different ZIF-8 synthesis techniques on the morphology and performance of the adsorptive membranes for lead(II) removal. The details on batch adsorption study along with the adsorption mechanism and important parameters involved are also described. In addition, the filtration performance includes flux and removal of lead(II) using ZIF-8 membrane are also explained in this chapter. The reusability and regeneration study of ZIF-8 membrane is also being discussed in this chapter. The last part of this chapter discusses the optimization study for the removal of lead(II) using the ZIF-8 membrane. The optimization study was carried out by selecting the membrane showing the highest lead(II) removal. The desirability test was also performed to verify the adequacy of the developed model.

Chapter 5: Summarizes the research study, includes the contribution of knowledge as well as the recommendations for future work.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Awang Chee, D.N,** Ismail, A.F., Aziz, F., Mohamed Amin, M.A., & Abdullah, N. (2020). The influence of alumina particle size on the properties and performance of alumina hollow fiber as support membrane for protein separation, *Separation and Purification Technology, 250*, 11714. (Q1, IF: 5.774).
2. **Awang Chee, D.N,** Ismail, A.F., Aziz, F. & Mohamed Amin, M.A. (2021). Copper Adsorption on ZIF-8/ Alumina Hollow Fiber Membrane: A Response Surface Methodology Analysis, *Arabian Journal of Science and Engineering, 46*, 6775-6786.(Q3, IF: 1.711)
3. **Awang Chee, D.N.,** Aziz, F., .Ismail, A.F., Mohamed Amin, M.A. Abdullah, M.S. (2021). Dual-function ZIF-8 membrane supported on alumina hollow fiber membrane for copper(II) removal, *Journal of Environmental Chemical Engineering, 9 (4),* 105343. (Q2, 5.909).

Book Chapters

1. **Chee D.N.A.,** Amin M.A.M., Aziz F., Jaafar J., Ismail A.F. (2020) Development of Advanced Nanocomposite Membranes by Molecular Sieving Nanomaterials (zeolite and MOF). Editor(s): Sadrzadeh M., Mohammadi T. In Micro and Nano Technologies, Nanocomposite Membranes for Water and Gas Separation, Elsevier.
2. Amin M.A.M., Goh P.S., Ismail A.F., **Chee D.N.A.** (2020) Recent Strategies in Designing Antifouling Desalination Membranes. In: Saji V., Meroufel A., Sorour A. (eds) Corrosion and Fouling Control in Desalination Industry. Springer, Cham.