

ZEOLITE HOLLOW FIBRE MEMBRANE FOR NICKEL REMOVAL

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ABSTRACT

This work was aimed to develop the zeolite hollow fibre adsorptive membrane and also examine the feasibility test of composite zeolite hollow fibre membranes, with UV-curable resin (UCR) as a secondary coating material for the removal of Ni(II). The preparation of the membrane began with the preparation of an alumina hollow fibre membrane as a support. After that, the zeolite crystals were deposited on the outer surface of alumina support using two different techniques namely *in-situ* crystallization and secondary growth method. The X-ray diffraction analysis (XRD) and morphological study using field emission-scanning electron microscopy (FESEM) showed the zeolites were well crystallized on alumina support for both methods. The membranes' performance in Ni(II) removal were tested using batch adsorption study with both preparation methods shows a slightly different adsorption capacity. Under optimum conditions, the S5 membrane prepared using secondary growth method showed a slightly high adsorption capacity of 126.2 mg g⁻¹ with 63% of nickel ions removal from aqueous solution within 180 min of contact time. However, due to a slightly different performance, the *in-situ* crystallization method has been selected as a preparation method for zeolite membrane throughout the current studies because this preparation method is simpler. Parameters affecting the growth of zeolite layer on alumina support were also studied to obtain the most efficient membrane. The concentration of zeolite, the time of crystallization and the number of zeolite coating layers affect the morphological characteristics of the zeolite membrane. In order to increase the stability of zeolite layer on the surface of alumina support, a photopolymerization process has been done. The addition of UCR layer on the outer surface of zeolite membranes has preserved the zeolite particles from being detached from the alumina support. The addition of UCR layer also has increased the hydrophobicity as the zeolite membranes themselves were exceptionally hydrophilic. The hydrophobicity of the membrane was determined using water contact angle measurement. PMDL membrane gave the highest contact angle value (117.75°) which means that this membrane is very hydrophobic. The performance of zeolite membranes were tested for forward osmosis (FO) process with the most efficient membranes were applied for the removal of Ni(II) from aqueous solution. Filtration test proved that the zeolite membrane enabled to reduce the concentration of heavy metal at ppb level. For the FO process, the result shows that MSL3 membranes enabled a reduction of reverse solute once incorporated with UCR. The lowest reverse solute flux obtained was 0.008 kg m⁻² h⁻¹. However, the UCR was unstable in the presence of Ni(II), which later formed complex ions. Adsorption of Ni(II) ions has caused an agglomeration of zeolite particles, causing membrane defects. The present work has successfully developed an alternative zeolite membrane for the removal of heavy metals from water sample.

ABSTRAK

Tujuan kajian ini adalah untuk membangunkan membran penjerap gentian berongga zeolit dan juga membincangkan tentang ujian keupayaan komposit membran gentian berongga zeolit, dengan resin rawatan-UV (UCR) sebagai bahan salutan kedua dalam penyingkiran Ni(II). Penyediaan membran dimulakan dengan menyediakan membran gentian berongga alumina sebagai sokongan. Selepas itu, kristal zeolit didepositkan ke atas permukaan luar sokongan alumina dengan menggunakan dua teknik berbeza yang dinamakan kaedah pengkristalan in-situ dan kaedah pertumbuhan sekunder. Analisis pembelauan sinar-X (XRD) dan kajian morfologi menggunakan medan pemancaran pengimbasan mikrografi (FESEM) menunjukkan zeolit telah dikristalisasi dengan baik di atas sokongan alumina bagi kedua-dua kaedah. Prestasi membran dalam penyingkiran Ni(II) telah diuji menggunakan kaedah penjerapan berkumpulan dengan kedua-dua kaedah penyediaan menunjukkan hanya sedikit perbezaan dari segi kapasiti penjerapan. Di bawah keadaan optimum, membran S5 yang disediakan melalui kaedah pertumbuhan sekunder telah menunjukkan kapasiti penjerapan yang sedikit tinggi iaitu 126.2 mg g^{-1} dengan 63% pemindahan ion nikel dari sampel akues dalam masa 180 min waktu sentuh. Walaubagaimanapun berdasarkan kepada perbezaan prestasi yang sedikit, kaedah pengkristalan in-situ telah dipilih untuk digunakan dalam keseluruhan kajian semasa kerana kaedah ini lebih ringkas. Parameter-parameter yang memberi kesan kepada pertumbuhan lapisan zeolit di atas sokongan gentian berongga alumina telah dikaji bagi mendapatkan membran yang paling berkesan. Kepekatan zeolit, masa pengkristalan dan bilangan lapisan zeolit telah memberi kesan kepada karakter morfologi membran zeolit. Dalam meningkatkan kestabilan lapisan zeolit di atas permukaan sokongan alumina, satu proses foto-pempolimeran telah dilakukan. Penambahan lapisan UCR di atas permukaan luar membran zeolit telah memelihara zarah zeolit daripada terlepas sokongan alumina. Penambahan UCR juga telah meningkatkan sifat hidrofobik kerana membran zeolit itu sendiri terlalu hidrofilik. Sifat hidrofilik telah dikenalpasti melalui kaedah pengukuran sudut sentuh air. Membran PMDL mempunyai nilai sudut sentuh air yang tinggi (117.75°) yang mana bermaksud membran ini sangat hidrofilik. Prestasi membran zeolit telah diuji untuk proses osmosis ke hadapan dengan membran yang lebih efisien telah diaplikasi untuk memindahkan Ni(II) dari sampel akues. Kajian penapisan telah membuktikan membran zeolite berupaya untuk menurunkan kepekatan logam berat pada tahap ppb. Bagi proses osmosis ke hadapan, keputusan menunjukkan bahawa MSL3 membran membolehkan pengurangan zat terlarut berbalik setelah digabungkan dengan UCR. Fluk zat terlarut berbalik paling rendah yang diperolehi adalah $0.008 \text{ kg m}^{-2} \text{ h}^{-1}$. Walaubagaimanapun UCR tidak stabil dengan kehadiran Ni(II), yang mana membentuk ion kompleks kemudiannya. Penjerapan ion Ni(II) telah menyebabkan pengumpulan zarah zeolit dan mengakibatkan kecacatan pada membran. Kajian ini telah berjaya membangunkan zeolit membran sebagai alternatif kepada penyingkiran ion logam berat dari sampel air.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
ED	-	Electrodialysis
FESEM	-	Field emission scanning electron microscopy
FO	-	Forward osmosis
FTIR	-	Fourier transform infrared spectroscopy
MEUF	-	Micellar enhanced ultrafiltration
NF	-	Nanofiltration
NMP	-	N-methyl-2-pyrrolidone
PEG	-	Polyethyleneglycol
PEI	-	Polyethylenimine
PES	-	Polyethersulfone
PEUF	-	Polymer enhanced ultrafiltration
ppb	-	part per billion
Q	-	Adsorption capacity
RO	-	Reverse osmosis
SEM	-	Scanning electron microscopy
TMA	-	Tetramethylammoium
UCR	-	UV-curable resin
UF	-	Ultrafiltration
XRD	-	X-ray diffraction

LIST OF SYMBOLS

%	-	percentage
°C	-	degree celcius
µm	-	micrometer
Å	-	Angstrom
C ₀	-	initial concentration
Cm	-	centimeter
g	-	gram
h	-	hour
kDa	-	kilo Dalton
L	-	litre
mg	-	miligram
mg/L	-	milligram per litre
min	-	minute
mmol/g	-	mili mol per gram
pH	-	pH value
rpm	-	rotary per minute
t	-	time
V	-	volume

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CHAPTER 1

INTRODUCTION

1.1 Background Information

Heavy metals are generally defined as elements that have the atomic weight in the range of 63.5 to 200.6 and a specific gravity more than 5.0 (1). Heavy metals are found naturally in the earth and are not harmful to our health and environment because they only appeared in very small amount. However, rapid growth of the world population, industrialization, unplanned urbanization, agricultural activities as well as excessive use of chemicals increases the level of heavy metals in aquatic system. In recent years, water contamination become a serious environmental issue and had caused international concern (2). Shazili et al (3) has reviewed the status of heavy metal pollution in the Malaysian aquatic environment and based on their report, the major source of metals in the aquatic environment of Malaysia came from metal finishing process such as electroplating, etching, and preparation of metal components for various industries. They have concluded that the sediments of coastal environments received from areas of high human activities such as Juru and the Johor Strait were contaminated with lead and zinc. The water of some rivers near major cities and township were also contaminated.

Heavy metal ions are reported as priority pollutants, due to their mobility in natural ecosystems and due to their toxicity. The heavy metal ions are stable and persistent environmental contaminants since they cannot be degraded and destroyed. The presence of heavy metals such as copper, zinc, cadmium, lead, mercury, iron, and nickel in aquatic environment are highly risk to human health and ecosystem. These metals are considered toxic for human and can cause fatal diseases such as cancer, even in trace amount (4,5). As a result, Malaysian-Environmental Quality Act 1974 has set the discharge limit of standard B of these metals is below 1.0 mg/L

(6). Therefore, the elimination of heavy metals from water is important to protect public health.

Since heavy metals are highly toxic to human and ecosystem, therefore, there are increasing concerns regarding the removal of these compounds. Various methods have been widely studied for the removal of heavy metals from water in recent, includes chemical precipitation (7), coagulation (8), sedimentation (9), filtration (10), adsorption and disinfection (11,12). Among these techniques, adsorption method is the most preferable to be used due to its simplicity and effectiveness. However, there are some drawbacks and limitations with those methods as it only transfers the non-biodegradable matter into sludge which resulted in the formation of secondary pollution. Moreover, a large operation area for a complex process with more complex system is required. Typically, an adsorption process to eliminate heavy metals is carried out in a fixed bed column (13). However, this process is associated with high intra-particle diffusion resistance, low flow rate, high pressure drop, channeling and wall-effect (14). Thus, researchers have gathered an interest of using membrane technology to overcome these problems.

Membrane technology has gained an attention in the wastewater treatment during the past few decades due to its advantages such as cost effective, no phase change, easy to fabricate and high removal efficiency. Membrane technology works without the chemicals addition, low energy usage and easy and well-arranged process conduction. In membrane technology, the membrane act as a very specific filter where it catches the suspended solids and other substances and let the water flow through it (15). Polymeric and ceramic are two common materials that used to construct the membranes. Compared to polymeric membrane, ceramic membrane is usually used in the treatment of industrial wastewater since it has a good filtration performance (16).

Recently, there is a renewed interest in combining membrane technology with adsorption process for heavy metal removal. This hybrid system is not only producing pure water on the permeate side but heavy metal can be simultaneously captured thus minimizing the heavy metal concentration in retentate stream. Various

works have reported that an adsorbent dispersed in polymeric membrane enables a reduction in heavy metal concentrations both in permeate and retentate streams. In this study, ceramic hollow fibre membrane incorporated with adsorbent was developed and being used as an alternative to polymeric membrane since it not only remove heavy metal from industrial wastewater but retain most particulate matters prior to discharge into environment.

Various type of adsorbents are available for the removal of heavy metals, i.e. limestone (6), chitosan (17), carbon nanotube (18) and zeolite (19). Of all these adsorbents, zeolites also have been extensively used not only in heavy metal removals but also in the separation and purification applications. An open-framework of aluminosilicates materials named zeolite has been proposed by some authors as an efficient and cost-effective resource to treat heavy metal contaminated effluents. Zeolites can be categorized into four different sizes according to their pore opening windows namely, small-pore (≤ 8 -ring), medium-pore (10-ring), large-pore (12-ring), and extra-large-pore (>12 -ring). Their unique physical characteristic and outstanding chemical properties have made them widely used as catalysts, detergents, adsorbents, and ion-exchangers in many chemical processes (20–22). In these applications, the zeolites are usually used in powder and pellet forms. Recently, much interest has been paid to membrane of the zeolites, because their unique structures and physicochemically properties of zeolites make the zeolite membranes attractive for a wide variety of practical applications including gas and vapour separations, membrane reactors, chemically selective sensors and optoelectronic devices (23).

Zeolite membranes can be prepared either self-supported or supported on a porous support. Composite polymer-zeolite membranes have been and are still largely studied but supported zeolites were by far the most frequently studied and detailed in the current study. The advantages of using porous support include a relatively high strength of resulting material, the ease of handling of the membrane, and the possibility to obtain a thin selective layer of the zeolite (24). Various types of supports have been used for the synthesis of supported zeolite membrane. When selecting the support, its thermal and chemical resistances to the synthesis media, to the thermal treatment or to the application conditions, as well as the difference in

thermal coefficients between the support and the zeolite layer, are important parameters which have to be considered. Carbon and glass support are being used in literature but they have poor resistance to either oxygen or basic media is restrictive. Thus $\alpha\text{-Al}_2\text{O}_3$ is by far the most largely studied, due to its chemically inert characteristic (25).

At present, there are three variant methods involved in the preparation of supported zeolite membrane namely, *in-situ* direct crystallization, dry or wet-gel conversion method and the seeding and secondary growth method. Zeolite membranes are commonly prepared by *in-situ* hydrothermal synthesis on porous supports. The essence of the *in-situ* direct crystallization approach is to bring the surface of a porous support in contact with a zeolite precursor solution and keep the system under controlled conditions in order to nucleate and grow a thin continuous zeolite film on the surface and/or in porous support. The second type of method includes the so-called dry gel and wet gel conversions. It involves the deposition of an amorphous aluminosilicate gel on the support and its subsequent crystallization under vapours of triethylamine, ethylenediamine and water. This type of synthesis method allows avoiding homogenous nucleation and has advantages in minimizing waste generation and reduced chemical consumption that are desirable for large scale production of zeolite membranes. The seeding and secondary growth method is classically used in zeolite synthesis. By decoupling the nucleation step from the crystal growth, the seeds can grow in low concentrated solutions, while secondary nucleation is avoided. In the case of membrane synthesis, it involves the growth of a layer of seeds crystals previously nucleated and deposited on the support. This method is now considered as one of the most attractive and flexible approaches for orienting the formation of consolidated thin membranes. Indeed, the seeding strategy brought in improved flexibility for crystal growth and greater control of films microstructure as well as higher reproducibility and scalability (25). Regarding to these advantages, *in-situ* direct crystallization and secondary growth method being selected to be use throughout the current study.

1.2 Problem Statement

Normally, the adsorbents are being used in a fixed bed column which considered as very easy and cheap method. The adsorption happened rapidly and effectively at early stage of the process, but the adsorbent becomes gradually saturated towards the end of the process. Therefore, the adsorption efficiency of the adsorbent reduces as well leads to high intra-particle diffusion resistance, low flow rate, high pressure drop, channeling and wall-effect. These limitations should be avoided as they would affect the removal efficiency of the pollutant. Thus many researchers have recently focused towards the development of adsorptive membranes where the advantages of membrane technology and adsorption process are being combined. In this work, zeolite adsorbent was synthesized and grown hydrothermally on the outer layer of ceramic support which then being used as membrane for the filtration and adsorption of selected heavy metal from aqueous solution. Despite the advantage of adsorptive membrane, there still a challenge to fabricate the zeolite membranes with stable coating of zeolite on the support materials. The zeolite adsorbent is too hydrophilic thus made the coating so weak and could be easily washed by liquid stream. Therefore, the detachment of zeolite layer from support might happen and will result in an ineffective use of these adsorbent coatings. In view of this, the the potential of covering a zeolite coating by a hydrophobic resin layer will be discovered in this research. The addition of hydrophobic resin layer preserved the zeolite coating and improved the stability of the membrane.

1.3 Objectives of the Study

The objectives of the study are to:

- 1 To determine the effect of two synthesis methods, *in-situ* crystallization and secondary growth method, on zeolite membrane characteristic and performance.
2. To study the effect of synthesis parameters on the characteristic and the performance of zeolite membrane in terms of forward osmosis .

3. To study the function of hydrophobic coating on zeolite membrane and to evaluate the simultaneous performance of composite zeolite membrane in water permeation and heavy metal removal by forward osmosis

1.4 Scope of the Study

Steps involved in this study are listed below:

Scope objective 1:

- a) Preparation of alumina substrate using phase inversion and sintering technique.
- b) Deposition of zeolite on the outer surface of alumina hollow fibre using *in-situ* crystallization and secondary growth method via hydrothermal technique.
- c) Synthesis of zeolite membrane at three different crystallization time (24 h, 48 h and 120 h) for both *in-situ* crystallization and secondary growth method at 100 °C.
- d) Characterize all samples using Field Emission Scanning Electron Microscopy (FESEM) and X-ray Diffraction (XRD).
- e) Evaluate the performance of all samples in term of water permeability, solute rejection.
- f) Apply the zeolite membrane with the best performance in removing nickel

Scope objective 2:

- a) Screening test of zeolite membrane prepared at wide range of zeolite concentration (0.05 M up to 1.0 M) and the select the best three concentration to proceed in the performance test.
- b) Prepare the zeolite membrane at various crystallization time at 16 h, 20 h and 24 h by using the best concentration condition.
- c) Double the zeolite layer onto alumina support using the best concentration condition and optimum crystallization time.
- d) Characterize the zeolite membrane using field emission electron microscopy (FESEM), X-ray diffraction (XRD) and Brunauer-Emmet-Teller (BET).
- e) Evaluate the performance of prepared membrane in terms of water flux and reverse solute flux using forward osmosis test: feed solution (deionized water) and draw solution (100,000 mg L⁻¹ NaCl).
- f) Application of the membrane with the best performance for heavy metal removal using forward osmosis process: feed solution (100 mg L⁻¹ nickel solution) and draw solution (100,000 mg L⁻¹ NaCl), and evaluate using Atomic Absorption Spectroscopy (AAS).

Scope objective 3:

- a) Coat the zeolite hollow fibre membrane with hydrophobic UV-curable resin (DEFENSA OP-4003) using dip-coating with UV-curing process to activate it.
- b) Characterize the zeolite membrane using Fourier transform infrared spectroscopy (FTIR) with ATR mode, field emission electron microscopy

(FESEM), X-ray diffraction (XRD), Brunauer-Emmet-Teller (BET) atomic force spectroscopy (AFM) and water contact angle.

- c) Evaluate the performance of prepared membrane in terms of water flux and reverse solute flux using forward osmosis test: feed solution (deionized water) and draw solution ($100,000 \text{ mg L}^{-1} \text{ NaCl}$).
- d) Application of the membrane with the best performance for heavy metal removal using forward osmosis process: feed solution (100 mg L^{-1} nickel solution) and draw solution ($100,000 \text{ mg L}^{-1} \text{ NaCl}$), and evaluate using Atomic Absorption Spectroscopy (AAS).

1.5 Significance of Study

An adsorptive membrane with zeolite as adsorbent material and alumina hollow fibre membrane as support was prepared in this study. The emerging membrane adsorption can be a powerful and attractive tool for the removal of hazardous materials like heavy metal ions, from water resources. The use ceramic material (alumina) as the support make the membrane have the stability of characteristic during operation, improved mechanical strength and structural hardness, thus made it long-lasting based on all the advantages they have. Zeolite was chosen as adsorbent material due to the larger pores with smaller crystallites, which helps in high ion exchange and adsorption molecular sieving capacity. The hydrothermal method is used for the preparation and deposition of zeolite onto alumina support in order to eliminate the effect of using organic template method. Membrane incorporated with inorganic adsorbents can be a better option as it not only removes heavy metal from water/wastewater but retain most particulate matters prior to discharge into environment. The addition of polymer resin as coating material has improved the stability of zeolite membranes.

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APPENDIX A

LIST OF PUBLICATIONS

1. **Muhamad, N.**, Abdullah, N., Rahman, A.M., Abas, K.H., Aziz, A.A., Othman, M. H. D., Jaafar, J and Ismail, A.F. (2018). Removal of nickel from aqueous solution using supported zeolite-Y hollow fiber membranes. *Environ. Sci. Pollut. Research*. 25, 19054-19064. **(Q2, IF: 2.914)**
2. **Muhamad, N.**, Makhtar, S.N.F.M., Abdullah, N., Pauzi, M.Z.M., Mahpoz, N. M., Othman, M. H. D., Jaafar, J., Abas, K.H., Fadil, N. A and Rahman, A.M. Composite zeolite hollow fiber membrane for the removal of nickel using forward osmosis. *J. Water. Process. Eng.*, 101806. **(Q1, IF: 3.173)**
<https://doi.org/10.1016/j.jwpe.2020.101806>
3. Makhtar, S.N.F.M., **Muhamad, N.**, Rahman, A.M, Abas, K. H., Aziz, A.A., Sokri, M.N.M., Othman, M.H.D. and Jaafar, J. (2020). Zeolite-A deposited on glass hollow fiber for forward osmosis application. *J. Water. Process. Eng.* 33, 100991. **(Q1, IF: 3.173)**