ADSORPTIVE NATURAL ZEOLITE CERAMIC MEMBRANE FOR AMMONIA REMOVAL IN WASTEWATER

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DEDICATION

This thesis is specially dedicated to my father, Mr. Adam Salikin and my mother, Ms. Elon Mohd Said,

For their advices, their patience, their prays and their faith.
Thank you for always understood.

For my family,
Thank you for encouraging me to fly towards my dreams.

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ABSTRACT

Adsorption process is known as a promising way for ammonia elimination. Owing to the fact that natural zeolite (clinoptilolite) has a superior property of ion-exchange and adsorption, the removal of ammonia by natural zeolite is a strategic approach. However, the conventional approach of powder suspension adsorption might be ineffective due to some drawbacks such as the requirement of secondary treatment after the adsorption has taken place as well as the loss of adsorbent during the filtration and regeneration processes. The use of membrane technology can overcome this difficulty by combining adsorption and filtration in a single step. Therefore, this study aims to develop hybrid adsorptive natural zeolite based hollow fibre ceramic membrane (HFCM) via phase-inversion and sintering techniques. The fabrication parameters namely ceramic loading, air-gap distance, bore fluid flowrate, sintering temperature and natural zeolite clinoptilolite sieved particle size were studied. The properties of the prepared ceramic membranes were characterized in terms of morphologies, bending strengths, water permeabilities and porosities. The performance of the membrane for ammonia removal was studied using the synthetic ammonia wastewater in a crossflow system. The factors affected the adsorption performance specifically the membrane sintering temperature, natural zeolite clinoptilolite particle size, pH of the ammonia feed solution, ammonia initial feed concentration and HFCM dosage were examined in this study. The regeneration process of the used adsorptive ceramic membrane was investigated for the reusability study. Clinoptilolite has shown great potential as a good adsorptive membrane that targeting the uptake of contaminant cations in water treatment process. The results have also shown that the fabricated HFCM was successfully produced when spun at 45 wt.% ceramic loading, 5 cm of air-gap distance and 15 mL/min of bore fluid flowrate, sintered at 1050 ˚C and best fabricated using 36 μm sieved particle size. The produced HFCM exhibited desired morphologies with good bending strength and water permeation. The adsorptive HFCM demonstrated ammonia rejection of more than 90%. The optimization study has shown that the optimum condition of the adsorptive HFCM was found to be at pH 7.04, 75.00 mg/L and 0.35 g of feed pH, feed concentration and HFCM dosage, respectively. The optimum water permeability and ammonia removal were found to be of 281.9 L/m2·h and 94.14 %, respectively. The confirmatory test has revealed that the optimum performance was acceptable with average error of 1.64% and 1.85% for water permeability and ammonia removal, respectively.

ABSTRAK

Penjerapan merupakan satu cara yang berkesan bagi penyingkiran ammonia. Zeolit semulajadi (klinoptilolit) yang mempunyai ciri pertukaran ion dan penjerapan yang hebat telah menjadikannya satu bahan strategik bagi proses penyingkiran ammonia. Namun, kaedah tradisional iaitu penjerapan menggunakan serbuk ampaian mungkin tidak efektif dan mempunyai beberapa kelemahan antaranya keperluan rawatan sekunder setelah penjerapan berlaku serta kehilangan bahan penjerap semasa proses turasan dan penjanaan semula. Teknologi membran dapat mengatasi masalah ini dengan menggabungkan proses penjerapan dan turasan dalam satu langkah. Oleh itu, kajian ini bertujuan membangunkan membran seramik gentian geronggang (HFCM) penjerap berasaskan zeolit semulajadi melalui kaedah penyongsangan fasa dan sinteran. Parameter pembuatan seperti muatan seramik, jarak ruang udara, kadar alir bendalir gerek, suhu sinteran dan saiz partikel zeolit semulajadi klinoptilolit telah dikaji. Pencirian membran seramik telah dilakukan dari segi morfologi, kekuatan lengkungan, kebolehtelapan air dan keliangan. Keberkesanan membran ke atas penyingkiran ammonia telah dikaji menggunakan air sisa ammonia sintetik melalui sistem alir-lintas. Faktor-faktor yang menjejaskan keberkesanan penjerapan terutamanya suhu sinteran membran, saiz partikel zeolit semulajadi klinoptilolit, pH larutan suapan ammonia, kepekatan awal suapan ammonia dan dos HFCM juga telah dikaji. Proses penjanaan semula membran seramik penjerap yang telah digunakan telah dikaji untuk kajian gunaan semula. Zeolit semulajadi klinoptilolit menunjukkan potensi yang besar sebagai membran penjerap yang baik yang memfokuskan pengambilan kation bahan pencemar di dalam proses rawatan air. Hasil kajian pula menunjukkan HFCM telah berjaya dihasilkan pada muatan seramik 45% berat, 5 cm jarak ruang udara dan 15 mL/min kadar aliran bendalir gerek, disinter pada 1050 ˚C dan dihasilkan dengan partikel bersaiz 36 μm. Membran yang dihasilkan menunjukkan morfologi yang dikehendaki dengan kekuatan lengkungan serta kebolehtelapan air yang sangat baik. HFCM penjerap menunjukkan kadar penyingkiran ammonia melebihi 90%. Kajian pengoptimuman pula menunjukkan keadaan optimum bagi HFCM penjerap ini ialah pada pH 7.04, 75.00 mg/L kepekatan suapan ammonia dan 0.35 g dos HFCM. Keputusan optimum bagi kebolehtelapan air dan penyingkiran ammonia ialah masing-masing 281.9 L/m2·j dan 94.14%. Ujian pengesahan pula menunjukkan keberkesanan optimum adalah diterima dengan ralat purata 1.64% dan 1.85% masing-masing bagi kebolehtelapan air dan penyingkiran ammonia.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
BOD	-	Biological oxygen demand
COD	-	Chemical oxygen demand
FTIR	-	Fourier transform infrared spectroscopy
HFCM	-	Hollow fibre ceramic membrane
MIP	-	Mercury intrusion porosimetry
NMP	-	N-methyl-2-pyrrolidone
PESf	-	Polyethersulfone
SEM	-	Scanning electron microscopy
TEM	-	Transmission electron microscopy
WHO	-	World Health Organization
XRD	-	X-ray diffraction
XRF	-	X-ray fluorescence

LIST OF SYMBOLS

A	-	Effective membrane area (m^2)
R	-	Rejection (%)
J	-	Permeate flux ($L/m^2 \cdot h$)
σ_F	-	Bending strength (MPa)
R_a	-	Surface roughness (μm)
t	-	Time (min)
C_i	-	Initial concentration (mg/L)
C_f	-	Final concentration (mg/L)
V	-	Volume (L)

CHAPTER 1

INTRODUCTION

1.1 Introduction

Ammonia is regarded as one of the major undesirable contaminants that introduced into receiving streams by industrial, agricultural and domestic wastewater discharges (Urbini *et al.*, 2015). Ammonia can exist in two forms in the dissolved state. The highly toxic is in the form of ammonia (NH_3) while the other less harmful form is ammonium ion (NH_4^+). Additionally, the composition of these elements is highly depending on the temperature and pH of the surrounding. Toxic ammonia can be detrimental to the aquatic life. It is reported that concentrations of 0.01 mg/L might have negative effects on fish whereas 0.1 mg/L can be fatal to other species (Mandowara and Bhattacharya, 2011). For that reason, it is vital for the industries to eliminate the dissolved gases and to reutilize the water for further use. Moreover, the environmental regulations and laws governing safe discharge levels of ammonia are becoming increasingly stringent.

In the past few decades, many great efforts have been committed to the removal of ammonia nitrogen (NH_3–N) from wastewater. Various methods of ammonia nitrogen removal from waters have been established, but the purpose of worldwide assessment has been to determine the new potentials for NH_3–N removal in the waters. The established techniques that have been applied for the elimination of dissolved ammonia including break-point chlorination, air stripping, selective ion exchange, chemical precipitation, electrochemical conversion, biological treatment, and so on (Capodaglio *et al.*, 2015; Khuntia *et al.*, 2013; Sarioglu, 2005; Welander *et al.*, 1998). One of the conventional methods in ammonia removal is biological treatment. However, the ammonia concentration after this treatment is still high due to the high concentration of ammonia that eventually leads to the low ratio of carbon/nitrogen (C/N). Thus, this process is only appropriate for the elimination of relatively low ammonia concentration due to the compulsion of proper C/N ratio (Crab *et al.*, 2009; Yang *et al.*, 1999). As a result, biological processes are usually difficult to meet the discharge standards (Aspé *et al.*, 2001; Qian Yi *et al.*, 1994). In addition, the efficiency of this method is limited by the lengthy bioconversion and unfavourable environmental factors.

There are numbers of limitation possessed by other ammonia removal processes that currently available. Apart of biological treatment, it is also difficult to treat the ammonia contained wastewater via typical biochemical practice when its NH_3–N concentration is greater than 300 mg/L. In these cases, the reduction of the ammonia concentration is a must prior to biochemical conduct. Ion-exchange and chlorination procedures have also infrequently been employed to the high levels of ammonia containing industrial wastewaters. Nevertheless, the drawbacks of these processes are expensive and challenging maintenance requirement due to the chemicals used in the oxidation and regeneration phases (Belhateche, 1995; Huang *et al.*, 2014; Jorgensen and Weatherley, 2003). Due to the inefficacy of most of the available treatment processes, therefore, the emergence development of the low cost and yet effective ammonia removal process is becoming vital in these coming days. Several developments particularly in the membrane processes may resolve this problematic.

Adsorption is regard as well-established process for water and wastewater treatment, particularly for the elimination of heavy metal and dyes pollutants. The efficacy of the adsorbents used in adsorption activities principally hinge on their morphology and chemistry. In recent years, the advance in adsorptive hollow fibre membrane offers an appealing option for the eradication of numerous volatile pollutants including ammonia (Ozturk *et al.*, 2003; Yang *et al.*, 2014). The preference adsorption process in the form of membrane is due to straightforwardness separation after process and prerequisite of no post treatment process of the filtration. The hollow fibres used are commonly microporous and hydrophilic. Hence, the volatile compound will evaporate from the feed, diffuse through the gas-filled membrane pores, and then be adsorbed by the adsorptive membrane.

Adsorptive hollow fibre membranes have shown to be beneficial for eradicating even low-concentration solutes from wastewater thus, they can be verified to be a smart substitute for the existing work. Adsorptive hollow fibre membrane offers many benefits over traditional suspension adsorption processes. Adsorptive membrane provides a large and stable interfacial area. The feed fluid flowing across the hollow fibre thus, producing the mass transfer between the fluid and the membrane. Additionally, the hydrophilic nature of the microporous membrane offers the adsorption site and thus, the water permeation (Suen, 2015). On top of that, the adsorption process occurs at the pore opening or exit as well as inside the pore.

1.2 Problem Statement

The utilisation of natural zeolite as the superior ion exchange for the removal of ammonium ions through adsorption process and the recovery of the adsorbed ammonium via desorption process have been widely studied for the past decades. The ammonia adsorption onto the natural zeolite is attributed by the superior ion-exchange properties of the zeolite. The ammonia exchange with the Australian clinoptilolite possesses the kinetic and equilibrium characteristics under binary and multicomponent conditions (Cooney *et al.*, 1999). The zeolite highest selectivity for ammonium ions over the other cations present in the water has made this material as a good tool for the ammonia treatment process. The major constituent of the natural zeolite namely aluminosilicate has eventually aided the ion exchange of this compound to that of ammonia presented in its surrounding (Haralambous *et al.*, 1992)

On top of that, the usage of the natural adsorption which normally in the powder suspension form has some drawbacks namely the requirement of the secondary filtration process upon the adsorption process and the loss of the adsorbent during the filtration process have limited the utilization of this approach in real applications. Thus, the membrane technology was attempted by embedding this adsorbent into the membrane matrix to form the adsorptive membrane (Mukherjee and De, 2016).

iii. Fabricating adsorptive natural zeolite based hollow fibre ceramic membranes (HFCM) via phase inversion technique at various fabrication parameters namely air – gap distance ranging from 1 – 25 cm and bore fluid flowrate in the range of 5 – 20 mL/min (to accomplish objective 1).

iv. Performing the sintering technique at different temperatures ranging from 900 – 1150 °C in order to produce the best zeolite based HFCMs (to accomplish objective 1).

v. Characterizing the morphological and water permeation analyses in order to compare the properties and performances of the fabricated HFCMs via scanning electron microscopy (SEM) and water permeation test (to accomplish objective 1).

vi. Preparing and studying the effect of the natural zeolite sieved particle size namely 36 μm, 50 μm and 75 μm, onto the physicochemical properties of the fabricated adsorptive hollow fibre ceramic membrane (to accomplish objective 1)

vii. Studying the adsorption performance of the adsorptive membrane onto the ammonia removal from wastewater (to accomplish objective 2)

viii. Evaluating the effect of factors that determine the performance of membrane adsorptivity via one-factor-at-a-time (OFAT) approach including membrane sintering temperature, natural zeolite clinoptilolite sieved particle size, ammonia feed solution pH (pH 2 – pH 11), initial feed solution concentration (50 – 500 mg/L), HFCM dosage (0.1 – 0.5 g) as well as investigating the regeneration process of the used HFCMs (to accomplish objective 2)

ix. Studying the optimum parameters and conditions of adsorptive membrane system towards the optimization of the ammonia removal from wastewater namely pH of feed solution (pH 5 – pH 10), initial concentration of the feed solution (50 – 100 mg/L) and the HFCM dosage (0.2 – 0.5 g) (to accomplish objective 3).

1.5 Significance of the Study

This study consists of the development of natural zeolite based adsorptive HFCM via phase inversion and sintering techniques which consequently to be used in adsorptive membrane system for the ammonia removal in wastewater treatment. The usage of ceramic instead of polymer in the fabrication of the hollow fibre leads to the improvement in the membrane properties to which could withstand the harsh environments namely high alkalinity and acidity, high temperature as well as pressure. This could prolong the lifetime of the HFCM and its reusability and eventually leads to the reduction in the production cost. The choice of natural zeolite as the ceramic material which has a lower melting point as compared to the commercial ceramics as well as the needs of lower sintering temperature will be resulted in reducing the cost of production. Moreover, the usage of natural zeolite in the form of HFCM instead of powder suspension in the removal of ammonia is such a novel and innovative approach. The hybrid system with immobilized zeolite membrane will be the most promising set – up for the wastewater treatment application because all processes including the degradation of the pollutant, the filtration of the effluent as well as the treatment of the ammonia into a useful product can be done at once in a single chamber.

1.6 Organization of the Thesis

This thesis is divided into seven chapters including the preparation of the natural zeolite clinoptilolite for the fabrication of the adsorptive hollow fibre ceramic membrane, the evaluation of the fabrication process and the factors affecting the process, the examination of the membrane performance for the ammonia removal as well as the optimization of the adsorption process of the adsorptive hollow fibre ceramic membrane via RSM approach.

Chapter 1 describes the brief introduction on the ammonia contamination in water and the current technology of the treatment devoted to this matter. Then, the details of the problem statement, objectives, scopes and the significance of the study have thoroughly stated.

Chapter 2 denotes the literature reviews of the topics of interest in this thesis. In this chapter, the details information of the conventional and to date technologies of the ammonia removal treatment are comprehensively discussed. The toxicity and adverse effects of the ammonia and the conventional removal approaches are well addressed. The intervention of the membrane technology and the techniques of producing the membrane are also conferred. This chapter also deliberates the advantageous and limitations of the current adsorptive membrane technology in treating the ammonia in wastewater.

Chapter 3 emphases on the materials, techniques and working principles, characterization approaches and the adsorptive membrane setup for the ammonia removal in wastewater.

Chapter 4 discusses the characterization and the properties of the natural zeolite clinoptilolite as an alternative material to be developed as the adsorptive hollow fibre ceramic membrane for the ammonia removal in water treatment. The characterizations are included the physicochemical properties evaluation namely microtopographical behaviour, crystallinity properties, chemical composition and zeta potential of the natural zeolite clinoptilolite powder. This chapter also describes in detail the fabrication of the adsorptive hollow fibre ceramic membrane from natural zeolite clinoptilolite via phase inversion and sintering techniques. The effects of the extrusion parameters onto the physicochemical properties of the fabricated membrane ware carefully characterized and explained. The effect of the natural zeolite clinoptilolite sieved particle size onto the physicochemical properties of the fabricated adsorptive membrane was also studied and discussed. In addition,

In **Chapter 5,** the potential of the ammonia adsorption by the adsorptive natural zeolite clinoptilolite HFCM was successfully performed using crossflow filtration system. The factors that influenced the adsorption process of ammonia via adsorptive HFCM were studied in detailed. The performance effects of membrane sintering temperature, natural zeolite clinoptilolite sieved particle size, ammonia feed solution pH, ammonia initial solution concentration, HFCM dosage as well as effect of HFCM regeneration process were studied by the approach of one-factor-at-a-time (OFAT). The characterizations of some factors were also discussed in this chapter.

On the other hand, **Chapter 6** discusses the optimization study of the adsorptive HFCM performance for 3 significant factors namely pH of the feed solution, feed solution concentration and the HFCM dosage via RSM approach. The adsorptive performances of the membrane were carried out using the best membrane of 36 μm natural zeolite clinoptilolite sieved particle size and sintered at 1050 ˚C of sintering temperature. The desirability test was also performed to verify the adequacy of the developed model.

Finally, **Chapter 7** stated the general conclusion for each chapter consisted in the thesis. The suggestion and recommendation for the future work have also discussed in this chapter.

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LIST OF PUBLICATIONS

International Peer-Reviewed Journals

1. **Mohd Ridhwan Adam**; Takeshi Matsuura; Mohd Hafiz Dzarfan Othman*; Mohd Hafiz Puteh; Mohamad Arif Budiman Pauzan; A. F Ismail; Azeman Mustafa; Mukhlis A Rahman; Juhana Jaafar; Mohd Sohaimi Abdullah, Feasibility study of the hybrid adsorptive hollow fibre ceramic membrane (HFCM) derived from natural zeolite for the removal of ammonia in wastewater, Process Safety and Environmental Protection, 122 (2019) 378 – 385 (Impact factor 2018 = 4.384, Q1).

2. **Mohd Ridhwan Adam**; Mohd Hafiz Dzarfan Othman*; Rozaimi Abu Samah; Mohd Hafiz Puteh; A. F. Ismail; Azeman Mustafa; Mukhlis A. Rahman; Juhana Jaafar, Current trends and future prospects of ammonia removal in wastewater: A comprehensive review on adsorptive membrane development, Separation and Purification Technology, 213 (2019) 114 – 132 (Impact factor 2018 = 5.107, Q1).

3. **Mohd Ridhwan Adam**; Norliyana Mohd Salleh; Mohd Hafiz Dzarfan Othman*; Takeshi Matsuura; Mohd Hafizi Ali; Mohd Hafiz Puteh; A. F Ismail; Mukhlis A Rahman; Juhana Jaafar, The adsorptive removal of chromium (VI) in aqueous solution by novel natural zeolite based hollow fibre ceramic membrane, Journal of Environmental Management, 224 (2018) 252 - 262 (Impact factor 2018 = 4.865, Q1).

4. **Mohd Ridhwan Adam**, Mohd Hafiz Dzarfan Othman*, Siti Khadijah Hubadillah, Mohd Hafiz Puteh, Zawati Harun, A.F. Ismail, Azeman Mustafa, Mukhlis A. Rahman, Juhana Jaafar, Evaluating the Sintering Temperature Control Towards the Adsorptivity of Ammonia onto the Natural Zeolite Based Hollow Fibre Ceramic Membrane International Journal of Engineering Transactions B Applications, 31:8 (2018) 1398 - 1405 (Scopus Citation-Indexed Journal).

5. **Adam, M.R**., Othman, M.H.D.*, Puteh, M.H., Pauzan, M.A.B., Rahman, M.A., Jaafar, J., A fabrication of a low-cost zeolite based ceramic membrane via phase inversion and sintering technique, Malaysian Journal of Analytical Sciences, 21:2 (2017) 391-401 (Scopus Citation-Indexed Journal).

6. Siti Munira Jamil; Mohd Hafiz Dzarfan Othman*, Mohd Hilmi Mohamed; **Mohd Ridhwan Adam**; Mukhlis A. Rahman; Juhana Jaafar; Ahmad Fauzi Ismail, A novel single-step fabrication anode/electrolyte/cathode triple-layer hollow fiber micro-tubular SOFC. International Journal of Hydrogen Energy. 43 (2018) 18509-18515 (Impact factor 2018 = 4.084, Q2).

7. Mohd Haiqal Abd Aziz; Mohd Hafiz Dzarfan Othman*; Nur Awanis Hashim; **Mohd Ridhwan Adam**; Azeman Mustafa, Fabrication and characterization of mullite ceramic hollow fiber membrane from natural occurring ball clay. Applied Clay Science. 177 (2019) 51-62 (Impact factor 2018 = 3.890. Q1)

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