DUAL-LAYER ZIRCONIA-KAOLIN HOLLOW FIBRE IN MEMBRANE CONTACTOR FOR AMMONIA RECOVERY

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DEDICATION

Dear Me,

Thank you for not giving up

To Mak, Abah, Siblings, Drs, Friends and You,

This is for you

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ABSTRACT

This study was initiated to develop of a green technology in ammonia recovery by using ceramic membrane derived from natural ceramic material in membrane contactor (MC) system. The main objective of this study is to develop a high performance and superhydrophobic dual-layer ceramic hollow fibre membranes for effective ammonia recovery from alternative natural material, Malaysian kaolin. However, due to high content of silica in kaolin which dissolves in high concentration of ammonia has hindered it to be used in MC system which requires high concentration of ammonia to work efficiently. Hence, a range of zirconia was added in this work to tackle the problem. Plus, the fabrication of dual layer configuration to the membrane by creating a protecting layer to the mixed zirconia-kaolin membrane enhanced the performance of the membrane in high concentration of ammonia. The dual-layer mixed zirconia-kaolin hollow fibre membrane (DLZK) was fabricated by using single step co-extrusion phase inversion/co-sintering technique where the mixed zirconia-kaolin was on the inner layer while zirconia was only on the outer layer. Upon investigation on the effect of different co-sintering temperatures, DLZK co-sintered at 1300°C showed the best characteristics in term of high mechanical strength (116 MPa) and possessed low dissolution rate in high alkaline solution. Finally, prior to be used in MC application system, the surface of the membranes was modified to be hydrophobic by grafting with fluoroalkylsilane agent. Then, the membranes were subjected to MC operating system for ammonia recovery using two different concentrations of synthetic ammonia (NH_4OH), contacted with sulphuric acid and compared in term of properties between the membranes. DLZK was discovered to yield the highest mass transfer coefficient which was 3.77 x 10^{-5} Kms^{-1} at 10M (pH 11-13) of NH_4OH and was able to recover almost 80%. Then, the DLZK membrane was subjected to recover ammonia in the treated palm oil mill effluent. The membrane showed similar performance using synthetic wastewater of NH_4OH as almost 80% of ammonia was successfully recovered with mass transfer coefficient of 3.50 x 10^{-5} Kms^{-1}. This indicates that the fabricated DLZK membrane can be applied in MC system for ammonia recovery as an alternative naturally based membrane which possesses superhydrophobic properties and is able to withstand high alkaline condition. The ammonia recovered was in the form of ammonium sulphate, ($(NH_4)_2SO_4$) salt, which can be used as alternative fertilizer.

ABSTRAK

Kajian ini dicetuskan untuk membangunkan teknologi hijau di dalam perolehan ammonia menggunakan membran seramik dihasilkan daripada bahan seramik semula jadi dalam sistem membran penyentuh (MC). Objektif utama kajian ini adalah untuk membangunkan seramik membran gentian berongga dwi-lapisan yang berprestasi tinggi dan bersifat hidrofobik yang hebat untuk perolehan ammonia yang berkesan berasaskan bahan seramik semula jadi sebagai alternatif iaitu kaolin daripada Malaysia. Walaubagaimanapun, disebabkan oleh kandungan silika yang tinggi di dalam kaolin yang larut dalam ammonia berkepekatan tinggi menghalang ia digunakan dalam sistem MC yang memerlukan ammonia berkepekatan tinggi untuk beroperasi secara berkesan. Oleh itu, satu julat zirkonia ditambah dalam kajian untuk menangani masalah ini. Tambah pula, fabrikasi konfigurasi dwi-lapisan pada membran dengan mencipta satu lapisan perlindungan pada membran campuran zirkonia-kaolin meningkatkan prestasi membran di dalam ammonia berkepekatan tinggi. Membran gentian berongga campuran zirkonia-kaolin dwi-lapisan difabrikasi dengan menggunakan teknik satu langkah sonsangan fasa ko-penyemperitan/ko-pensinteran yang mana campuran zirkonia-kaolin di lapisan dalam sementara zirkonia hanya dilapisan luar. Berdasarkan siasatan terhadap kesan perbezaan suhu ko-pensinteran, DLZK diko-sinteran pada suhu 1300°C menunjukkan ciri terbaik dari segi kekuatan mekanikal (116 MPa) yang tinggi dan mempunyai kadar pelarutan yang rendah di dalam larutan beralkali tinggi. Akhir sekali, sebelum digunakan pada sistem aplikasi MS, permukaan membran diubahsuai menjadi hidrofobik dengan cantuman agen floroalkilsilana. Kemudian membran digunakan pada sistem pengoperasian MC untuk perolehan ammonia menggunakan kepekatan sintetik ammonia (NH_4OH) berbeza, disentuh dengan asid sulfurik dan dibandingkan dari segi sifat-sifat antara membran. DLZK ditemui menghasilkan pekali pemindahan jisim tertinggi iaitu 3.77×10^{-5} Kms^{-1} pada 10M (pH 11-13) NH_4OH dan mampu memperoleh hampir 80%. Seterusnya, membran DLZK yang digunakan dalam perolehan ammonia daripada air sisa efluen kilang kelapa sawit, membran menunjukkan prestasi yang setara menggunakan air sisa sintetik, NH_4OH dimana hampir 80% ammonia berjaya diperoleh dengan pekali pemindahan jisim bersamaan 3.50×10^{-5} Kms^{-1}. Ini menunjukkan bahawa membran DLZK boleh digunakan pada sistem MC untuk perolehan ammonia sebagai membran alternatif semula jadi yang bersifat hidrofobik yang hebat serta mampu bertahan di dalam keadaan larutan berlalkali tinggi. Ammonia telah diperoleh dalam bentuk garam ammonium sulfat, ($(NH_4)_2SO_4$) yang boleh digunakan sebagai baja alternatif.

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LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscopy
APHA	-	American Public Health Association
COD	-	chemical oxygen demand
DLZK	-	dual layer mixed zirconia-kaolin hollow fibre membrane
EDX	-	energy dispersive x-ray
FA	-	free ammonia
FAS	-	fluoroalkylsilane agent
LEPw	-	liquid entry pressure
MC	-	membrane contactor
NH_3-N	-	ammoniacal nitrogen
POME	-	palm oil mill effluent
PP	-	polypropylene
PTFE	-	polytetrafuoroethylene
PVDF	-	polyvinylidenefuoride
RAMAN	-	RAMAN spectroscopy
SDG	-	sustainable development goal
SEM	-	scanning emission microscopy
SLZK	-	single layer mixed zirconia-kaolin hollow fibre membrane
TN	-	total nitrogen
TOC	-	total organic carbon
TSS	-	total suspended solid
VCs	-	volatile species
XRD	-	X-ray diffraction
XRF	-	x-ray fluorescence
YSZ	-	yttria-stabilized zirconia
ZK	-	zirconia-kaolin

LIST OF SYMBOLS

r	-	Ramping rate
F	-	Maximum load
σF	-	Mechanical strength
D, d	-	Diameter
L	-	Length
J	-	Water permeation
V	-	Volume
A	-	Area
t	-	Time
2θ	-	Diffraction angle
W	-	Weight
C	-	Concentration
ε	-	Porosity
K	-	Mass transfer coefficient
X	-	mean

CHAPTER 1

INTRODUCTION

1.0 Research background

Amidst a fast-growing world population and the improvement of living standards, human activities have been causing an imbalance to the cycle of nature by converting the inert nitrogen to more and more into several reactive forms in which at high concentrations will cause hazards to the environment (Smith, 2003). Unbeknownst, the ammoniacal nitrogen, NH_3-N (the combination of ammonium ions, NH_4^+, and ammonia, NH_3) is the main compound of the active nitrogen introduced into natural water by industrial, domestic, and agricultural wastewater and as such is the target of most nitrogen removal processes (Rongwong and Sairiam, 2020). Usually comes as a by-product of fertilisers used in plantation and the production of the fertilisers itself are drained into rivers (Sancho *et al*, 2017), ammonia can also originate from the anaerobic reaction from anaerobic digester (Tao *et al.*, 2017), decomposition of solid waste and agricultural waste (Abdullah *et al.*, 2020; Pauzan *et al.*, 2020) and by-product of radioactive waste (Liu and Wang, 2016).

Researchers as well as industries are trying to use different technologies to eliminate ammonia. This is because the accumulation of such pollutants will cause oxygen consumption due to nitrification, so in addition to toxicity, it will also harm aquatic organisms such as fish (Hassan *et al.*, 2019). Membrane filtration technology, particularly, membrane contactor system was one of the membrane system used to treat ammonia in wastewater (Norddahl *et al.*, 2006; Tan *et al.*, 2006; Zhu *et al.*, 2005) has attracted more interest due to its capability for filtration to remove ammonia from wastewater while at the same time, recover the removed ammonia to be transformed into fertiliser (Darestani *et al.*, 2017). Previous studies as such from landfill leachate (Kurniawan, *et al.*, 2021) as well as domestic wastewater (Lee *et al*, 2021) have proven that apart from to be removed, ammonia from these wastewaters can be recovered to be used as not only for fertilizer but also as an alternative sources of ammonia production.

One of the emerging techniques for the removal and recovery of ammonia from wastewater is membrane contactor (MC) system which is a technology that uses hydrophobic and microporous membranes to complete the mass transfer between two phases without dispersion. Compared to conventional contactor methods (such as scrubbers), the use of membrane contactor has several advantages including operational flexibility, reduced capital costs and an easily predictable design (Lauterböck *et al*, 2013; Ansaloni *et al.*, 2019). Interestingly, the concept of MC technology is membrane typically in hollow fibre configuration, with assumption only gaseous species such as ammonia gas was allowed to be transferred through the hydrophobic porous wall of the membrane (feed) and an acid such as sulphuric acid is flowed counter-currently on the lumen side (permeate) of the membrane in order to react with the ammonia gas to create ammonium sulphate (Tan *et al.*, 2006). Notably, water is repelled by the hydrophobic membrane surface unless a pressure exceeding the breakthrough pressure is applied (Luis, 2018).

Normally, ammonia originated from anaerobic digester and landfill leachate are usually in high concentration which eventually might be in high alkaline condition and the pH is more than 10 (Li *et al*, 2021). Hence, if MC technology was intended to apply to treat this ammonia-rich wastewater, the membrane must be able to withstand these high concentration and pH of ammonia. The membrane also needs to be porous enough to allow ammonia to pass through the wall of the membrane and hence reduce the weakness of the membrane due to high alkaline condition of ammonia. Hydrophobic polymeric membranes, such as polyvinylidenefuoride (PVDF) (Tan *et al.*, 2006; Damtie *et al.*, 2020), polytetrafuoroethylene (PTFE) (Lin *et al.*, 2018; Ahn *et al.*, 2011) and polypropylene (PP) (Licon Bernal *et al.*, 2016; Zhang *et al.*, 2020), are commonly employed for MC because of their low surface energy and high hydrophobicity. However, polymers have disadvantages which are unable to act in harsh conditions such as high temperature and non-chemical resistance, which are crucial properties for membranes in MC. Ceramic membranes able to alleviate this problem, as they can withstand harsh conditions due to their excellent mechanical and chemical stability as well as thermal resistance.

Researchers have tremendously explored the application of ceramic membranes for wastewater treatment from different sources such as industries, households, and restaurants due to their excellent characteristics such as high mechanical strength, high chemical and thermal stability, easy maintenance, and highly resistance to membrane's fouling. Usually, alumina is the most common material for fabrication of ceramic membranes. Unfortunately, alumina-based ceramic membranes showed some drawbacks due to high sintering temperature of up to 1500°C apart from the high cost of the alumina powder as well as makes these ceramic membranes fabrication extremely expensive. In fact, when a high sintering temperature is used, the fabrication process will be prolonged. Nevertheless, these ceramic membranes possess hydrophilicity behaviour that inhibits their application in membrane contactor systems (Hubadillah *et al.*, 2019).

Recently, ceramic materials derived from natural occurring aluminosilicate minerals such as pozzolan (Achiou *et al*, 2017), kaolinite (Hubadillah *et al.*, 2017), bentonite (Bouazizi *et al.*, 2017), bauxite (Ismail *et al.*, 2020), ball clay (Abd Aziz *et al.*, 2019a), and industrial/agricultural wastes like fly ash (Jedidi *et al.*, 2009), aluminium dross (Aziz *et al.*, 2019b), hydroxyapatite (Hubadillah *et al.*, 2020), and rice husk ash (Hubadillah *et al.*, 2017) have emerged as alternative materials for ceramic membrane fabrication (Hubadillah *et al.*, 2020). Not only the cost of these materials is inexpensive, but they also possess similar characteristics to materials commonly used in fabricating ceramic membranes which are alumina (Al_2O_3) and silica (SiO_2) as membrane support. According to Hubadillah *et al.*, (2018), kaolin ($Al_2Si_2O_5(OH)_4$) or kaolinite is one of the promising alternative materials for ceramic membrane fabrication. It is made up of aluminosilicate (Al_2SiO_5), which has similar properties to commercial alumina and silica and has been used for various membrane applications (Hubadillah *et al.*, 2019; Bouzid Rekik *et al.*, 2017; Hubadillah *et al.*, 2017; Abdulhameed *et al.*, 2017; Kumar *et al.*, 2013; Harun *et al.*, 2014). Another advantages of kaolin that it is non-toxic, high plasticity, low refractory, environmental-friendly and naturally abundant available in many countries such as United States, China, Germany, India, and Malaysia.

In addition, the issue of ceramic membrane hydrophilicity of kaolin can be solved by simple modification process via FAS grafting method. Not only it is superhydrophobic and it improves the porosity of the membrane. For example, Hubadillah *et al*, (2017) pioneered the fabrication of ceramic hollow fibre membrane from kaolin towards oily-wastewater separation and obtained a high flux and oil removal. Meanwhile, Abdulhameed *et al.*, (2017) and Mohtor *et al.*, (2018) fabricated composite ceramic hollow fibre membrane from kaolin and alumina for carbon dioxide capture and dye removal from water, respectively. It was found in the studies that the addition of alumina in kaolin ceramic membrane improved the pore size and porosity of the ceramic hollow fibre membrane. Another study by Abd Aziz *et al.*, (2020) of using FAS to modify mullite membrane to omniphobic produced a high performance of desalination by using membrane distillation showed that the effect of FAS to remove oil.

The research on the hollow fibre membrane configurations has been extensively investigated due to its high surface area and compact design. Nevertheless, the enhancement on commercial membrane by various types of modifications has been one of the interests in order to reduce the fabrication cost as such by using alternative low-cost materials, diversifying fabrication techniques to improve the separation, porosity, pore size, and enhancing the morphology, properties, and performance of the membrane. However, modification on the membrane surface such as hydrothermal treatment (Ismail *et al.*, 2020) and sol-gel coating (Chougui *et al.*, 2019) is time-consuming. The difficulty to control the hydrolysis parameter and condensation rate of the reactive precursor to match the chemical reactivity of two alkoxides during the gelation process (Kongwudthiti *et al.*, 2003) is another drawback of membrane modifications. Thus, dual-layer hollow fibre membrane fabricated using co-extrusion and co-sintering technique is the best solution to encounter aforementioned obstacles. Meanwhile, ceramic dual-layer hollow fibre membrane fabricated using co-extrusion technique is commonly applied for fuel cell (Othman *et al.*, 2010a and 2010b; Jamil *et al.*, 2015 and 2017). Plus, for water separation application, this technique was commonly fabricated from polymeric materials (Dzinun *et al.*, 2015; Wu *et al*., 2011 and Wang *et al.*, 2011).

In congruity to Sustainable Development Goal (SDG) (Franco and Abe, 2020; MacDonald *et al*, 2018), on the 17 goals presented, goal no 3 and 6 which are good health and well-being, clean water, respectively, could present the goals of this work. Pradhan *et al.*, (2017), also mention that no 3 and 6 was included on the top 10 of global ranking of SDG pairs with high shares of synergies. Previously, very limited studies have been reported on the application of ceramic hollow fibre membrane derived from alternative materials such as kaolin towards the ammonia recovery/removal via membrane contactor system. Hence, in this study, hydrophobic ceramic hollow fibre membrane derived from kaolin was fabricated and modified to investigate the feasibility of the membrane for ammonia removal via membrane contactor system to propose as an alternative for conventional polymeric membrane. Unfortunately, it should be mentioned here that the kaolin exhibits low chemical stability when in contact with high concentration of ammonia and this hinders the use of kaolin ceramic membrane in membrane contactor system. Considering this, there is a need to develop composite ceramic hollow fibre membrane derived from kaolin and zirconia. Based on our previous study, zirconia has been chosen to be fabricated together with kaolin in order to reduce the dissolubility of the ceramic membrane in high alkaline condition (Pauzan *et al.*, 2021). In the first attempt, hydrophobic single layer hollow fibre membrane derived from kaolin and zirconia was prepared and tested towards ammonia removal through membrane contactor. Then, dual layer hollow fibre membrane in which the inner layer derived from kaolin and zirconia, whereas the outer layer derived from zirconia alone was prepared. It should be stated that the study on dual layer hollow fibre membrane was conducted to compare the performance of membrane contactor. Two different types of membranes were chosen to be compared in this study, firstly, in order to investigate the effect of different particle on the surface of the membranes toward the reaction with FAS agent and secondly, to investigate the effect of different membrane's surface in different concentration of ammonia in membrane contactor application. Through this work, the use of alternative material which is kaolin which is environmental-friendly material can be used to recover/remove dangerous pollutant of ammonia via MC can provide sufficient information to be used in real life i.e. ammonia-containing-wastewater treatment either from in leads to SDG's no 3 and 6 goals by providing a good well-being and clean water.

1.2 Problem Statement

Typically, most of the commercially available membrane to be used in membrane contactor for ammonia recovery are made from polymeric material. These polymers possess hydrophobicity characteristic which is attractive to be used in this system. However, most of these polymeric hollow fibres are vulnerable to chemical and thermal stresses, resulting in morphological changes and membrane swelling. These changes have weakening effects on the membrane performance (Ahn *et al.*, 2011). Therefore, polymeric membranes are only limited to the applications with mild operating conditions, namely low acidity, alkalinity, and low temperature. Ceramic membranes have shown some advantages to be used as substitute for polymeric membrane. Several studies have shown the success in the preparation of ceramic hollow fibre membranes using phase inversion/sintering technique from alumina (Al_2O_3) (Abdullah *et al.*, 2016), yttria-stabilized zirconia (YSZ) (Paiman *et al.*, 2015) and titania (TiO_2) (Liu and Li, 2003) possess superior characteristics compared to polymeric membranes in more extreme environment. However, the higher cost of production of these materials of membrane has made it less favourable. Thus, the production of alternative ceramic membrane from naturally occurred clay or aluminosilicate materials such as kaolin has emerged as alternative starting ceramic material for ceramic membrane fabrication.

Previously, kaolin hollow fibre membrane has been successfully fabricated by Hubadillah *et al.*, (2017) and (2020) which possessed similar characteristics to commercial ceramic membrane such as alumina and silica. Kaolin is natural clay or aluminosilicate that is known to have effective criteria to be fabricated as ceramic membrane due to its natural and commercially available in naturally abundant especially in Malaysia. Not only kaolin rich with alumina and silica content which make it suitable to be as ceramic membrane fabrication starting material, kaolin also equipped with valuable properties such as non-toxicity, high porosity and refractory as well as low plasticity. Nonetheless, it was discovered that due to the high content of silica in kaolin (aluminosilicate), the membrane showed drawback in high alkaline solution, specifically high pH or concentration of ammonia. The membrane was discovered to be dissoluted in that solution which hindered the use of the membrane in MC system for ammonia recovery as one of the criteria for the system to operate is the membrane must be able to withstand high pH of ammonia used. Technically, ammonia is a basic solution due to lone pair present on the nitrogen, N atom but it will react with –OH and later becoming high basic as the pH increased. Kaolin contains high composition of silica dioxide (SiO_2) in which this unstable silica negatively surface charged which leads to highly polarized interatomic, Si-O.

In order to overcome this drawback, zirconia was added to the kaolin membrane dope suspension as previously reported by Nishiyama *et al.*, (2003) and Park *et al.*, (2003) where the addition of zirconia to the silica based membrane successfully improved the dissolution of the silica based material in high concentration of alkali. They reported that small amount of zirconia (~10 wt%) effectively enhanced the resistance against alkaline solution. In fact, Yang *et al*., (1998) stated that zirconium dioxide (ZrO_2), also known as zirconia membrane, has been one of the famous ceramic membranes due to its high chemical resistance which allows steam sterilization and cleaning procedures at very high and low pH, excellent pure water permeability and high membrane flux in separation and filtration due to their specific surface properties as well as high thermal stability leading to its wide utilisation as a ceramic material (Shimoda *et al.*, 2017). Puthai *et al.* (2016) on the other hand, altered the surface of kaolin-based membrane by adding zirconia-based materials to be used in alkaline condition. Considering the fact that zirconia has appeared to be used in ceramic membrane along with the use of other ceramic membranes fabricated from silica, alumina and titania, zirconia has been recognized to be a popular choice compared to polymeric membrane in microfiltration for wastewater due to its capability to withstand high temperature, pressure and chemical stability (Paiman *et al.*, 2015; Hubadillah *et al.*, 2018). Therefore, these two materials were chosen in this work. Previous studies on the effect of hydrophilicity of kaolin (Abbasi *et al.*, 2010;

Kumar *et al*., 2015) and zirconia (Zhou *et al*., 2010; Wang, 2000; Boussemghoune *et al.*, 2020; Yang *et al.*, 1998) have shown that the membrane support coated with zirconia showed higher flux compared to uncoated and pristine membranes. However, due to high cost of zirconia, the ratio of zirconia added to the kaolin membrane required to be minimized and hence, the newly zirconia-kaolin composition required to be studied.

In addition, dual-layer hollow fibre ceramic membrane configuration was normally applied in fuel-cell membrane-based application. In fact, dual-layer hollow fibre membrane configuration has been used in water filtration but was dominated by polymeric membrane. No previous study of using dual-layer hollow fibre configuration ceramic membrane for water filtration regardless its advantages. In addition, previous studies have shown that most method to deposit zirconia on membrane's surface was either by hydrothermal or sol-gel, which required multiple steps to fabricate the membrane. Hence, by inducing zirconia only on the surface of the mixed zirconia-kaolin hollow fibre membrane via single step phase inversion co-extrusion and co-sintering technique, creating a protective layer to the silica particles in kaolin which prevents the dissolution of the membrane in high concentration of ammonia and reduces the exposure of the kaolin part to the high concentration of ammonia. The technique also reducing the time and cost required to fabricate a dual-layer hollow fibre membrane configuration. Also, by having zirconia only on the outer layer, creating a different particles sizes for the dual-layer hollow fibre configuration compared to single layer hollow fibre configuration, enhanced the performance of MC since kaolin particle size practically bigger compared to zirconia particle size. In order to operate the MC system, the membrane required to be porous enough to allow only ammonia gas to pass through the membrane's wall, hence, by having smaller particle sizes compared to kaolin, the membrane will only allow small ammonia molecule to pass through and prevent bigger molecule such as water to pass through the membrane's wall to the permeate. The presence of different layer with different particles sizes also enhanced the mechanical strength of the membrane which is most desirable characteristic in ceramic membrane which make this membrane suitable to be used in MC system.

1.3 Objectives of Study

The main goal of this study is to develop an alternative robust dual-layer hollow fibre membrane made from composite zirconia and kaolin via single step extrusion phase inversion/sintering technique and evaluate the performance of the fabricated membrane in high concentration/pH of ammonia prior to be used for the application of ammonia recovery in membrane contactor (MC) system. To achieve this goal, following objectives need to be accomplished as follows:

i. To investigate the effect of sintering temperature and different zirconia content for an ideal mixed zirconia-kaolin hollow fibre membrane.

ii. To acquire optimized co-sintering temperature for dual-layer mixed zirconia-kaolin hollow fibre membrane in high concentration of ammonia.

iii. To evaluate the performance of the modified fabricated membranes via MC operating system for ammonia recovery application using synthetic and treated ammonia wastewater.

1.4 Scope of Study

This study was conducted to present the robust alternative dual layer hollow fibre membrane form zirconia and kaolin for ammonia recovery via MC with superhydrophobic properties. In order to achieve the objectives, research scopes are:

1.4.1 Fabrication of mixed zirconia-kaolin (ZK) hollow fibre membrane

i. Fabricating a mixed 10 wt% of zirconia and 30wt% of kaolin hollow fibre membrane (ZK) via phase inversion technique.

ii. Determining the best sintering temperature of the fabricated mixed ZK hollow fibre membrane at 1200 to 1400°C.

iii. Investigating the effect of zirconia content on the optimized sintering temperature mixed zirconia-kaolin hollow fibre membrane by varying the zirconia content added at 5, 7 and 10wt%.

iv. Characterizing the composition of elements in kaolin powder, the morphology of the fabricated (cross-sectional and surface) using scanning electron microscopy (SEM) and energy dispersive X-Ray (EDX), mechanical strength, pore size distribution and porosity, water permeability and the dissolution of the membrane in high concentration of ammonia in term of weight loss of the membrane.

1.4.2 Fabrication of dual layer hollow fibre membrane from mixed zirconia and kaolin

i. Fabricating a dual-layer hollow fibre membrane (DLZK) from the previous optimized ZK membrane composition at inner layer and pure zirconia only at the outer layer by using single step phase inversion co-extrusion method.

ii. Determining the best co-sintering temperature of the fabricated DLZK membrane at 1200 to 1500°C.

iii. Characterizing the morphology of DLZK membrane (cross-sectional and surface) mechanical strength, pore size distribution and porosity, pure water permeability and weight loss of the membrane for the dissolution at high concentration of ammonia.

1.4.3 Membrane contactor application for ammonia recovery using optimized mixed ZK hollow fibre membrane

i. Modifying the hydrophilic surface of the fabricated mixed zirconia-kaolin hollow fibre membranes (SLZK and DLZK) via immersion grafting method with FAS agent.

ii. Characterizing the hydrophobicity of the modified ceramic membrane in term of its surface roughness, contact angle measurement while the wettability of the membranes was characterized via liquid entry pressure (LEPw).

iii. Constructing membrane contactor (MC) reactor system for the performance of the modified membranes for ammonia recovery and removal by contacting two concentration of ammonium hydroxide (NH_4OH) i.e., 5M (pH 9-11) and 10M (pH 11-13) with sulphuric acid (H_2SO_4) at 0.5 ms^{-1} of flow rate and 1 bar of pressure for 180 minutes of duration time.

iv. Comparing the performance of the fabricated mixed zirconia-kaolin hollow fibre membrane (SLZK and DLZK) in term of transferred rate of ammonia or mass transfer coefficient (Kms^{-1}), percentage of reduced ammonia concentration, analysed using HACH reagents and read using DR5000 spectrophotometer and lastly, mean comparison using statistical test.

v. Evaluating the purity of the ammonia recovered from the system by using Fourier transform infrared spectroscopy-attenuated total reflectance (FTIR-ATR).

vi. Investigating the performance of optimized ZK hollow fibre membrane for ammonia recovery in MC application with same operating condition by using treated wastewater i.e. treated palm oil mill effluent (POME).

vii. Analysing the quality of the wastewater by using HACH reagents and read using DR5000 Spectrophotometer.

1.5 Significance of Study

The study contributes to the alternative approach of using ceramic membrane in membrane contactor (MC) for the removal and recovery of ammonia from wastewater operation which dominantly by polymeric membrane. By doing so, this work indirectly contributes to exploration on the interaction of naturally silica-rich aluminosilicate clay (kaolin) with ammonia or alkali solution especially at high concentration or pH. Besides, the fabrication of ceramic dual-layer hollow fibre membrane also contributes to pioneer study of using naturally ceramic materials for water filtration purposes especially in treated wastewater and provide opportunities for mixed zirconia and kaolin in dual-layer hollow fibre membrane configuration which has not been reported previously. Last but not least, this study produces a robust membrane fabricated from alternative ceramic materials which worked well in harsh condition i.e. high alkaline condition.

1.6 Thesis Organization

This thesis provides clear information on the fabrication of two different configurations of ceramic hollow fibre membrane made up of kaolin and zirconia for ammonia recovery via membrane contactor. The organization of this thesis is as follows:

Chapter 1 depicts the issues and gaps of current membrane contactor for ammonia recovery related work. In order to affiliate this studies, three objectives were proposed and continued with the scopes that serve to accomplish the objectives. The significance of this study also fully explained. Chapter 2 presents the current and past reviews on the literature related to this study which amplify the fundamental of ammonia removal/recovery via variety of methods or techniques, the advantage of ammonia recovery via membrane contactor, problems related to its operations as well as comparison of using this method by other researchers. The benefit of using ceramic membranes are also reviewed in this chapter especially the problem which hindered the use of kaolin as ceramic membrane in MC. Meanwhile, Chapter 3 describes the methodology of this study which includes step-by-step obliged towards reaching the main goal. The research framework which comprises of all materials used, experimental operation, laboratory procedures and concluded by characterization of the fabricated membranes and the performance of MC application via analytical and numerical method was also described in detail.

In continuation to previous chapters, the results and discussion of this study were divided to three chapters, chapter four to six which compromises of respective objective at consecutive chapter. Chapter 4 elucidates the fabrication of mixed zirconia-kaolin (ZK) hollow fibre membrane at different sintering temperature and zirconia content. Followed by the description of the membrane properties including its morphology, mechanical strength, permeability, pore size, porosity as well as dissolution study of the membrane in high concentration of ammonia in term of weight loss. The optimized sintering temperature and zirconia content of this fabricated membrane will be used for next chapter. Chapter 5 annotates the fabrication of dual-layer mixed zirconia-kaolin hollow fibre membrane (DLZK) via single step phase inversion co-extrusion technique from optimized ZK membrane based on previous chapter at inner layer and zirconia only on outer layer. The characteristics and properties of the fabricated membrane was investigated in term of different co-sintering temperature with similar characterization and test as previous chapter. Chapter 6 describes the comparison study of those two fabricated membranes (ZK and DLZK) for ammonia recovery via membrane contactor (MC) application. The ZK membrane was renamed to SLZK for clear comparison. Started with modification of the fabricated membranes from hydrophilic to hydrophobic by grafting method prior to be used in MC system using FAS agent. The surface analytical and wettability of the membrane was presented in detail and the membranes were compared to investigate effect of having different layers of the membrane towards hydrophobicity. In membrane contactor system for ammonia recovery, comparison was done using two different concentration/pH of ammonium hydroxide, contacted with sulphuric acid for both membranes. Later, the performance was evaluated in term of percentage of ammonia recovered, percentage of ammonia reduction and mass transfer coefficient. The optimized membrane was then evaluated in MC by using treated wastewater which is from palm oil mill effluent (POME). Chapter 7 concludes all the discussions and findings from the study and recommendation for future work to fill the gap or information towards better understanding and knowledge.

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LIST OF PUBLICATIONS

Article in Impact Factor Journal

i. **Pauzan, M. A. B.**, Hubadillah, S. K., Kamal, S. N. E. A. M., Othman, M. H. D., Puteh, M. H., Kurniawan, T. A., & Kadir, S. H. S. A. (2021). Novel ceramic hollow fibre membranes contactor derived from kaolin and zirconia for ammonia removal and recovery from synthetic ammonia. *Journal of Membrane Science*, 638, 119707. (**Q1, IF: 8.742)**

ii. **Pauzan, M. A. B**., Othman, M. H. D., Ismail, N. J., Puteh, M. H., Ismail, A. F., Rahman, M. A., & Jaafar, J. (2021). Fabrication of zirconia-kaolin dual layer hollow fiber membrane: Physical and performance study for industrial wastewater treatment. *Journal of Water Process Engineering*, 41, 102031. (**Q1, IF: 5.485)**

iii. **Pauzan, M. A. B**., Hubadillah, S. K., Ismail, N. J., Othman M. H. D., Puteh M. H., & Kadir, S. H. S. A. (2021). Fabrication and characterization of robust zirconia-kaolin hollow fiber membrane: Alkaline dissolution study in ammonia solution. *Korean Journal of Chemical Engineering*, 1975-7220. (**Q2, IF: 3.309)**

iv. **Pauzan, M. A. B.**, Puteh, M. H., Yuzir, A., Othman, M. H. D., Abdul Wahab, R., & Zainal Abideen, M. (2020). Optimizing ammonia removal from landfill leachate using natural and synthetic zeolite through statically designed experiment. *Arabian Journal for Science and Engineering*, 45(5), 3657-3669. (**Q2, IF: 2.334)**

Book Chapter

i. **Pauzan, M. A. B.**, Abd Rahman, M., & Othman, M. H. D. (2021). Hydrocarbon Separation and Removal Using Membranes. In Membrane Technology Enhancement for Environmental Protection and Sustainable Industrial Growth (pp. 73-90). Springer, Cham.