STUDY ON THE GAS PERFORMANCE OF CERAMIC MEMBRANE FROM KAOLIN PREPARED BY PHASE INVERSION TECHNIQUE

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Special dedication to:

My Beloved Father, Mr Hubadillah Hj Mohamed

My Beloved Mother, Mrs Rosnah Bt Hashim

and

For Beloved Siblings,

Madam Siti Aminah Bt Hubadillah

Kapten Ibrahim Bin Hubadillah

Dr. Siti Fatimah Bt Hubadillah

Prof. Mohamed Ismail Bin Hubadillah

and

My intelligent friend, Nurul Nadia aka Alya

I love you all….

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ABSTRACT

Membrane for gas application have been widely used. Apart from that, ceramic membrane is gaining much attention towards separation technology due to its characteristics of offering high mechanical strength, chemical resistivity and thermal compatibility. However, production of ceramic membrane for gas separation in term of cost and energy reduce still remains as challenge until now, therefore this work addressed to the development of ceramic membrane from kaolin via simple phase inversion technique. First, ceramic membrane suspension have been prepared by stirring kaolin as raw material, N-methyl-2-pyrollidone (NMP) as solvent and polyethersulfone (PESf) as binder. Phase inversion tchnique conducted by casted the suspension on the glass plate with casting knife. In order to achieve the aims of this study, the development of ceramic membrane from kaolin were conducted into two objectives: (1) effect of particles sizes and, (2) effect of non-solvent coagulant bath. The types of kaolin particle sizes devoted as Type A kaolin (0.4-0.6µm) and Type B kaolin (10-15µm) whereas for different types of non-solvent were distilled water, ethanol and mixture of 70% NMP and 30% distilled water. Overall analysis showed that both effect of particle size and different caogulant generated different structure, properties and characteristic of membrane at two different composition. Polymer phase inversion is dominated at kaolin content of 24 to 34wt.% that caused the formation of finger-like voids of the phase inversed structure with Type A kaolin and strong caogulant of distilled water. An opposed condition was shown at highest kaolin content of 39 wt.% for both parameter that can be correlated to the viscous fingering mechanism in the formation of ceramic membrane structure. A slightly similar results trends and pattern was demostrated with Type B kaolin and weakest coagulant (mixture of 70% NMP and 30% distilled water). Overall performance showed that membrane with Type A kaolin and immersed into ethanol as coagulant at kaolin content of 39 wt.% showed the highest rejection (5.49 and 5.82 for CO_2/N_2 and O_2/N_2, respectively).

ABSTRAK

Membran bagi aplikasi gas telah digunakan secara meluas. Selain itu, membran seramik mendapat banyak perhatian terhadap teknologi pemisahan kerana ciri-cirinya dalam menawarkan kekuatan mekanikal yang tinggi, rintangan kimia dan keserasian terma. Walau bagaimanapun, pengeluaran membran seramik untuk pemisahan gas dari segi kos dan tenaga masih kekal sebagai cabaran sehingga sekarang, oleh itu kerja-kerja ini ditujukan kepada pembangunan membran seramik dari kaolin melalui teknik fasa penyongsangan mudah. Pertama, membran seramik telah disediakan dengan teknik kacau daripada kaolin sebagai bahan mentah, N-methyl-2-pyrollidone (NMP) sebagai pelarut dan polyethersulfone (PESf) sebagai pengikat. Teknik fasa penyongsangan dijalankan pada plat kaca dimana membrane dibentuk oleh pisau pembentuk. Untuk mencapai matlamat kajian ini, pembangunan membran seramik dari kaolin telah dijalankan kepada dua matlamat: (1) kesan zarah saiz dan, (2) kesan mandi koagulan bukan pelarut. Jenis-jenis saiz kaolin dikhaskan sebagai Jenis A kaolin (0.4-0.6μm) dan Jenis B kaolin (10-15μm) manakala bagi jenis bukan pelarut ialah air suling, etanol dan campuran 70% NMP dan 30% air suling . Analisis keseluruhan menunjukkan bahawa kedua-dua kesan saiz zarah dan koagulan berbeza menghasilkan struktur berbeza, sifat-sifat dan ciri-ciri membran daripada dua komposisi yang berbeza. Fasa polimer penyongsangan dikuasai pada kandungan kaolin daripada 24 hingga 34wt.% Yang menyebabkan pembentukan lompang jari daripada kaolin Jenis A dan koagulan kuat air suling. Keadaan bertentangan ditunjukkan pada kandungan kaolin tertinggi 39 wt.% Untuk kedua-dua parameter yang boleh dikaitkan dengan mekanisma jari likat dalam pembentukan struktur membran seramik. Trend yang sama telah dihasilkan oleh membran daripada kaolin Jenis B kaolin dan koagulan paling lemah (campuran 70% NMP dan 30% air suling). Prestasi keseluruhan menunjukkan bahawa membran dengan kaolin Jenis A dan etanol sebagai koagulan di kandungan kaolin 39 wt.% menunjukkan gas penolakan yang paling tinggi (5.49 dan 5.82 untuk CO_2 / N_2 dan O_2 / N_2).

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LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscopy
Al	-	Aluminum
Al_2O_3	-	Alumina Oxide
BSCF	-	Ba0.5Sr0.5Co0.8Fe0.2O3-δ
CH_4	-	Methane
CO_2	-	Carbon Dioxide
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
MF	-	Microfiltration
MIP	-	Mercury Intrusion Porosimetry
N_2	-	Nitrogen
NF	-	Nanofiltration
Ni	-	Nickel
NiO	-	Nickel (II) Oxide
NMP	-	N-methylpyrrolidone
O	-	Oxide
O_2	-	Oxygen
PESf	-	Polyethersulfone
PSA	-	Particle Size Analysis
PSD	-	Pore Size Distribution
PVDF	-	Polyvinylidenedifloride
RO	-	Reverse Osmosis
SEM	-	Scanning Electron Microscopy
Si	-	Silica
SiO_2	-	Silica (II) Oxide
SOFc	-	Solid Oxide Fuel Cell
ZrO_2	-	Zirconia Oxide

LIST OF SYMBOLS

σ_b	-	Mechanical Strength
R_a	-	Surface Roughness
F	-	Fracture Force
L	-	Membrane Span Length
w	-	Membrane Width
t	-	Membrane Thickness
P	-	Gas Permeability
Q	-	Flowrate
A	-	Membrane Area
Δ_p	-	Pressure Gradient

CHAPTER 1

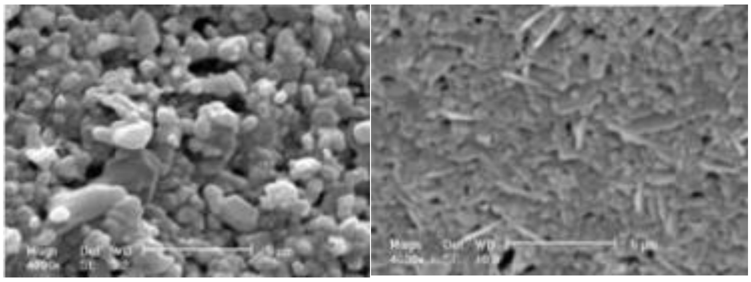
INTRODUCTION

1.1 Background of Study

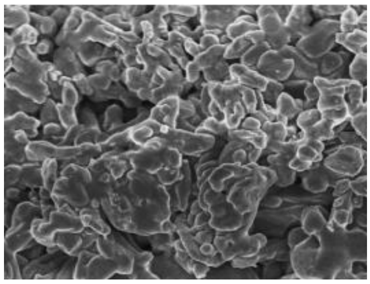
The impact of global warming caused by discharges of CO_2 from various sources is a major worry. According to the Environmental Protection Agency, the U.S. emitted 6,673 million metric tons of CO_2 into the atmosphere in 2013 [1]. The escalating level of atmospheric CO_2 has caused widespread public concern [2]. One of the major sources of CO_2 emission is fossil fuel combustion. Today, coal-fired power plants alone emit about 2 billion tons of CO_2 per year [3]. Imagine for another 30 years, fossil fuels will remain an important source of the world's energy. For instance, high indoor levels of CO_2 could lead to severe health effects, even death. In fact, carbon dioxide level is allowed to be at the 5% level or 600 ppm [4] in normal environment. The exceed value will help increase a poisonous quality which harm to health. A recent data from Intergovermental panel on climate change in 2014 reveal that the CO_2 concentration has reached 394ppm [5] in the atmosphere which is far beyond the upper safety limit of 350ppm [6]. Hence, separation of CO_2 removal is crucially required for most of industry actions to avoid excessive CO_2 gas pollution. Other than CO_2, the separation of oxygen from nitrogen in the air also is an important procedure that is widely used in manufacturing. Oxygen is utilized to give advantages in various chemical processes such as natural gas combustion, coal gasification, sewage treatment and welding. Whilst, nitrogen used as a low-temperature coolant, an inert diluent and in the production of ammonia.

There are various method used in gas separation technology such as cryogenic distillation, pressure swing adsorption and liquid solvent absorption have been proposed. However, the cost associated with the above mentioned processes are about 80% of the total cost of CO_2 sequestration and thus are not fiscally viable. Membranes offer a promising separation technology to execute process intensification strategies, that is the cutback of production costs by minimizing equipment size, energy consumption and waste production [7]. Apart from that, there are two types of membrane: (1) polymeric membrane and (2) inorganic membrane. Polymeric membranes are widely used among all types of membranes. This has, in accordance of polymeric membranes offered a cheaper method, and yet the simple fabrication process involved. However, it is well known challenges of low selectivity and permeability in polymer membranes which have been addressed with some degree of success with advanced engineering polymers [7]. Furthermore, the physical and properties of polymer which cannot endure the application requiring high thermal, chemical and strength has limit the application of polymer membrane for critical applications. Because of this, researchers have taken attention towards inorganic membranes that has the ability to sustain at high temperature and extreme condition. In general, ceramic membrane can be subdivided into another three types: (1) ceramic membrane, (2) metal membrane and, (3) carbon membrane. The latest development has shown that ceramic membrane is gaining a place in gas separation compared to other membranes as it is offer simple sieving mechanism through the pore size obtained [8]. Since ceramic membranes offer significant advantages over polymeric membranes, therefore, it has been widely used especially in oilywater separation [9], wastewater separation [10] and water separation [11]. However, for the past few years the application of ceramic membrane still dominated the application in many gas industries which demanded high thermal and pressure.

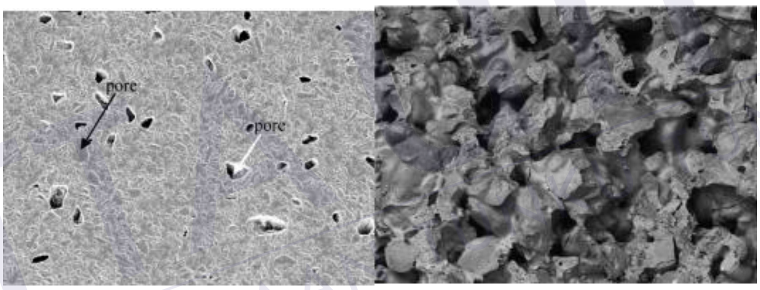
There are sundry unique structure could be obtained in ceramic membrane fabricated at different method as shown in Figure 1.1. In gas application, each type of dense membrane structure could be applied according to felicitous gas application based on their pore size and surface properties in order to have a high selectivity and a low permeation rate. The ability of ceramic membranes could be produced or fabricated towards desired structure such as dense or porous membranes has attracted intensive study of ceramic membrane development and modification such as high temperature, highly acidic or basic feed [12-14]. For instance, the past decade has witnessed major changes in this area as dense ceramic membranes exhibiting high oxygen ionic and electronic conductivity have become of greatest interest as it is economical, clear and effective way of producing oxygen by separation from air or other oxygen containing gas mixtures.



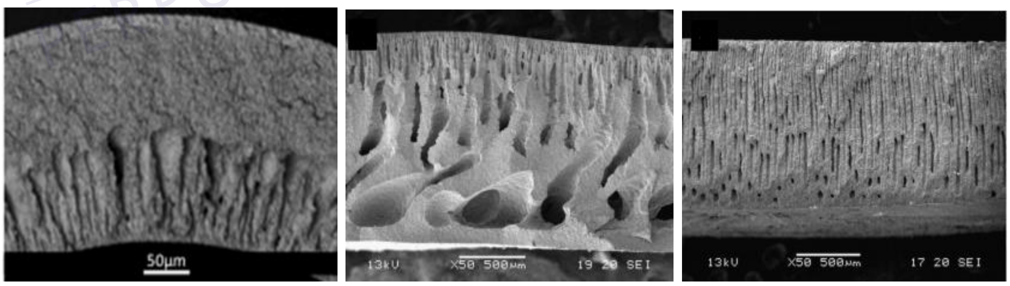
Slip casting



Tape casting



Pressing method



Phase Inversion

Figure 1.1 :Ceramic membrane structure prepared at different techniques

In fabricating ceramic membranes, there are many methodc can be used as tape casting, slip casting, pressing method and extrusion. All of these methods have been established and applied for years. Fabrication of ceramic membranes by these techniques generally involves multiple steps. Generally, it can be subdivided into three stages: (1) preparation of the ceramic powder paste or suspension, (2) shaping of the ceramic powder into the desired geometry and, (3) heat treatment which including calcination and sintering [15]. After completing these main steps, additional deposition layer or coating layer needs to be formed to allow selective mechanism at the top layer, Then, further heat treatment steps need to be conducted in orders to have a good selectivity as well as to improve other membrane properties. The choice of method for each step depends on the desired membrane configuration, quality, morphology, mechanical, chemical stability and selectivity of the desired final membranes. However, the fabrication method should also be economical and easy to replicate without compromising the quality of final membrane [16].

Phase inversion is a simple technique of membrane fabrication which commonly used in polymeric membranes. The first invention by Loeb and Sourirajan in 1963 is one step technique that able to form asymmetric membrane structure offer a high ability of high flux as well as rejection and selectivity [17]. Recent development of ceramic membrane have shown the implementation of this technique is able to improve gas separation property. As reported, membrane fabrication via phase inversion has promoted a dense top layer with smallest pore size. However, ceramic membrane fabricated via phase inversion so far appear to hold two types of structure: finger-like and sponge-like structure. Figure 1.2 illustrates the structure of membrane that could be obtained by phase inversion. The structure of macrovoids is sometimes observed in ceramic membrane structure. In phase inversion technique, commonly the description of structure always been explained by the concept of ternary diagram, involving three main components: solvent, non-solvent and polymer. However, due to ceramic membranes involve an additional component of ceramic powder, the formation of ceramic membranes can be explained by the viscous fingering mechanism as demonstrated by previous researcher [12, 18]. Viscous fingering is the responsible mechanism that permits the establishment of dense porous structure with certain selectivity. The capability of phase inversion in formation of ceramic membrane lifetime and properties in single step technique has attracted many researchers in developing ceramic membrane especially in gas application.

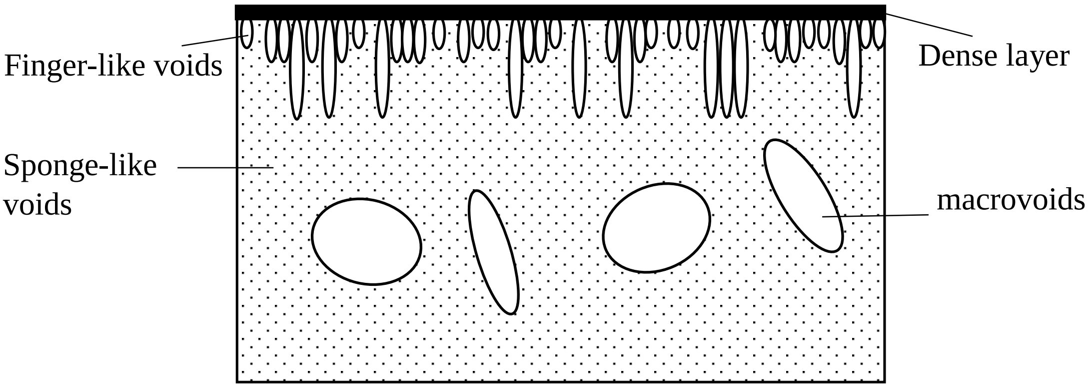


Figure 1.2: Ceramic Membrane Structure fabricated via phase inversion [15]

Ceramic membrane fabricated from phase inversion technique used materials like Alumina, Zirconia and Titania which considered as very expensive materials. Overall comparisons showed that polymeric membranes are still more inexpensive to get compared to ceramic membranes. Even so, a technical and economical comparison between different membrane processes must take into account both investment and maintenance. Investment involves the price of equipment for pretreatment and post treatment of fluids in addition to the monetary value of the membrane plant. Maintenance includes replacement of membranes, electricity consumption, cleaning products and labor prices. Consequently, a comparison of overall costs, including membrane lifetime, cleaning procedures and pretreatment has put a ceramic membrane is a preferable material in a number of applications. More than that, the technical progress made in the manufacture of ceramic membranes with production costs getting closer to those of many polymeric membranes explains why they are entering markets much more extensive than those accessible of the first polymer membranes. In fact, ceramic membrane developed from kaolin and clay has gained attention recently as it employs to offer better properties and at the same time cut off the cost of materials.

1.2 Statement of Problems

Gas separation application seems to be an important research nowadays. This is due to the problem of carbon dioxide emission that could affect severe health. In addition, others gas separation such as oxygen and nitrogen that could be used in medication and production of ammonia, respectively have attracting many researchers in this research.

Membrane for gas separation application have been used in past few years according to the advantages have been promoted. There are two types of membrane which are polymeric and inorganic membrane. However, polymeric membrane has disadvantages which cannot endure high temperature, chemical resistivity and mechanical strength, thus, not suitable for gas application. Due to this, ceramic membrane, a part of inorganic membrane have gaining attention. The combination of high chemical, thermal and mechanical resistance has made ceramic membranes an attractive alternative to polymeric membranes.

In ceramic membrane fabrication process, two most important parameters that always being investigated are materials and fabrication method. For materials, previous study have focused towards refractory ceramic materials such as alumina, nickel and zirconia. These materials are just not requiring high sintering temperature, but expensive too. Due to this, kaolin have become attractive materials to be study in ceramic membrane fabrication. Kaolin is not only cheap, but provides low plasticity and high refractories properties to the membranes [19]. For fabrication methods, pressing method is the one most method that have been used in ceramic membrane fabrication and is well-known method for ceramic membrane. However, pressing method could only produce symmetric membrane structure and need additional method to produce separation layer. Furthermore, pressing method is the most expensive method. Clearly, combining a suitable materials and fabrication method which can cut the time and cost, and hence ceramic membrane price is desirable.

Phase inversion into ceramic membrane fabrication is a promising technique to produce ceramic membrane in a single step at lower cost. It is fast and cost effective technique which hence can promote unique structure (finger-like and sponge-like structure). However, phase inversion in ceramic membrane requiring complex system as many parameters can be involved during fabrication process. Thus the investigation on ceramic membrane fabrication via phase inversion technique was be conducted in this study by study the effect of kaolin particle size, kaolin content and different types of non-solvent coagulant bath.

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