An Experimental Investigation of Humidity Effect on fabrication of Green Ceramic Membrane Prepared Via Dry Phase Inversion

Mohd Riduan Jamalludin¹*, Siti Khadijah Hubadillah², Zawati Harun³, Mohd Hafiz Dzarfan Othman³ Muhammad Ikman Ishak¹, C.Y. Khor³, M.U. Rosli³, M.A.M Nawi¹, Mohamad Syafiq A.K.¹, Lailina N.M¹, Ras izzati Ismail¹ and Lee Yit Leng⁶

¹ Faculty of Engineering Technology, Universiti Malaysia Perlis (UniMAP), Kampus UniCITI Alam, Sungai Chuchuh, Padang Besar 02100 Perlis Malaysia.
² Advanced Membrane Technology Research Centre (AMTEC), Faculty of Petroleum and Renewable Energy Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor Darul Takzim, Malaysia.
³ Advanced Materials and Manufacturing Centre (AMMC), Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Batu Pahat, Johor Darul Takzim, Malaysia.

Email: riduanj@unimap.edu.my

Abstract. Ceramic membranes are gaining a market demand nowadays due to its characterization and performance. However, in the preparation of ceramic membrane for water and wastewater separation, the processes involved are taking a higher cost. In this work, ceramic membranes were prepared via phase inversion within controlled humidity by using different composition of low cost and green ceramic material, kaolin. The preparation of ceramic membranes was conducted via phase inversion in high humidity; i) 3oC and, ii) room temperature. In this paper, we report on the high humidity affected the viscosity of membrane suspension. Thus, the viscosity of ceramic suspension was giving integration towards membrane characterization and performance. A dense membrane structure was observed when the ceramic membranes were prepared in high humidity and kaolin composition. The morphology of overall, porous and dense structure of ceramic membranes was characterized by field emission scanning electron microscope (FESEM) at x50, x500 and x5000 magnification respectively.

1. Introduction

Recently the use of membrane technology is widely employed into water treatment processes and industrial wastewater treatment due to their low cost production [1]. In fact, stringent regulations in industrialized countries and increasing water problem have promoted the use of membrane for water treatment. For two decades experience, water companies now operate thousands of membrane plants of treated water every day using membrane processes such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO). Compared with conventional water treatment
process, such as clarification and sand filtration, membrane technology is an absolute barrier and therefore offers the advantage of selectively removing contaminants based on their sizes.

In membrane technology, polymeric membranes have been almost exclusively dealt with; however, alternative membrane materials have been getting attraction nowadays. Ceramic membranes have much greater advantages, which, because of its endurance to high temperature, high chemical resistance as well as high pressure [2-4]. In selectivity of raw materials, several studies on ceramic membrane preparation using ceramic materials such as Alumina (Al₂O₃), Zirconia (ZrO₂), metal oxide or metal alloys were carried out by Liu et al. [5]. However, in water and wastewater treatment, the production of ceramic membrane has been limited due to high cost production. Therefore, the low cost ceramic such as kaolin, raw clay, moroccan clay, Tunisian clay, sepiolite clay, Algerian clay and dolomite was intensively studied by many researchers in the preparation of low cost ceramic membrane. Kaolin is one of the cheapest membrane raw materials and easily available [6-8]. In addition, kaolin is hydrophilic and can be dispersed in water and in some other systems. Because of the nature of the chemistry of its surface, kaolin can be chemically modified so that it will become hydrophobic or organophilic, or both.

As far as we are concerned, the investigations in preparation of ceramic membrane with low cost ceramic materials (kaolin) composition via dry phase inversion system were rarely studied. Thus, in this work, the effect of different composition with low cost ceramic, kaolin was studied by phase inversion system within the controlled higher humidity involved. The prepared membranes were characterized via scanning electron microscopy (SEM) while the performance of the ceramic membrane was tested by flux within the pressure of gravity.

2. Methodology

2.1 Materials

Kaolin powder was purchased from QReC as the ceramic material. 1-Methyl-2-pyrrolidinone (NMP) was purchased from Merck. Polyether Sulfone (PESf, UDEL) was provided by BGOIL CHEM. The kaolin powder and the PESf were stored in 100°C in the oven before use to remove adsorbed moisture, and the other materials were used without any purification.

2.2 Fabrication of Green Ceramic Membrane via Dry Phase Inversion

To fabricate flat-sheet ceramic membranes within controlled humidity, the ceramic membranes were casted on the glass. The masking tape with 1.5mm was used as to control the thickness of flat-sheet ceramic membrane. The membrane was placed in the ice box set up within 3°C in order to let the phase inversion occur at high humidity. In order to investigate the preparation of flat-sheet ceramic membrane via high humidity, the membrane was differentiated with the fabrication of flat-sheet ceramic membrane in room temperature. The ceramic membranes casted on the glass were placed in desired controlled humidity for 24 hours. The phase inversion occurred at this time. After that, the glass with suspension is immersed into the non-solvent bath horizontally (Fig 1) for another 24 hours. The non-solvent used was distilled water. This immersion was done in order to remove NMP from the ceramic membrane and solidified the membrane completely.

The membrane was dried at room temperature for 24 hours. Then, the membrane was cut into the circle shape in order to record the diameter and thickness before and after sinter. This step was repeated for different composition of kaolin.
2.3 Characterization of Green Ceramic Membrane

Observation of the membrane structure was conducted using a scanning electron microscope (JEOL JSM6380LA, Germany). The diameter and thickness of ceramic membrane before and after sinter were recorded to study the effect of humidity on the shrinkage of ceramic membrane prepared by low cost ceramic material. The samples were cut into the circle shape before sinter. In order to study the difference of structure formed by the effect of different humidity of the phase inversion method, the membrane were prepared in the 2x2 cm shape for porosity and density of ceramic membrane test. The test was carried by Mettler Toledo density test.

3. Results and Discussion

3.1 Morphology of Green Ceramic Membrane

In this study, the macrostructure of membrane cross section do not showed any finger-like formation of macrovoid (Figure. 2). This is due to the structure have been solidified at top of the flat-sheet ceramic membrane during phase inversion method at high humidity. Previously, during the phase inversion system, the NMP will move out from the bottom of the membrane while the droplet of water formed the fingerlike structure on the surface of the ceramic membrane. In this work, the phase inversion system is controlled within the high humidity in the dry condition. The phase inversion in this work was studied by drying method in order to avoid the unbalanced membrane solidified. This method was also successfully prepared in the formation of the dense layer at the top of ceramic membrane.

Ceramic membranes are usually asymmetric or composite type consisting of several layers of one or more different ceramic materials. In this study, the structure morphology was significantly showed that ceramic membrane that fabricated at higher humidity was more porous compare to membrane fabricated at room temperature. Previous study has shown that low humidity shrink the surface pore size. In the study, the effect of high temperature water have been studied towards the structure of pore on Cellulose Acetate (CA) membrane and found that low humidity made the pore size getting smaller [9].
3.2 Porosity and Density of Green Ceramic Membrane

Figure 2. FESEM images of prepared ceramic membranes: (a) whole view x50 (b) dense layer cross section x5000 at (M1) 60g of kaolin at 3°C; (M2) 70g of kaolin at 3°C; (M3) 80g of kaolin at 3°C; (M4) 60g of kaolin at room temperature; (M5) 70g of kaolin at room temperature; (M6) 80g of kaolin at room temperature.

Figure 3. Experimental porosity decline and density incline within the composition of membrane and at temperature of A) room temperature; and B) 3°C

Figure 3 above presents the porosity and density of green ceramic membrane prepared at two different humidity. Accordingly, sintering temperature at 1100 ºC lead the porosity surface appeared. Theoretically, when the porosity is high, the density opposed the value of porosity. Water absorption from the high porosity is high due to number of pore consist in the specimen. P. Hristov et al. prove
the same result as plotted at the porosity and density graph. In their work, they studied about the 
porosity decrease with the increasing sinter temperature. In meantime, when the porosity declined, the 
density inclined as well [10]. In this work, we observed that the same trend plotted for both condition. 
However, in comparing the porosity of the ceramic membrane fabricated by different temperature, the 
porosity of ceramic membrane fabricated in low temperature (3°C) was higher than room temperature. 
This was supported by the morphology structure (Fig.2). The ceramic membranes fabricated at high 
humidity (3°C) had bigger pores sizes.

4. Conclusions
This investigation verified the following two distinguishing characteristics of green ceramic 
membrane suspension:
1) The structural morphology of membrane’s dense and porous layer are affected by the factor 
involved during phase inversion system. In this work, we are focused on the composition of the kaolin 
and temperature of the phase inversion system. We attribute this system produced a dense membrane 
which hence more suitable in the gas separation application.
2) By controlled the humidity of the phase inversion system, the unsolidified of the membrane can 
be solved. As, expected, the humidity will affect the viscosity of the ceramic suspension, hence, 
develop the non-fingerlike structure of membrane.

References
1. Yunos M Z et al 2013 Jurnal Teknologi 65 111.
2. Wang H et al 2010 Mater. Sci. Eng. 527 2881.
3. Ishak M I et al 2017 MATEC Web of Conferences 97 01040.
4. Shohur M F et al 2013 Jurnal Teknologi 65 59.
5. Ishak M I et al 2017 Journal of Medical and Biological Engineering 37 336.
6. Liu L et al 2010 Ceramics International 36 2087.
7. Hubadillah S K et al 2018 Ceramics International 44 10498.
8. Harun Z et al 2014 Applied Mechanics and Materials 575 31.
9. Jamalludin M R et al 2016 Chemical Engineering Research and Design 114 268.
10. Ghaseminezhad S M et al 2019 Composites Part B: Engineering 161 320.
11. Hristov P et al 2012 Journal of the University of Chemical Technology and Metallurgy 47 476.