Effect of sintering temperature on composite hollow fibre membrane derived from hydroxyapatite cow bone and kaolin

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Effect of sintering temperature on composite hollow fibre membrane derived from hydroxyapatite cow bone and kaolin

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Abstract. A modern application of composite hollow fibre membrane derived from hydroxyapatite cow bone and kaolin has been developed in wastewater treatment. The common fabrication method, a process that combined both phase inversion and sintering technique was used to fabricate the membranes. The hollow fibers membrane were developed using the spinning process by using a dope that have ratio of 20:20 (kaolin:cow bone hydroxyapatite). The sintering temperature that used in this study are 1000 ℃, 1100 ℃, 1200 ℃ and 1300 ℃. The effect of sintering temperature on hollow fiber membranes were characterized by scanning electron microscopy (SEM), 3-point bending and pure water flux. The results showed that the hollow fibre membrane sintered at 1200 ℃ showed a unique membrane's morphology, the highest mechanical strength (13.33 Mpa) and a stable pure water flux (140.6 L/m^{2}h).

1. Introduction

Due to growth of human population and rapid industrialization, water pollution has become one of the most serious global problems of this century. Water contamination is a significant challenge to the human health and environment, so work to improve the technologies of water treatment are still ongoing. Treating water contaminated is a tough duty for the people that work in wastewater industry. Various methods have been proposed to remove contaminants from aqueous systems. The methods are chemical precipitation, adsorption, ion exchange and membrane technology. Membrane technology is included as a promising method for treatment of water because of its consistent water quality, high energy efficiency, flexibility, ease of operation and low cost [1].

The available membranes that most commercially in the industry is usually made from polymers materials. Nowadays polymer membranes are widely utilized in the industry as it is low cost and mass production of polymers [2]. However, polymer membranes cannot stand at harsh condition such as high temperature and high pH. Thus, most of the researcher nowadays give attention to turn the possibility of ceramic membranes to be alternative to replace polymer membranes. Ceramic membrane have benefits such as great thermal stability, high mechanical strength, good resistance to solvents and chemicals, and long-lasting stability. In fact, the use of ceramic membrane has proven to be more beneficial and effective than conventional steps in wastewater treatment [3]. Zirconia, silica, alumina and titania are generally the common raw materials for the manufacture of commercialized ceramic membranes [4]. However, these materials are expensive and induce high melting point, making the cost for ceramic membrane fabrication extremely high [5]. Therefore, replacing those expensive materials with alternative natural sources such as clay and waste are vital.

Nowadays, hydroxyapatite powder has been utilized as an inexpensive material compared to other materials. The teeth and animal bones main component is hydroxyapatite (HAp) with the common formula of $Ca_{10}(PO_4)_6(OH)_2$ [6]. Hydroxyapatite are now have been the materials to make bioceramics [7]. The properties of hydroxyapatite are non-toxic, biocompatible and bioactive [8]. HAp powder has been produced by using bio-products such as cuttlefish shells, natural calcite, corals, bovine bone and natural gypsum. Chemical analysis proved that all of these products are full sources of calcium that form in oxide and carbonates [9]. Compared to hydroxyapatite that produced via synthetic techniques such as radio frequency thermal plasma and microwave irradiation, the extraction of hydroxyapatite from bio-waste is environmentally safe since it has no chemical use, so this situation creates a demand for bio-ceramics hydroxyapatite.

Meanwhile, kaolin [$Al_2Si_2O_5(OH)_4$] is a typical phyllosilicate mineral, that are belong to a large general group that called clays [10]. Of all kinds of clays, kaolin is the most noticeable because of its special physical features such as kaolin gives the membrane with high refractory properties and low plasticity[11]. Furthermore, kaolin is hydrophilic [12], which is extremely important for the fabrication of membrane for water filtration. Kaolin is the raw materials that is selected for porous ceramic membranes because of its characteristics such as mineralogical properties, chemical composition and crystal order. Kaolin have encouraged research to establish ceramic membrane at a lower cost due to its outstanding properties [13].

In this study, ceramic hollow fibre membrane derived from hydroxyapatite based cow bone and kaolin clay will be fabricated via common phase inversion and sintering technique at various sintering temperature ranging from 1000 ℃ to 1300 ℃. The strength of the prepared composite membrane will be characterized in term of mechanical strength using 3-point bending strength. Later, the performance of the composite membrane will be tested through permeability test.

2. Materials and methods.

2.1. Materials.

Cow bone hydroxyapatite (HAp) were obtained from previous work [14] and kaolin were purchased from Kaolin Malaysia Sdn Bhd. N-methyl-2-pyroolidone (NMP) (HPLC grade, Rathbone), polyethersulfone (PESf, Radel, A-300, Ameco Performance, USA) and Poyethyleneglycol 30-dipolyhydroxystearate (Arlacel P135) were used in this study. They were used as solvent, polymer binder and dispersant. Tap water was also used as the bore fluid and coagulant bath.

2.2. Fabrication of hollow fibre membrane.

Prior to preparing dope suspension, the pre-milled kaolin and HAp powder was sieved through a 36 μm sieve and dried in the laboratory oven to expel the moisture. The composition of the suspension was tabulated in the Table 1. The spinning parameters were 10mL/min, 10 ml/min and 5 cm for bore fluid flow rate, extrusion rate and distance of air gap. The dope suspensions were done by dissolving poyethyleneglycol 30-dipolyhydroxystearate (Arlacel P135) that act as dispersant into N-methyl-2-pyroolidone (NMP) (HPLC grade, Rathbone) solvent so that the ceramic powder can be added. The suspensions were ball milled by using NQM-2 planetary ball mill at 190 rpm for 48 hours. Later, polyethersulfone (PESf, Radal A-300, Ameco Performance, USA) was added into the suspension as polymer and the suspensions were ball milled for next 48 hours [14].

Table 1. The composition for membrane fabrication.

Composition	Hydroxyapatite based cow bone (wt.%)	Kaolin (wt.%)	NMP	PESf	Arlacel
A	20	20	54	5	1

Before the suspensions undergo the phase inversion process, the dope suspensions were degassed for one hour to remove the air bubbles that trapped in the suspensions. This process is vital to make sure that there is no defect in the formation of pore on the membrane structure. Then, the dope suspensions were extruded using a syringe pump via a tubular orifice spinneret. In order to complete the phase inversion technique, the membrane that passed through the air-gap distance were immersed in a coagulant bath for 24 hours. After that, the fibre membrane precursors were dried at room temperature at least for one day. Then, the fibre membrane precursors were sintered at temperature of 1000 °C, 1100 °C, 1200 °C and 1300 °C.

2.3. Characterization and performance of ceramic hollow fibre membrane derived from hydroxyapatite cow bone and kaolin.

By using a Hitachi Model TM 3000 scanning electron microscopy (SEM) at 3000μm magnification, the morphology of the hollow fibre membrane were analyzed. The hollow fibre membranes' mechanical strength were assessed via a three-point bending test. To determine the force with which the membrane split, an Instron Model 3342 tensile tester was used. The bending strength was gained by using equation:

$$\sigma F = \frac{8FLD_o}{\pi(D_o{}^4 - D_i{}^4)} \quad (1)$$

where F is the measured load at which split occurred (N), while L, D_o and D_i are the length of the membrane, the outer and inner diameters (m).

A cross- flow filtration system was developed to determine the pure water flux (PWF) of the hollow fibre membrane derived from hydroxyapatite based cow bone and kaolin. The performance of water flux was operated at room temperature with 1 bar pressure. The pure water flux was calculated by using equation:

$$J = \frac{V}{A \times t} \quad (2)$$

Where V is the volume of permeate collected (L), A is the surface area of the membrane (m^2) and t is the permeation time (s).

3. Results and discussion

The SEM images of the hollow fibre ceramic membrane at different sintering temperature are shown in Figure 1. The sintering temperature ranged from 1000 °C (A1), 1100 °C (A2), 1200 °C (A3) and 1300 °C (A4) at magnification 3000μm. From Figure 1, hollow fibre ceramic membrane from sintering temperature from 1000 °C (A1) to 1200 °C (A3) shows a finger like and microvoid look like. However, at sintering temperature 1300 °C (A4), the SEM images shows that the growth of grain of the ceramic has been happened. The densification of the hollow fiber ceramic membrane is significantly observed along with the increase in sintering temperature (1300 °C), as shown in Figure 1. This phenomenon is deeper in cross-sectional micrograph because the ceramic particles were discovered combine to one another, resulting in the formation of larger grains at high temperature. In addition, the densified hollow fibre ceramic membrane sintered at higher temperature had a larger void due to the interparticle space remaining after phase of grain growing. These voids will gradually create the membrane's dead-end pores or tunnels [15]. Similar results have been recorded in many studies that study the effect of sintering temperature on the properties of physicochemical of ceramic membranes [16][17]. The densification of the hollow fibre ceramic membrane may have an effect on the performance of the membrane in terms of water permeability and mechanical strength, rather than just its physical appearance [15]. From the SEM images, it is possible to evaluate that the increase in sintering temperature caused the difference in the superficial aspects of the ceramic membrane [18].

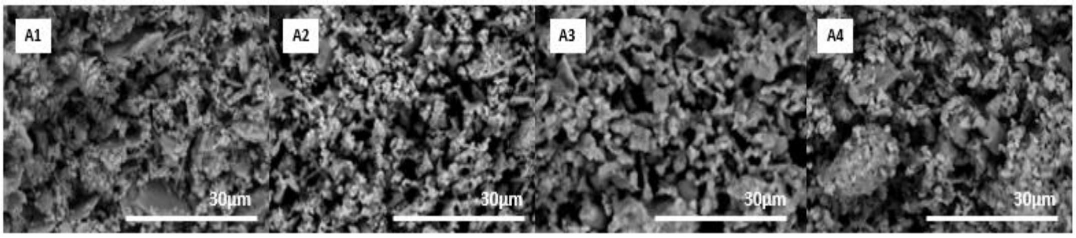

Figure 1: The SEM images for composite hollow fiber membrane derived from hydroxyapatite cow bone and kaolin at different sintering temperature.

The mechanical strength of the membrane that sintered at sintering temperature 1000 ℃ to 1300 ℃ was investigated and the results are shown in Figure 2. The connection clearly shows that the membrane's mechanical strength is primarily influenced by the sintering temperature. Hubadillah et al. [14] stated that the increase the membrane sintering temperature will result in the increase of mechanical strength. It can be seen that the increase in sintering temperature from 1000 ℃ to 1200 ℃ led to the slight increase in mechanical strength in the average from 11.12 Mpa to 13.33 Mpa. The hollow fibre ceramic membrane sintered at 1200 ℃ showed the highest mechanical strength because the maximum neck growth between the grains. Jamalludin et al. [19] stated that the formation of necks between the particles increased the membrane mechanical strength. However, when the sintering temperature was increased to 1300 ℃, the mechanical strength of the membrane decrease significantly to 11.42 MPa. This situation happened due to the changes of structures that forms in micron size during densification process of the membranes particles. This similar trend was also reported in Hubadillah et al. [14] that studied on hydroxyapatite based ceramic hollow fibre membrane derived from waste cow bone for textile wastewater treatment.

The water flux performance of sintered hollow fibre ceramic membrane at different temperatures has been tested. The water flux of the membrane is a key membrane operating parameter by dividing the permeate flow by total surface area of the membrane at the appropriate time, as shown in Figure 3. The membrane permeate flux during the 120-minutes output test are depicted in Figure 3. The membrane's water permeability is highly influenced by the sintering temperature. Both water mechanical strength and water permeation flux are inversely proportional. Membrane that have higher mechanical strength might due to the structure around the membrane cross-section that are denser. The tightly closely-packed particle, on the other hand, will gradually block water penetration through the membrane, reducing water permeation [2]. The ceramic membrane's general concept are when the sintering temperature increased, the water flux should be decreased [20]. This effect of the membrane's water permeability as the sintering temperature rises is a natural occurrence in the fabrication of ceramic membranes. This situation can be seen at Figure 3 at the water flux of hollow fibre ceramic membrane sintered at 1000 ℃ to 1200 ℃ (275.1 L/m^{2}h to 140.6 L/m^{2}h). However, the hollow fibre ceramic membrane at 1300 ℃ was not in the sequential pattern as its water flux is slightly increase (176.2 L/m^{2}h) than the temperature at 1200℃. This situation might due to the densification effect to the membranes particle as mention in paragraph 1. The membrane sintered at 1200 ℃ showed the most stable flux (140.6 L/m^{2}h) among all of the membranes.

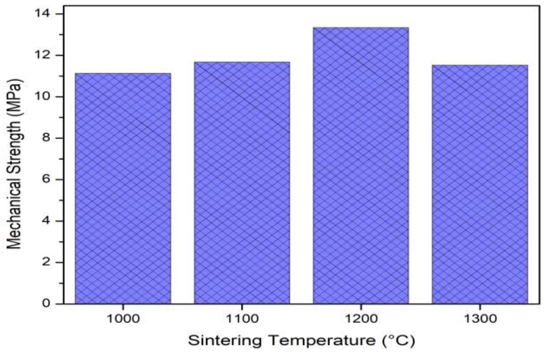

Figure 2: Mechanical strength of the ceramic membrane at different sintering temperatures.

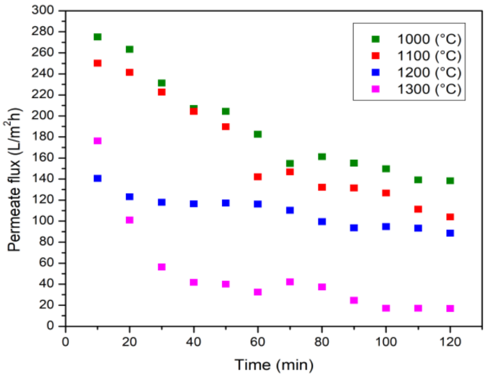

Figure 3: Permeate flux of the ceramic membrane at different sintering temperatures.

4. Conclusions

In this study, the composite hollow fibre membrane derived from hydroxyapatite cow bone and kaolin were successfully fabricated using both phase inversion and sintering process at different sintering temperature (1000 °C to 1300 °C). In fact, the structure of the membrane, mechanical strength and pure water flux are all affected by the sintering temperature of the membrane. The effect of sintering temperature on the hollow fibre ceramic membrane's physicochemical properties was investigated and it was discovered that 1200 °C was the best temperature for producing the best performance membrane, with 13.33 Mpa of mechanical strength and 140.6 L/m^{2}h of water permeability, respectively.

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