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Ceramic Membrane Development and Characterization for Microfiltration

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Abstract. The preparation and characterization of tubular ceramic membranes aiming to do microfiltration using alumina and kaolin powders as raw materials were carried out. The membranes were obtained by doing slip casting of the suspensions. The suspensions consisted of alumina-kaolin powders, water, and 0.9 wt.% of dolapix CE64 as deflocculant. The variations made during suspension preparation includes the use of different ratios between alumina-kaolin powder and distilled water of 60 wt.% : 40 wt.% and 65 wt.% : 35 wt.%, and also the use of addition of different percentage of starch of 0.5, 1, 2, and 3 wt.% as pore former into the suspension. Upon casting, the manufacture was then followed by sintering process in a furnace, which was also varied into up to 1000ºC, 1100ºC, and 1200ºC. The procedure was then completed by doing subsequent characterizations of the sintered membranes by determining their microstructure using SEM (Scanning Electron Microscopy) and pure water permeability and E. coli bacteria rejection experiment with McFarland turbid solution that were executed in a customized microfiltration module. All of the membrane samples succeeded to have 100% E. coli rejection. However, out of all the membrane samples, membrane with powder to distilled water ratio of 65 wt.% : 35 wt.% with 0.9 wt.% dolapix sintered at 1100ºC gave results to the highest water permeate flux.

INTRODUCTION

Unit operation which applies membrane processes has become more popular in a variety of separations and filtrations in industries (Sh. Akbarnezhad, Mousavi, & Sarhaddi, 2010). One of the possible materials to be used in membrane fabrication is polymer which has been commercially used in membrane applications due to its inexpensive price with sufficient performance for some extent of applications (Perry & Green, 1997). Another potential material to be used to produce membrane is ceramic. Ceramic is known to have several advantages over polymeric membranes which include thermal stability, high resistance to harsh chemical conditions, high mechanical strength, high separation efficiency, and also long lifetime (Sh. Akbarnezhad et al., 2010 & Lorente-Ayza et al., 2015).

However, research and utilization of ceramic membranes, especially in Indonesia, is very limited or undeveloped whereas the resources of the materials are abundant. And not only ceramics, the manufacture of commercial membranes, which are usually made of polymer, does not exist in Indonesia. These commercial membranes are still imported. On the other hand, Indonesia is very rich in mineral resources, and one of them is bauxite, a type of rock consisting alumina around 45%-65% alumina, 12% silica and more than 3% titania, which are common materials for ceramic membranes (Kementrian Energi Dan Sumber Daya Mineral, 2012). In 2010, Indonesia was recorded to have around 726,585,010 tons of bauxite whereas the reserve was around 179,503,546 tons. During the same year, Indonesia was also recorded as the seventh largest producer of bauxite with total amount of production of 10.28 million tons (Qomaruddin & Ferdika, 2016). Due to its local high resource and production rate of bauxite, the usage of alumina as a material for the development and the characterization of ceramic membranes will be discussed.

Although the resources of the raw materials are abundant, ceramic membrane fabrication still remains expensive. This is because of the high cost it takes to extract and purify alumina from the bauxite ore to produce alumina powder

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used in the ceramic membrane fabrication. Therefore, this research will also address the use of kaolin as natural material that is cheaper to be mixed with alumina powder in order to minimize the cost since kaolin is believed to be an efficient alternative solution in extent to low cost wastewater treatment application at which is related to the objective of this work. (Rekik, Bouaziz, Deratani, & Baklouti, 2017). Kaolin itself is a type of clay mineral which is also existence is also available abundantly in Indonesia.

Ceramic membranes can also be applied in separation processes which require micro- and ultrafiltration. In this research, the fabrication is aimed to be applicable for microfiltration separation process. This is due to wide use of microfiltration in water and wastewater treatment, as well as purification (Amin, Abdallah, Roushdy, & El-Sherbiny, 2016). This corresponds with many existing local environmental problems that require this separation process.

One of the existing problems is the limited availability of clean water for drinking, sanitation, and hygiene. One of the real cases is the existence of E. coli bacteria in the drinking water in Yogyakarta. In 2015, it was recorded that 89% of the total surveyed household drinking water in Yogyakarta still contained E. coli bacteria (Saroh, 2016). This condition obviously did not meet the requirement for drinking water that was regulated by Regulation of Indonesian Ministry of Health No. 32 in 2017 in which was stated that there should be 0 CFU/ml concentration of E. coli. The same condition also applies in water for sanitation and hygiene purposes (Menteri Kesehatan Republik Indonesia, 2017). This clean water issue is only in one of the many places in Indonesia. One of the bacteria removal ways is by boiling the water. However, this requires heat, and by heat means energy. With the use of ceramic microfiltration membranes, as what would be developed in this research, there will be no heat energy required as it is expected that ceramic membranes could E. coli bacteria so then it could be the answer for fulfilling the water quality standard regulated by Indonesian Ministry of Health.

Ceramic microfiltration membrane can also be used in purification process, it has the potential to be used in biodiesel production for the removal of impurities (Saleh, 2011). This is due to its high chemical and organic solvents resistance, especially over polymeric microfiltration membranes. It has also the potential to be used for treating wastewater containing chemicals from chemical industries and oily wastewater from industries with highly chemical resistant technologies needed that also exist in Indonesia.

Therefore, the purpose of this study was to develop tubular ceramic microfiltration membranes with alumina and kaolin as raw materials. This research also intended to study the effects of concentration of deflocculant, pore former, different heating rates of sintering process, and different ratio of raw materials during the ceramic membrane manufacture. The study was followed by the characterization of the membranes based on their microstructure, porosity and pore size. Last but not least, the performance of the membranes as microfiltration membranes was studied based on their permeability and bacteria percentage rejection.

EXPERIMENTAL PROCEDURE

Materials

Alumina (α-Al_2O_3) powder A12 and kaolin powder (Bintang Dunia, Bangka, Indonesia) and distilled water were used as the main raw materials of the membranes in this study. Alumina grinding balls were used to grind the ceramic powders to get the desired slip suspension suitable for slip casting. Dolapix CE64 (Zschimmer & Schwarz GmbH Co., Germany) was used as deflocculant. Starch (pro analysi, Merck, Germany) was used as pore former. Gypsum was used for making the handmade tubular mould for casting the membranes. $BaCl_2$ 99% (BDH, England) and H_2SO_4 (Mallinckrodt Baker, USA) were used for making McFarland turbid solution as the representation of certain concentration of E. coli cells used for bacteria rejection experiment.

Preparation of Alumina-Kaolin Suspension

Concentration Determination of Dolapix CE64 as Deflocculant

Different concentrations – 0.7, 0.9, 1.1, 1.3, and 1.5 wt.% of dolapix CE64 as deflocculant were examined in the alumina-kaolin milled suspension. The right amount of deflocculant will indicate the lowest viscosity of the suspension. Raw materials for the suspension were prepared; alumina powder, kaolin powder, and water with ratio of 4:2:5. The ratio of dry power, alumina and kaolin, always remained to be 2:1. The raw materials were then milled for 8 hours in a pot mill with alumina grinding balls that were prepared to be 1:1 to the total dry powders. The milled

suspension was then dried in a drying oven at 110°C for 20 hours until the mass of the dried milled suspension was constant. The dried milled suspension was then sieved with 40 mesh sieve and was mixed with water (65wt.% : 35 wt.%). Different concentrations of dolapix CE64 was then mixed until homogeneous suspension was obtained. The viscosities of each of these suspensions containing different concentrations of deflocculant were then measured using flow cup viscometer.

Concentration Determination of Starch as Pore Former

Different concentrations of starch as pore former were also examined in the alumina-kaolin suspension with the same purpose of determining the lowest viscosity of suspension. The procedure was the same as determining dolapix CE64 concentration previously with the starch concentrations varied into 0.5, 1, 2, and 3 wt.% based on the dry milled suspension powder.

Preparation of Tubular Mould

The tubular mould used for casting the membranes was first prepared by mixing gypsum and water until a homogeneous suspension was obtained. The procedure was then followed by lathing the suspension into a solid cylinder with a dimeter of 11 mm and length of 110 mm, which took around 10 hours until a cylinder model was obtained. Subsequently, the membrane mould was made using the same gypsum suspension. The cylinder model was then used to make the model hollow. The hollow mould was then put into a furnace for drying for around 8 hours at 1300°C. The furnace that was used works by using isolate insulating firebrick as it is specifically designed for drying ceramics. Finally, the mould was then taken out of the furnace and cooled until it reached room temperature and was ready to be used for slip casting of the membrane suspensions.

Preparation of Tubular Ceramic Membrane

Alumina and kaolin powder were prepared with ratio of 2:1. The alumina-kaolin powder was then mixed with water with ratios that were varied as follows; 65 wt.% alumina kaolin powder in 35 wt.% water and 60 wt.% alumina kaolin powder in 40 wt.% water. The ceramic powders and water were then mixed in a pot mill for 8 hours with alumina grinding balls of which the amount was 1:1 to the powders. The procedure was then followed by the addition of the determined concentration of dolapix CE64 (that indicated the lowest viscosity in the previous pre-experiment in 2.2.1) into the suspension. Starch was also added into the suspension with all of the concentrations explained in 2.2.2 in order to see the difference in the characteristics, although only one concentration indicated the lowest viscosity of the suspension. The suspension was then casted in the tubular mould using slip casting method, where the suspension was poured into the mould, allowing it to stay in there for certain period of time. The period of slip casting was varied into 10, 20, 30, 40, 60, and 120 s. Subsequently, the casted suspension was then dried in ambient overnight and in the oven at 60°C for 20 hours. The dried casted suspension was then sintered in a furnace up to elevated temperature that was also varied into to 1000°C, 1100°C, and 1200°C. The setup of each of sintering temperature is explained as follows. The example of the setup of the sintering process at 1000°C was described in Table 1 below. The rates were applied in the same way for 1100°C and 1200°C.

Table 1. Sintering process setup example for 1000°C.

Temperature (°C)	Heating Rate	Time
25 - 800	2°C/min	6 hour 30 min
800 - 1000	5°C/min	40 min
1000	Holding	2 hour
1000 - 40	2.4°C/min	6 hour 40 min
Total Sintering Time		**9 hour 10 min**

Membrane Characterization using SEM

The microstructure of the fabricated tubular ceramic membranes was then studied using scanning electron microscopy (SEM). The membrane pore size and particle size were also estimated using SEM.

Microfiltration Experiment

After characterized using SEM, the membranes were then studied for its performance as microfiltration membranes. The study included permeation experiment in the self-assembled microfiltration module using pure water and simulated wastewater flowing through the membranes with pressure of 0.3 kg/cm^2. On the other hand, by flowing simulated wastewater, the amount of bacteria to be rejected or the bacteria rejection were also able to be determined.

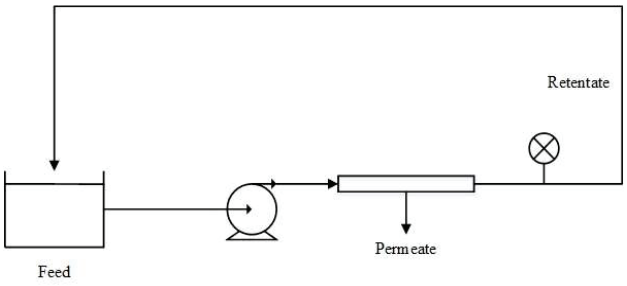

Figure 1. Flow diagram of microfiltration experiment.

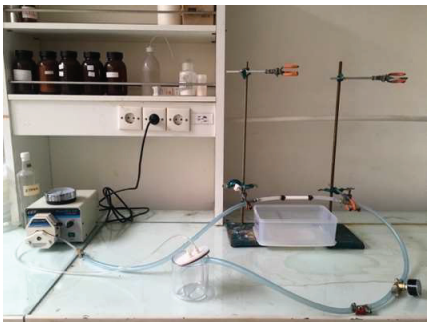

Figure 2. Self-assembled microfiltration module.

Permeate Flux Calculation

Permeate flux of pure water and simulated wastewater was calculated using the following equation.

$$J = \frac{V \cdot d}{A \cdot \Delta t}$$

Where, J = permeate flux (L.mm/m^{2}h), V = volume of permeate (l), d = membrane thickness (mm), A = effective membrane area (m^2), and Δt = time (h). The permeate flux is multiplied by the membrane thickness in order to make all membranes comparable to each other, because the use of slip casting method results to uncontrollable membrane thickness for each membrane containing different suspensions.

Bacteria Percentage Rejection Calculation

In this experiment, the bacteria percentage rejection of the membrane was determined by flowing 1 M McFarland solution as simulated wastewater through the membrane, which represented 300 x 10^6 CFU/ml concentration of E. coli bacteria in the feed. The absorbance of the permeate and the turbid feed was then measured in a spectrophotometer, so then the represented concentrations of E. coli bacteria could be determined. The bacteria percentage rejection was then calculated using the following equation, where % R = Percentage of rejection, C_p = Number of bacteria in permeate (CFU/ml), and C_f = Number of bacteria in feed (CFU/ml).

$$\% R = \left(1 - \frac{C_P}{C_f}\right) \times 100\%$$

E. Coli McFarland Standard Solution Preparation

McFarland standard solution was used to approximate bacteria concentration in a suspension. This solution was used as feed solution for bacteria percentage rejection experiment in the self-assembled microfiltration module. The standards were made by preparing 1% solution of barium chloride and 1% solution of sulfuric acid, and mix them in the proportions exhibited in the table below. Every solution representing all of the scales bellow were then examined in spectrophotometer in order to determine the absorbance each of them represents.

Table 2. McFarland turbidity standard.

McFarland Scale	CFU ($x10^6$/ml)	1% $BaCl_2$/ 1% H_2SO_4 (ml)
0.5	<300	0.05/9.95
1	300	0.1/9.9
2	600	0.2/9.8
3	900	0.3/9.7
4	1200	0.4/9.6
5	1500	0.5/9.5
6	1800	0.6/9.4
7	2100	0.7/9.3
8	2400	0.8/9.2
9	2700	0.9/9.1
10	3000	1.0/9.0

RESULTS AND DISCUSSION

Preparation of Alumina-Kaolin Suspension

Concentration Determination of Dolapix CE64 as Deflocculant

From the pre-experiment, it was shown that the 0.9 wt.% dolapix CE64 indicated the lowest viscosity as an optimum deflocculant concentration compared to the other suspension with different dispersant concentrations at room temperature. This minimum viscosity relates to the best dispersion of the alumina-kaolin suspension, indicating that there is no specific flocculation of the raw material powders. Therefore, it is really suitable for slip casting, the casting method that is employed in this research.

Among other concentrations tested in the alumina-kaolin suspension, 1 wt.% starch indicated the minimum viscosity. From the graph below, it can be seen that the addition of starch, even at 1 wt.% concentration, gave results to higher viscosities of the overall suspensions. This happened to be due to the insolubility of starch in water.

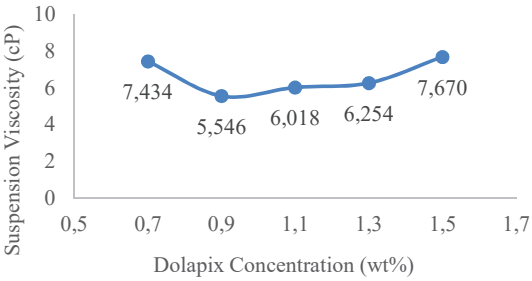

Figure 3. Suspension viscosity vs. dolapix CE64 concentration.

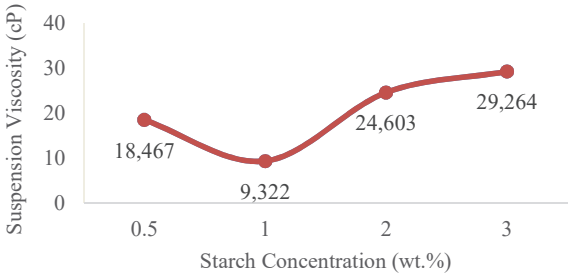

Figure 4. Suspension viscosity vs. starch concentration.

Preparation of Tubular Ceramic Membrane

In this research, there were numbers of parameter changes in preparing membrane supports which resulted to several membrane samples shown in the tables below. Table 3 shows different samples consisted of only alumina-kaolin powder, water, and dolapix CE64 without the addition of starch. These samples differ in the ratio between alumina-kaolin powder and water, with dolapix CE64 concentration remained the same. Sample A and B contained more powder and less water ratio in the suspension they were casted from, resulting to a more viscous suspension of 6.313 cP compared to the suspension of sample C were casted from having only 1.77 cP suspension viscosity. On the other hand, table 4 shows different suspension viscosities along with the addition of starch as pore former. Although there were rather significant differences in the viscosities of each of the suspensions, all of these suspensions were all casted in the handmade tubular moulds as they were still thin enough to implement the slip casting method, which is why all of the membranes having starch in their suspensions were also characterized in this study.

Table 3. Specifications of raw materials, suspension viscosity, sintering of temperatures of membranes without percentage of starch

Membrane	Powder (wt.%)	Water (wt.%)	Dolapix CE64 *(wt.%)	Suspension Viscosity (cP)	Sintering Temp. (°C)
A	65	35	0.9	6.313	1000
B	65	35	0.9		1100
C	60	40	0.9	1.77	1100
D	60	40	0.9		1200

Table 4. Specifications of raw materials, suspension viscosity, sintering of temperatures of membranes with varied percentage of starch.

Membrane	Powder (wt.%)	Water (wt.%)	Dolapix CE64 *(wt.%)	Starch *(wt.%)	Suspension Viscosity (cP)	Sintering Temp. (°C)
E	65	35	0.9	0,5	18.467	1100
F	65	35	0.9	1	9.322	1100
G	65	35	0.9	2	24.603	1100
H	65	35	0.9	3	29.264	1100

*weight percentage of dolapix CE64 and starch is based on the alumina-kaolin dry powder.

Characteristics of the Membranes

Figure 5 below shows the SEM pictures using topography or ETD contrast of the inner surfaces of membrane B, C and G with 100 times magnification. The lighter the color indicates a higher structure in the membrane. From the figure, it can be seen the differences between the inner surface structure of each of the membranes is not significant. The inner surfaces are not really homogeneous topographically with the darker and lighter parts throughout the surfaces. In membrane B, pores clearly can be seen especially in the right part of the image. Membrane C looks a bit more structured than of the other membranes and also denser. On the other hand, membrane G with some percentage of starch, also shows the presence of pores although not much and not structured. However, there are two large dark parts in its surface, indicating the existence of holes or lower surface of the membrane. These are assumed to be resulted from the error in preparing the membrane suspension, specifically due to the inhomogeneous stirring of suspension after the addition of starch.

Figure 6 shows the SEM images of the inner surfaces of the membranes with 100 times magnification using VCD or material contrast that enables us to have some hints of the materials or atoms consisted in the membrane. The lighter color in the images shows a heavier atom in the membrane sample. The distribution of the materials in membrane B seems inhomogeneous as there are some darker parts in the left side and in the lower part of the image. In membrane C, the distribution of colors seems to be more homogeneous than of the other two membranes despite a little darker parts in the left part of the membrane surface image. However, this shows that the membrane lacks pores as they can be barely seen throughout the image. Meanwhile, in membrane G, the existence of holes can clearly be seen in the SEM image. This means that these parts lack of materials or consist of lighter atoms so that they are darker than the other parts.

On the other, figure 7 shows the cross sections of the membranes using material contrast with 1000 times magnification with an estimation about the particle and pore size. The structure of membrane B seems inhomogeneous as the distribution particles are not the same. It can be clearly seen that there are some large particles with around 11 – 12 μm size in the middle of the image that seem to be really close to each other, forming an aggregates, and barely have space or pores in between. However, inhomogeneous distribution of pores is formed in the right and left parts of the images, with the size of around 2 – 4 μm. On the other hand, the cross section picture of membrane C looks a bit more structured, with the approximation of particle sizes of around 2 – 5 μm. The pores of this membrane seems smaller than of the other two membranes, with around 1 – 2 μm estimated pore sizes, which verifies the previous figures 5 and 6 that shows that membrane C looks denser and lacks of pores than the others. Moreover, the cross section image of membrane G shows also an uneven distribution of both pore and particle sizes. The addition of starch in this membrane does not show any significant difference in the structure of the membrane, especially the pore sizes. The pore sizes are seemed to be more or less the same with of membrane B. Some big holes can also be seen in the right part of the image.

Based on all SEM images below, it can be concluded that the ratio between alumina-kaolin powder and water of 60 wt.% : 40 wt.% resulted to a rather more structured and evenly distributed membrane pores and particle sizes if seen using both material and topography contrasts, as shown in membrane C, compared to the other two membranes, which contained the ratio between powder and water of 65 wt.% : 35 wt.%. However, membrane C seemed to have

rather distinguishably smaller pore sizes rather than the other two SEM-characterized membranes, which would strongly affect the permeate flux to be lower than the others.

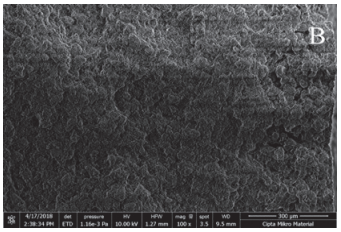

Figure 5. SEM images of the inner surfaces of membrane B, C, and G (topography contrast)

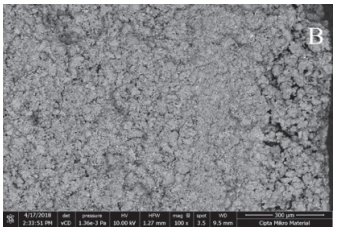

Figure 6. SEM images of the inner surfaces of membrane B, C, and G (material contrast)

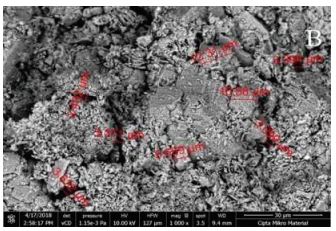

Figure 7. SEM images of the cross-sections of membrane B, C, and G (material contrast)

Microfiltration Permeation Experiment Results

Permeate Flux Results

The permeate flux of the membranes were then measured using pure water in the microfiltration module. The experiment was executed 90 – 120 minutes until each of the membrane fluxes are constant and by collecting the amount of water in the permeate for 1 minute every 5 minute. Since all of the membranes had different thicknesses due to the use of slip casting as a casting method, the permeate fluxes were multiplied by each of the membrane thicknesses that are expressed in mm, so then the unit was expressed as L.mm/m^2h.

Permeate Flux of Membranes without Starch

Figure 8 shows the permeate fluxes of membrane A and B which contained the same raw materials composition of 65 wt.% alumina-kaolin powder, 35 wt.% water, and 0.9 wt.% dolapix CE64, but sintered at different sintering temperature. Membrane A was sintered at 1000ºC, while membrane B implemented at 1100ºC. It can be seen that higher pure water permeate flux was obtained by membrane B that was sintered at higher sintering temperature compared to membrane A with lower sintering temperature. This conclusion was also confirmed by looking at the next graph, Figure 9.

Figure 9 shows the comparison between the pure water permeate fluxes of membrane C and D which consisted of the same raw materials compositions of 60 wt.% powder, 40 wt.% water, and 0.9 wt.% dolapix CE64. However, the sintering temperature of these two membranes differed, with membrane C sintered at 1100°C and membrane D at 1200°C. The graph also indicates that with the respective particular membrane composition, higher sintering temperature resulted to a higher membrane permeate flux.

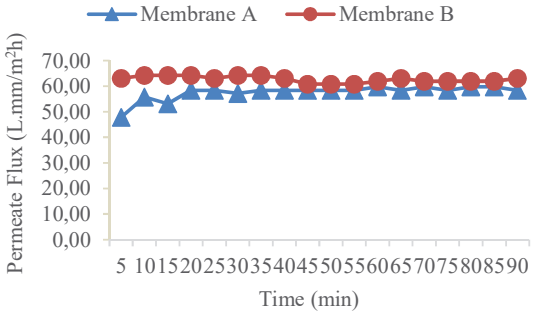

Figure 8. Effect of different sintering temperatures on membranes with compositions of 65 wt.% powder : 35 wt.% water : 0.9 wt% dolapix CE64

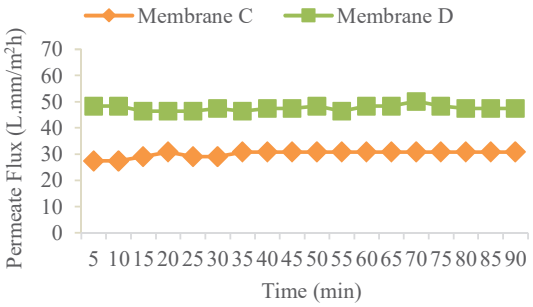

Figure 9. Effect of different sintering temperatures of membranes with compositions of 60 wt.% powder : 40 wt.% water : 0.9 wt% dolapix CE64 on the water permeate flux.

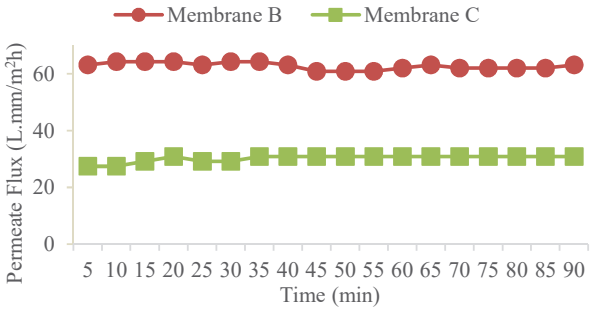

Figure 10. Effect of different ratio between alumina-kaolin powder and water on the water permeate flux (sintered at 1100°C)

Another thing that can be observed by comparing Figure 8 and 9 is that the membranes with raw materials composition of 65 wt.% : 35 wt.% : 0.9 wt.% had a higher pure water permeate fluxes, compared to the membranes having raw materials composition of 60 wt.% : 40 wt.% : 0.9 wt.%. This observation was approved by comparing membrane B and C with the respective difference in raw materials composition, however sintered at the same sintering temperature of 1100°C. The membrane fluxes of membrane B and C is described in Figure 10.

Comparing the permeate flux data from the three graphs above, it can be seen that membrane B that was made of suspension containing 65 wt.% powder, 35 wt.% water, and 0.9 wt.% dolapix CE64 and was sintered at 1100°C, had the highest pure water permeate flux compared to other three membranes.

Permeate Flux of Membranes with Starch

As can be seen from Figure 11, the permeate fluxes of the membranes roughly keep decreasing as more percentage of starch was added into the membrane suspension for membrane E, F, and G, with membrane E that contained only 0.5 wt.% starch having around 45 L.mm/m^{2}h permeate flux while membrane F that contained 1 wt.% starch produced more or less the same flux as membrane E. And in membrane G with 2 wt.% starch, the permeate flux seemed to decrease again to around 38 L.mm/m^{2}h. However, the decrease in the permeate flux along with the addition of more

percentage of starch seemed to be in contrast with the permeate flux of membrane H that contained 3 wt.% starch, which produced almost 60 L.mm/m^{2}h permeate flux.

These results were then compared with the permeate flux of membrane B that was made of the same suspension consisting of 65 wt. % alumina-kaolin powder, 35 wt.% water, and 0.9 wt.% defflocculant that was sintered at the same temperature of 1100^{o}C, yet contained no starch in its suspension. It can be seen that membrane B had a distinguishably bigger permeate flux than the other membranes that contained starch. This is in contrast with the aim of using starch as pore former in the first place in this research to create a bigger permeate flux in the membranes.

The highest permeate flux of the membrane B out of all these membrane samples, however, is still relatively low compared to the range of permeate fluxes of ceramic membranes from the previous research papers or journals which are calculated to be around 150 – 350 L/m^{2}h if applies the same pressure as in this research (0.3 kg/cm^2). This is caused by the membrane thicknesses of the membranes in this research that are too thick with relatively small estimated pore sizes based on SEM of only around 1 – 4 µm.

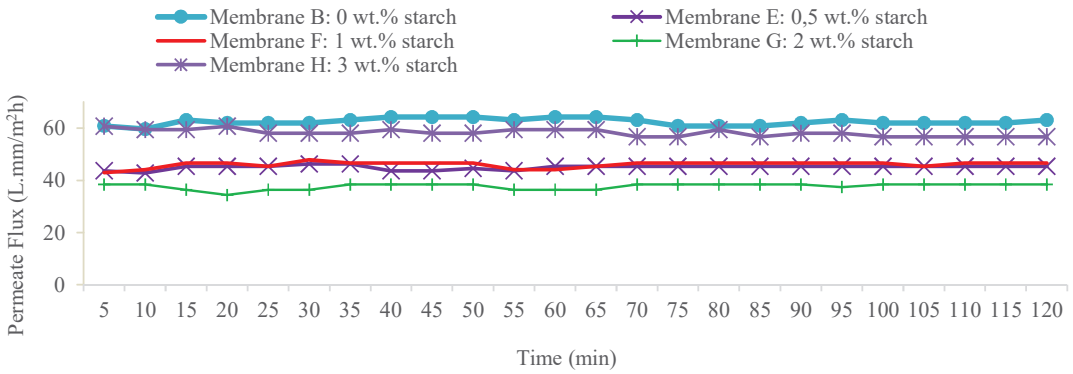

Figure 11. Effect of different ratio of starch in the membranes on the water permeate flux (and the comparison with the membrane without starch)

Bacteria Rejection Results

E. coli McFarland Standard Solution Preparation Results

The linear regression of the absorbance of all of the concentrations of McFarland standard solution from table 2 is $y = 0.1402x + 0.0544$ and $R^2 = 0.99768$.

E. coli Bacteria Rejection Results

Although all of the solutions were made with the same procedure for making 1 M McFarland turbidity standard, the absorbance measurement was regularly done for each of the membranes since the absorbance of each of the feed solutions might not be exactly the same as the E. Coli McFarland turbidity standard that was expressed in the linear regression where it is stated that 0.198 A is equal to the existence of E. coli bacteria of 300 x 10^6 CFU/ml, although undergoing the same procedure to make 1 M McFarland standard solution. The aim of doing this measurement was to do the calculation for the bacteria rejection more precisely for each of the membranes.

After filtration experiments, the bacteria concentrations in all of the membranes permeate resulted to be 0. The calculation example for bacteria rejection of membrane A is shown above, resulting to 100% E. Coli bacteria to be rejected. The calculation also applies in the other membranes. Therefore, by having feed solutions containing more or less 300 x 10^6 CFU/ml E. coli bacteria, all of the eight membranes were able to reject them all, resulting to 100% E. coli bacteria rejection.

Table 5. Bacteria concentration before and after filtration

Membrane	Bacteria Concentration (CFU x 10^6/ml)		Bacteria Rejection Rate	Average Permeate Flux (L.mm/m^2h)
	Feed (C_f)	Permeate (C_p)		
A	275	0	100%	57.59
B	284	0	100%	62.40
C	284	0	100%	30.15
D	292	0	100%	47.57
E	267	0	100%	45.01
F	301	0	100%	46.03
G	273	0	100%	37.69
H	279	0	100%	58.17

$$R_{membrane\ A} = \left(1 - \frac{C_p}{C_f}\right) \times 100\% = \left(1 - \frac{0}{275 \times 10^6}\right) \times 100\% = 100\%$$

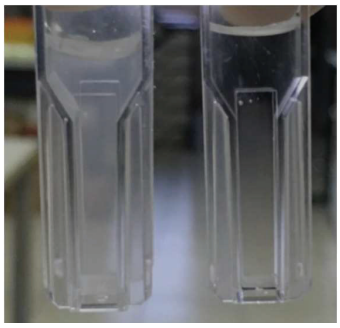

Figure 12. Permeate before (left) and after (right) filtration.

Fouling was not likely to happen during the experiment because the concentration of bacteria in the feed is relatively still low. Also, E. coli bacteria is usually rode-shaped (usually around 800 nm wide and 2.5 μm long) (Takeuchi, DiLuzio, Weibel, & Whitesides, 2005), so then they could still be retained by the membranes although estimated to have pore sizes ranging between 1 – 4 μm as mentioned. Other reason is that the pore size is only an approximation based on SEM, which may be different than the actual effective pore size. It is also possible that the actual effective pore size of the membranes is smaller than of what was observed using SEM.

CONCLUSIONS

Ceramic membranes manufacture by slip casting method with alumina and kaolin with ratio of 2:1 as raw materials succeeded to be developed in this work with the use of 0.9 wt.% dolapix CE64 as deflocculant to perform a stable suspension ideal for slip casting. The study examined the use of different ratio between alumina-kaolin powder and water in the membranes sintered at the same temperature of 1100ºC, where the membrane with powder to water ratio of 65 wt.% : 35 wt.% produced almost two times bigger water permeate flux of around 30 l.mm/m^2h than of the membrane with 60 wt.% : 40 wt.%. These permeate flux results were confirmed by the SEM pictures of these two membranes, showing the denser morphology of the membrane with lower permeate flux, with 1 – 2 μm estimated pore sizes. Membrane with 65 wt.% : 35 wt.% was estimated to have bigger pore size of 2 – 4 μm. Subsequently, this research studied the effects of the implementation of different heating rates of sintering process at 1000ºC, 1100ºC, and 1200ºC which indicated that higher sintering temperature resulted to a higher membrane permeate flux. Addition of variety of concentrations of starch, as pore former, were also executed (0.5, 1, 2, and 3 wt.%) in the suspensions having 65 wt.% alumina-kaolin powder, 35 wt.%, water and 0.9 wt.% dolapix CE64 that sintered at 1100ºC, which resulted to a declination of water permeate flux along the addition of starch into the membrane suspensions. All of the membranes in this work were also examined for its ability to reject bacteria by using flowing 1 M McFarland turbid solution that equals to 300 x 10^6 CFU ml E. coli bacteria through the membranes. And all of the membrane samples with variations in their preparation procedure succeeded to reject all of the impurities in the solution, therefore having 100% E. coli bacteria percentage rejection. Based on all of the characterizations and microfiltration experiments carried out in each of the different membrane samples with variations made in their preparation, the membrane B that was prepared with ratio of alumina-kaolin powder, water, and dolapix CE64 of 65 wt.% : 35 wt.% : 0.9 wt.% without percentage of starch that was sintered at 1100ºC gave results to the highest water permeate flux of 30 L.mm/m^2h and 100% E. coli percentage rejection.

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