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Fabrication of natural ball clay ceramic membrane using pore former and additive agents based on modified slip casting technique

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ABSTRACT

Abundantly available natural ball clay can be an alternative to expensive ceramic material in fabricating ceramic membrane. The local natural Malaysian ball clay has been chosen as the main precursor for elaborating the ceramic membrane. Since additive agents and pore-forming agent are crucial in obtaining the desired properties and performances of the ceramic membrane, the objectives of this research is to study the effect of additive agents (PEG 600 and Dispex N40) at different sawdust percentage applied (0%–40%) in fabricating natural ball clay ceramic membrane. Flat rectangular ceramic membrane with a dimension of 9.0 ± 0.5 cm^2 in area and 2.0 ± 0.2 cm in thickness was obtained through a modified slip casting method, followed by sintering at 1,000°C for 2 h. The ceramic membrane properties were then characterized using scanning electron microscopy, Archimedes method, mechanical strength and water permeability test. From the results obtained, the fabricated ceramic membrane of CM2 in the presence and absence of additive agents shows improved properties of 7%–10% thermal shrinkage, 36–38% in porosity, 8–12 MPa in compressive strength and 40,000–70,000 Lh^{-1} m^{-2} bar^{-1} in water permeability.

Keywords: Natural ball clay; Ceramic membrane; Porosity, Compressive strength

1. Introduction

Membrane technology is a well-established environmental application since access to clean water has become one of the challenges for certain countries in this century and for future generations [1,2]. The membrane is a selective barrier during the membrane process in which when a feed stream is allowed to pass through the membrane, the components that can pass through the membrane are called permeate, while the components retained in the feed stream are called retentate [3]. Microfiltration, ultrafiltration and nanofiltration are membrane processes applied to water and wastewater treatment [4]. Organic membrane,

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especially polymeric, is highly utilized in the industrial area due to their lower cost, scalability and good separation characteristics. However, they are low in fouling resistance, have a shorter lifespan, low in temperature ranges and highly susceptible to corrosion [5,6]. Due to the advantages shown by a ceramic membrane, ceramic membranes have attracted much interest in the scientific community compared to the polymeric membrane. The main advantages of ceramic membranes are that they excel in chemical, mechanical and thermal resistance, high fouling resistance, high-pressure application and long lifespan [5–7]. Unfortunately, these membranes' expensive capital cost might be due to an expensive ceramic material, such as zirconia, alumina and silica [2,8]. Thus, the development of low-cost ceramic membrane based on some waste powder (e.g., fly ash), food powder (e.g., starch, wheat flour, cornflour, sago) and natural materials (e.g., clay mineral, phosphate, and apatite) were studied by several researchers [9–16].

Natural clays are in abundance and need a low firing temperature compared to metal oxide material in achieving optimum porosity and mechanical strength [2,17]. However, different raw materials or mineralogy of natural clay as precursors offers a different function and a different ceramic product quality. Additive agents such as a binder, dispersant, flux and filler are commonly used in ceramic fields to promote a better product. Binder acts as a strength provider to the supporting body by building bridges between particles. In some cases, it also provides the plasticity and assists the process of body forming [14,18,19]. Dispersant creates homogeneity or stable slurries [20]. Flux is used to reduce the firing temperature while the filler is applied to reduce the shrinkage of clay bodies [21].

The main objective of this study is to develop a flat ceramic membrane made up of natural clay known as ball clay abundantly found in Besut, Terengganu, Malaysia using a modified slip casting method. In the slip casting method, slurry (a mixture or solution in slip casting) is poured onto a microporous plaster of Paris (POP) mold. The porous nature of the mold gives a capillary suction pressure, which then draws the fluid from the slurry into the mold as depicted by Darcy's law. A consolidated layer of solid or also known as a cast, forms on the walls of the mold. After an adequate thickness of the cast is shaped, the surplus slip is poured out and then the mold and cast are allowed to dry. Moreover, during the consolidation of the drying stage, the wall thickness is difficult to control and is generally thick [3]. This modified slip casting method was developed to enhance the simple slip casting method in which pressure is applied after the slurry resembles a dough in order to achieve a uniform thickness of the ceramic membrane. In this modified slip casting method, a wooden block is used as a pressing force is applied 20 times for each sample. The different weight percentage of sawdust was used as a pore-forming agent and in the presence or absence of additive agents. The effect of varying percentage of sawdust addition in the presence or absence of additive agents on shrinkage, microstructure, porosity, density, mechanical strength and water permeability of ceramic membrane aims to identify the best fabrication of natural ball clay ceramic membrane for water filtration with a good compromise between porosity, mechanical strength and water permeation.

2. Materials and methods

2.1. Raw materials preparation

Natural ball clay was obtained from Kampung Dengir, Besut, 107 km from Kuala Terengganu, Terengganu, Malaysia. The raw natural clay's impurities like woods, small particles, stones and others were removed by sieving. The fine clay was then poured onto the POP mold and dried overnight in an oven at 80°C to absorb the water content in the clay. The stone clay was then ground into powder form and sieved using 180 μm. Sawdust, wastes from local furniture and sawmilling industries, has been chosen as a pore-forming agent and obtained from Bakti Malaysia Sdn. Bhd., Wakaf Tengah, Batu Rakit, Kuala Nerus, Terengganu. Prior to use, the sawdust was dried in the oven at 80°C for 2 d, then ground and followed by filtering through a 425 μm-sieve to obtain fine sawdust particles.

2.2. Membrane fabrication and characterization

2.2.1. Membrane fabrication

The flat ceramic membrane was made from natural ball clay, homogeneously mixed with sawdust (pore-forming agent), water and additive agents (PEG 600 and Dispex N40) and the paste formulation is described in Table 1. The powder ratio (ball clay and sawdust) to water used is 1:1. Polyethylene glycol (PEG 600) acts as a binder to provide bonds between the elements to induce higher mechanical strength. Sodium base (Dispex N40) functions as a dispersant to prevent the particles from agglomerate [20]. The natural ball clay ceramic membrane fabrication process is described in Fig. 1.

The sintering process directly affected the ceramic membrane's structure or known as shrinkage behavior. The thermal shrinkage was calculated by considering the dimensional length of the ceramic membrane before and after sintering in a furnace using vernier caliper [22] as shown in Eq. (1):

$$\text{Shrinkage}_{\text{firing}}, \% = \frac{\ell_1 - \ell_2}{\ell_1} \times 100\% \quad (1)$$

where ℓ_1 is the length of the ceramic membrane before firing and ℓ_2 is the length of the ceramic membrane after firing.

2.2.2. Characterization techniques

The characterization involved two parts; (1) raw material and (2) ceramic membrane. The thermal stability characterization of natural ball clay was analyzed using thermogravimetric analysis-differential thermogravimetry (TGA-DTG). Element present in natural ball clay using X-ray diffraction analysis was already mentioned in the previous work [20]. Characterization techniques of natural ball clay ceramic membrane involve morphological study by scanning electron microscopy (SEM), thermal shrinkage, porosity and density measurement and mechanical strength by the compression test. Tabletop scanning electron microscope (Hitachi TM-1000) analysis was performed to analyze the fired ceramic's membrane by overviewing

Table 1
Formulation of natural ball clay ceramic membrane

Type of natural ball clay ceramic membrane	Membrane materials (dry basis)		Additive agents		Water (mL)
	Natural ball clay (wt.%)	Sawdust (wt.%)	Dispex N40 (mL)	PEG 600 (mL)	
Absence of additive agents (A)					
CM1(A)	100	0	0.00	0.00	100.00
CM2(A)	90	10	0.00	0.00	100.00
CM3(A)	80	20	0.00	0.00	100.00
CM4(A)	70	30	0.00	0.00	100.00
CM5(A)	60	40	0.00	0.00	100.00
Presence of additive agents (B)					
CM1(B)	100	0	0.05	0.50	100.00
CM2(B)	90	10	0.05	0.50	100.00
CM3(B)	80	20	0.05	0.50	100.00
CM4(B)	70	30	0.05	0.50	100.00
CM5(B)	60	40	0.05	0.50	100.00

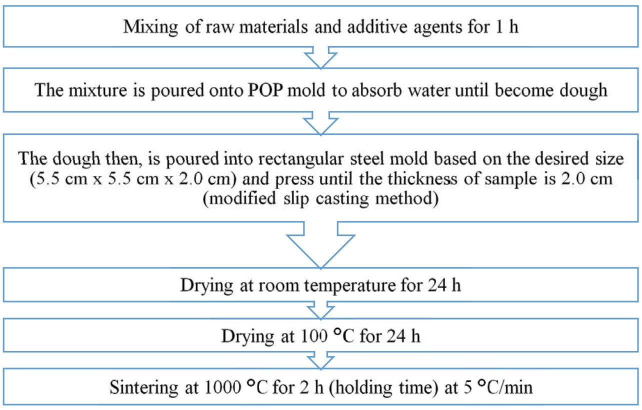

Fig. 1. Flowchart of overall natural ball clay ceramic membrane fabrication.

the membrane pore distribution. Prior to use, the cross-sectional area of the membrane sample was prepared and layered with gold using an auto fine coating machine (JEOL JSM-6360) and fixed on top of the stub, then pore size distribution assessment was analyzed. The porosity and density of the membrane after firing was evaluated using Archimedes' principle [23] as shown in Eqs. (2) and (3):

$$\text{Porosity, \%} = \frac{w_2 - w_1}{w_2 - w_3} \times 100\% \quad (2)$$

$$\text{Density} = \frac{w_1}{(w_2 - w_3)/\rho_{\text{water}}} \quad (3)$$

where w_1 is the dry weight of the ceramic membrane, w_2 is the wet weight of the ceramic membrane and w_3 is the weight of the suspended materials to be used for ceramic membrane fabrication.

The mechanical strength of the sintered membrane was determined by a compressive test at a constant rate of about 5 mm min^{-1}, using a universal testing machine, GoTech. A 5 cm × 5 cm × 2 cm sample applied was based on the actual size of the ceramic membrane prepared in the laboratory [24]. In each characterization, the average data obtained from the calculation is based on three measurements for each sample type.

2.3. Distilled water permeability test

The filtration tests were conducted using a dead-end filtration setup in batch mode operation as shown in Fig. 2. Ceramic membrane with an effective filtration area of 20.0 ± 0.2 cm^2 and a thickness of 1.5 ± 0.2 cm is placed in the membrane housing and distilled water is filled from the top of the system. Before the filtration test, the ceramic membranes were conditioned by immersing in distilled water for 24 h in order to obtain a stationary regime of each ceramic membrane. The distilled water permeability (DWP) study on the ceramic membrane in this experiment is performed at 2 bar and room temperature in triplicate for each sample type. The DWP was obtained from Ghouil et al. [25] with the equation:

$$\text{DWP} = \frac{Q}{StP} \quad (4)$$

where DWP is the permeation flux of membrane for distilled water (Lh^{-1} m^{-2} bar^{-1}), Q is the volume of permeates collected (L), S is the effective area of the membrane (m^2), t is the permeation time (h) and P is the operating pressure (bar).

3. Results and discussion

3.1. Natural ball clay ceramic membrane fabrication

The fabrication of natural ball clay ceramic membrane implies the following sequence of operation: (a) preparation of ceramic paste (CM1(A), CM2(A), CM3(A), CM4(A), CM5(A), CM1(B), CM2(B), CM3(B), CM4(B) and CM5(B)), (b) shaping using modified slip

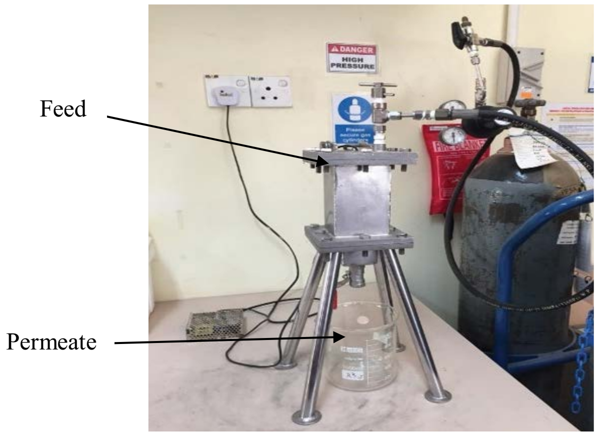

Fig. 2. Water permeability test.

casting method on flat configuration, and (c) consolidating by drying and thermal treatment. The principle is that the organic additives or pore-forming agent burns away or being eliminated by combustion during thermal treatment. Sintering is a method involved in preparing objects from powder form; by heating the materials (below its melting point) until its particles adhere to each other. It is known as solid-state sintering [12]. Fig. 3 shows the trend of thermal shrinkage increasing from around 6% to 13% as the percentage of sawdust increased from 0% to 40% at the sintering temperature of 1,000°C in the presence or absence of additive agents. These results showed that the shrinking of fabricated natural ball clay ceramic membrane is due to lost moisture during the sintering process. In addition, the sawdust as a pore-forming agent is totally degraded at this sintering temperature. Fig. 3 also illustrates that the shrinkage of a ceramic membrane with additives agent is lower than those without additive agents. This is due to the additive agent such as binder providing a bond between elements so it will increase the mechanical strength; thus, porosity decreases and densification increases. The higher packing density or densification of the ceramic membrane resulted in lower shrinkage [26].

3.2. Natural ball clay: raw material characterization

The thermal behavior of natural ball clay powder was analyzed by TGA-DTG as illustrated in Fig. 4 to determine the sintering temperature to achieve a good quality membrane. This analysis shows different weight losses encountered by the natural ball clay upon firing. The natural ball clay presents a total loss of mass around 12% between room temperature and 900°C. The TGA indicated a substantial three weight loss stages between 35.97°C and 194.85°C as a first stage, second between 194.85°C and 391.47°C, and third stage between 391.47°C and 771.49°C. The first stage loss is when absorbed surface water is removed with a mass loss of 1.35%. The second stage is when the structural water is eliminated, the hydroxyl group by dehydroxylation reaction occurs and is accompanied by a mass loss of 3.21%. A significant weight decrement occurs due to the thermal degradation of calcium carbonate to form calcium oxide and calcium dioxide in the temperature range of 391.47°C–771.49°C with

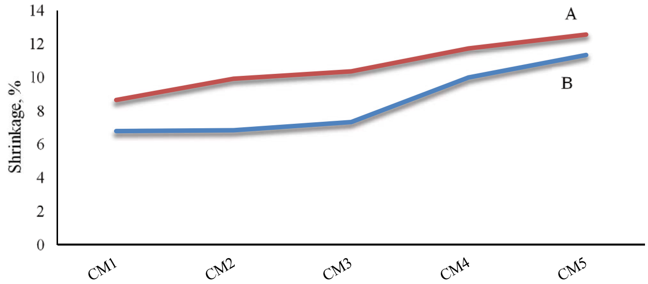

Fig. 3. Shrinkage of natural ball clay in the absence (a) or presence (b) of additive agents

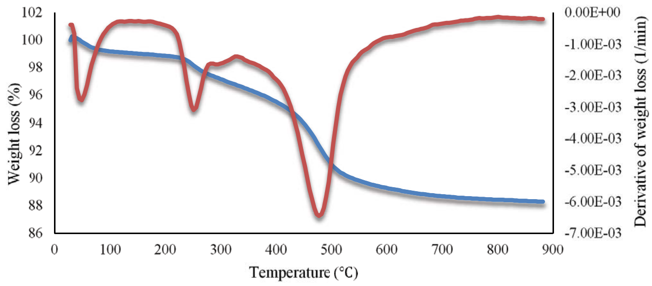

Fig. 4. TGA-DTG of natural ball clay.

7.55% total mass loss. Overall, it recommends that the membrane be sintered at a temperature above 900°C to obtain a good membrane quality. A good quality membrane must have an excellent surface, including no defects or cracks on the appearance, good mechanical and thermal strength, and high porosity [22,27].

3.3. Natural ball clay: ceramic membrane characterization

3.3.1. Cross-sectional area morphology

The SEM images illustrated in Fig. 5 show the cross-sectional morphology and pore size distribution of the flat ceramic membrane prepared from natural ball clay, sintered at 1,000°C for 2 h. Micrographs show the marked effect of sawdust percentage in the presence or absence of additive agents; a progressive enhance in porosity can be observed when the sawdust percentages increase. Additionally, based on the comparison of presence and absence of additives agents, the overall cross-section morphology analysis recommends that good distribution of pore sizes for all ceramic membrane was achieved in the presence of additive agents. Moreover, the ceramic membranes containing additive agents (CM(B)) have a smaller pore compared to the absence of additive agents (CM(A)). This might be due to the dispersant performing well, which is efficiently dispersing particles of sawdust and clay powder. Thus, during the sintering process, a good pore distribution was produced after all the sawdust particles have burnt out. Therefore, it is a key condition leading to an excellent quality ceramic membrane.

3.3.2. Porosity and density

The porosity and density of natural ball clay ceramic membrane with 1,000°C sintering temperature were

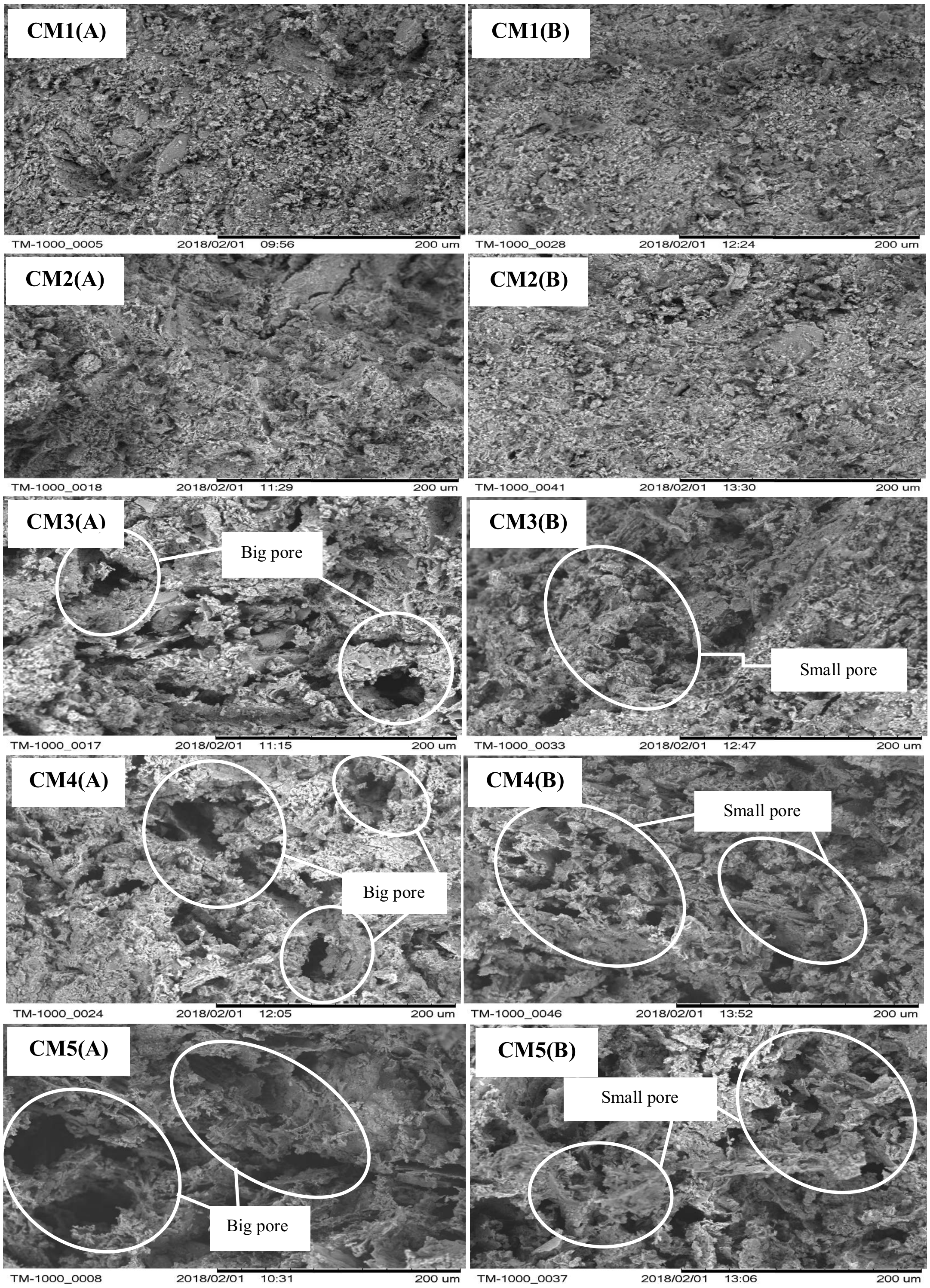

Fig. 5. SEM images of cross-sectional of natural ball clay ceramic membrane at a magnification of X500 in the absence (a) or presence (b) of additive agents.

observed. Fig. 6 depicts that both characteristics change appreciably with different sawdust amounts and in the presence or absence of additive agents. In the case of porosity, it increases approximately from 30% to 65% between 0% (CM1) to 40% (CM5) of sawdust for both additives agent condition. The increment of porosity is most likely, due to the higher amount of pore formations during the thermal process as the percentage of sawdust increased. It is also observed that the porosity of all membrane having is over 30%, which is a good achievement as a porous ceramic

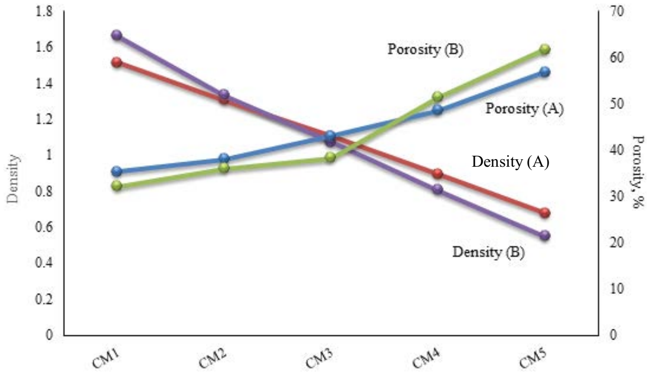

Fig. 6. Porosity and density of natural ball clay ceramic membrane in the absence (a) or presence (b) of additive agents.

membrane as highlighted by Guzman [28] and Obada et al. [29]. Converse evolution is observed for densification phenomena. The density of natural ball clay ceramic membrane decreased from CM1 to CM5 as shown in Fig. 6. The higher amount of pore formation is attributed to the lower packing particle. Thus, the particles become looser and less dense [26]. In terms of additive agents, the porosity of the elaborated membrane in the presence of additive agents gave lower values compared to the absence of additive agents. This result shows that the binder gave a good performance, which creates a bond between particles. Thus, the particle becomes denser leading to decreased porosity. However, at CM4(B) and CM5(B), the porosity of ceramic membrane increased (density decreased) in the presence of additive agents. In this case, this might happen because the mixture is not homogenous due to the higher amount of sawdust speeds up water absorbency.

3.3.3. Mechanical strength

Densification, porosity, sintering temperature and amount of pore-forming agent (e.g., sawdust, starch), as well as chemical additive agents (e.g., polyethylene glycol, sodium base), are several factors controlling the mechanical strength of the ceramic membrane. As indicated by Fig. 7, there was a trend of decreasing mechanical strength with the increment in sawdust percentage applied. The mechanical strength of the natural ball clay ceramic membrane was decreased approximately, from 17 to 1 MPa as the sawdust percentage increased from 0% (CM1) to 40% (CM5). This result shows that a highly porous membrane provides the least mechanical strength. However, the mechanical strength of CM1(A) and CM2(B) are almost and surpass 14 MPa, and this is quite appreciable or in good agreement with the revelation by Bose and Das [30,31] and Nandi et al. [32]. On the other hand, the mechanical strength of ceramic membrane increased in the presence of additives agent compared against without additive agents. This result shows that additive agents such as a binder gave a good performance, creating a bond between particles. Also, an increase in the interconnection between particles reduces the voids in membrane support or particle become denser, which causes an increase in strength and decrease in porosity [30]. Thus, it shows that the

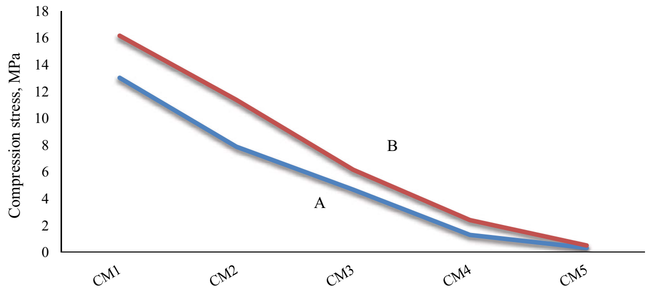

Fig. 7. Mechanical strength of natural ball clay ceramic membrane in the absence (a) or presence (b) of additive agents.

binder predominantly provides strength. However, for CM5, the mechanical strength stays the same regardless of the presence or absence of additive agents. This might be due to a weak point in the membrane leads to a decrease in mechanical strength during strength tests.

3.4. Determination of water permeability

The water permeability through a ceramic membrane is greatly influenced by several factors based on the Hagen-Poiseuille equation [33] such as pore size, porosity, density, the thickness of the membrane, the pressure difference across the membrane and also, the water viscosity [25]. Fig. 8 depicts the variation of water flux with an elaborated membrane containing different sawdust percentages in the presence or absence of additive agents sintered at 1,000°C at a pressure of 2 bar. As shown in Fig. 8, it is clearly seen that there is an increment in flux values (from 22,644 to 480,000 Lh^{-1} m^{-2} bar^{-1}) when the sawdust percentage (from CM1 to CM5) is increased. As the porosity of the membrane increased, the water permeability follows suit. The application of sawdust as a pore-forming agent induces porosity as well as water permeability. However, further sawdust addition, 40% in CM5, would deteriorate the performance of natural ball clay ceramic membranes. The study of Qu et al. [13] believed that the appropriate pore-forming agent is lower than 30% of volume, working as an efficient pore-forming agent, suppresses the driving force of densification without affecting the connections of neighboring grains while the excessive pore-forming agent would lead to the collapsed porous structure of the ceramic membrane.

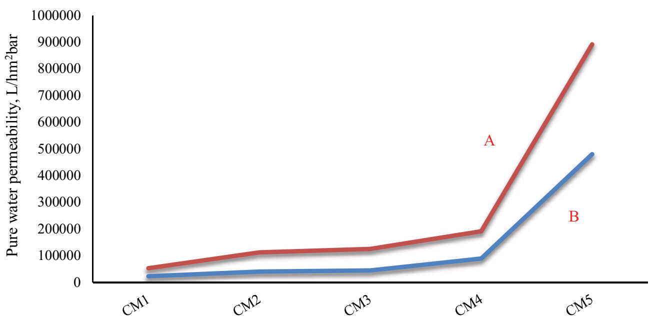

Fig. 8. Distilled water permeability test of natural ball clay ceramic membrane in the absence (a) or presence (b) of additive agents.

Table 2
Comparison between porosity and compressive strength of fabricated ceramic membrane in this present work and other literature

Materials	Shaping method	Pore-forming agent (wt.%)	Sintering temperature (°C)	Porosity (%)	Mechanical strength (MPa)	References
Natural ball clay (90 wt.%), sawdust (10 wt.%), PEG 600 and Dispex N40	Modified slip casting	Sawdust (10)	1,000	36–38	8–12	CM2 in the absence and presence of additive agents Present work
Natural kaolin-illitic clay (80 wt.%) and olive pomace (20 wt.%)	Pressing	Olive pomace (20)	1,000	30.00	6.70	[34]
Natural Moroccan clay (FCF) (100 wt.%)	Pressing	–	950	30.80	16.13	[35]
Natural bentonite (95 wt.%) and starch (5 wt.%)	Pressing	Starch (5)	950	32.12	22.00	[36]
Natural clay: ball clay (18 wt.%), feldspar (6 wt.%), kaolin (15 wt.%), pyrophyllite (15 wt.%) and calcium carbonate (18 wt.%)	Extrusion	–	950	53.00	12.00	[27]
Natural perlite powder (81.7 wt.%), Methocel derived from methylcellulose (4 wt.%), Amijel derived from starch (4 wt.%), corn starch (10 wt.%) and polyethylene glycol (0.3 wt.%)	Extrusion	Corn starch (10)	1,000	41.80	1.20	[12]
Natural Moroccan clay (81.7 wt.%), Amidon (10 wt.%), Methocel (4 wt.%), Amijel (4 wt.%) and polyethylene glycol (0.3 wt.%)	Extrusion	–	1,200	31.60	15.00	[37]

Based on previous discussion results, the higher the porosity, the lower will be the mechanical strength achieved in the fabricated ceramic membrane. Indeed, a good compromise between mechanical strength and porosity should be found to produce a good quality ceramic membrane product in terms of withstanding high pressure applied and producing high water flux. Thus, the fabricated membrane of CM2 in the presence and absence of additive agents presents a good balance in achieving optimum porosity and mechanical strength. A comparison between porosity and compressive strength in this present work (the best range of ceramic membrane) and other natural ceramic membrane studies are described in Table 2. The comparison shows that the properties of ceramic membrane in terms of porosity and mechanical strength are generally linked to the type and percentage of pore-forming agents used, type of shaping method used and sintering temperature. The fabricated ceramic membrane normally achieved an inverse relation between porosity and mechanical strength. As mentioned by Elomari et al. [22], the porosity of fabricated ceramic membrane increases with decreasing mechanical strength.

4. Conclusions

A natural ball clay ceramic membrane in a flat configuration was successfully developed. The ceramic membrane was obtained via a modified slip casting method using natural ball clay as precursor and sawdust as a pore-forming agent and sintered at 1,000°C (2 h). The influence of sawdust composition on the natural ball clay ceramic membrane properties was investigated. It was observed that an increment of sawdust composition invokes an increment in porosity and water permeability, on the contrary, a decrease in compressive strength and density. In the context of additive agents, the prepared membrane with the presence of additive agents offers better mechanical strength, however, with a poor result in porosity. Indeed, a good compromise between mechanical strength, porosity and water permeation should be found to produce a good quality ceramic membrane product. Thus, the overall conclusion is that the fabricated ceramic membrane of CM2 (containing 90 wt.% natural ball clay and 10 wt.% sawdust) in the presence and absence of additive agents show a good achievement with 7%–10% thermal shrinkage, 36%–38% porosity, 8–12 MPa compressive strength and 40,000–70,000 Lh^{-1} m^{-2} bar^{-1} in water permeability.

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