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Research Paper

Preparation and Characterization of Bentonite - Based Ceramic Hollow Fiber Membrane

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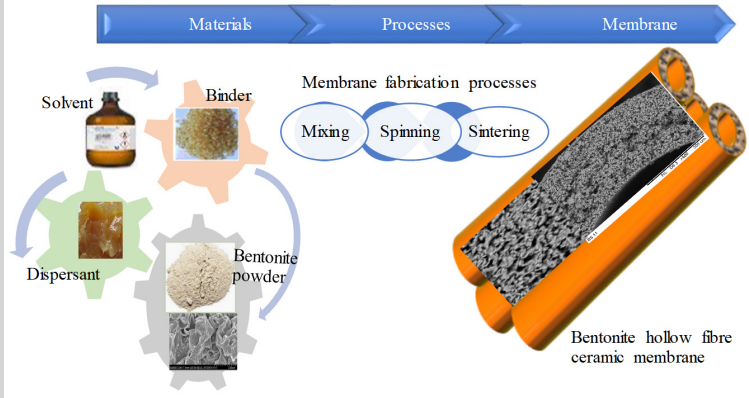
Highlights

- Fabrication of HF membrane from bentonite
- Membrane was successfully prepared via phase inversion/sintering processes
- Pure water flux increased with decreased in sintering temperatures
- Bentonite HF membrane is a promising membrane for oily wastewater

Graphical abstract



Abstract

The use of low-cost clay materials for the fabrication of ceramic membrane has attracted much interest from researchers, and the outcome would be beneficial to the industries. In this study, low-cost bentonite was used for the preparation of hollow fiber ceramic (HFC) membrane. Bentonite powder was initially characterized by field emission scanning electron microscope (FESEM) for powder surface morphology. The bentonite membrane was fabricated through dope suspension mixing, using phase inversion-based extrusion method and sintering processes. The dope suspension was prepared by mixing quantified bentonite powder, dispersant, polymer binder, and organic solvent on a planetary ball mill. This was followed by the extrusion of the dope suspension at a bore fluid rate of 10 mL/min and air gap of 5 cm and finally subjected to the sintering temperatures of 950°C, 1000°C, 1050°C, and 1100°C. FESEM images revealed that bentonite powder has a compacted interlayer order of heterogeneous surface morphology. The resulting bentonite HFC membrane surface morphologies were examined by scanning electron microscopy (SEM), and the structures exhibit asymmetric structure, which was composed of sponge-like and finger-like structures. Due to, its superhydrophilic property and pore size; the membrane contact angle and water flux performance were obtained at 1.90° and ~326 L/m².h, respectively. Overall, the results suggest that bentonite can be used in the fabrication of ceramic-based hollow fiber membrane and in addition, can as well be utilized in microfiltration for wastewater treatment.

1. Introduction

Membranes from organic and inorganic materials have been commercially available for the separation and filtration processes of wastewater. Organic membrane tends to have a lower cost. Still, the thickness required to withstand pressure drop difference during the fabrication processes brings to bear challenges associated with its chemical and thermal stability as compared with the inorganic membrane such as inorganic (ceramic) membrane [1,2]. The natural bentonite-based ceramic membrane has been widely used, due to its high flexibility, thermal and chemical stability, as well as inertness to microorganism's degradation, large surface area and affluence of cleaning after fouling. This has gained much interest

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from researchers and offers overwhelming performance over an extended service period which gives rise into a lower operating cost [3–14].

Studies from the fabrication of bentonite membrane based on different configurations have revealed that, for instance, Eom *et al*. fabricated microfiltration ceramic membranes from a silicate and clay mineral textures containing bentonite for oily wastewater separation. The results showed that bentonite-based ceramic was sintered at 1000ºC with the mean pore of 0.4 µm and water permeability of 32 L/m^2.h [9]. Bakalár *et al*. studied the influence of internal configuration on the MF membrane of bentonite (montmorillonite) suspensions with supported αAl_2O_3 and ZrO_2 in the fabrication of tubular ceramic MF membrane. The results on the performance suggested that mean pore was 0.120 µm and pure water flux was 160 L/m^2.h [15]. Bouazizi *et al*. also carried out elaborated works on the application of flat disk microfiltration (MF), ultrafiltration (UF), and nanofiltration (NF) membranes for industrial wastewater treatment from natural Moroccan bentonite clay using a uniaxial pressing and sintering method [16–18]. Accordingly, the results revealed that the flat disk membranes were sintered at 950ºC and the mean pore and water permeability showed 1.8 µm and 725 L/m^2.h, respectively. Chihi *et al*. examined the use of low-cost Tunisian bentonite clay for wastewater treatment from tubular ceramic MF membrane while applying the extrusion and sintering technique for the fabrication. The tubular membrane was consequently sintered at 1100ºC while mean pore value and water permeability indicate 1.7 µm and 525 L/m^2.h, respectively [4].

Meanwhile, the preparation and fabrication of bentonite hollow fiber membrane is still a mirage as most of the researchers from earlier discussed studies mainly develop flat sheet and tubular membrane modules from this lost-cost material using uniaxial pressing and extrusion techniques. These modules have limitations in terms of low surface area per unit volume. By implication, to treat high volume wastewater would require high flux and low – cost ceramic membrane [19].

To our best knowledge, there is no fabrication of hollow fiber ceramic membrane from bentonite powder using phase inversion technique. Therefore, the objective of this study was to fabricate hollow fiber membrane from bentonite powder using phase inversion and sintering techniques for water filtration. Most significantly, the effects of sintering temperature (i.e. 950°C, 1000°C, 1050ºC and 1100ºC) on the membrane were studied in terms of morphology, percentage porosity and water permeability test. By these parameters, low-cost bentonite hollow fiber membrane can be used for water filtration process.

2. Materials and method

2.1. Materials

Bentonite powder was acquired from Aladdin industrial corporation Shanghai, China and other additives such as Polyethylene glycol 30-dipolyhydroxystreate (Arlacel P135, CRODA), N-methyl-2-pyrrolidone (NMP) (HPLC grade, Rathbone), and Polyethersulfone (PESf) (Radel A-300, Ameco Performance) were used as a dispersant, solvent and a binder, respectively. All the materials were used without further purification. The deionized water and tap water were used for the spinning process and the permeability test, respectively.

2.2. Bentonite powder characterization

The bentonite powder was characterized using field emission scanning electron microscopy (FESEM) at 2 kV when the magnification was varied within the range of 10,000 – 70,000 (HITACHI model: SU8020). The percentage porosity test was investigated on the membrane at different temperature using mercury intrusion porosimetry (MIP, Autopore IV, micrometric, USA).

2.3. Preparation of ceramic suspension

Arlacel P135 (1 wt.%) was melted in an oven at 60°C, then dissolved in NMP solvent (76 wt.%). Bentonite powder (18 wt.%) was slowly added into the mixture of Arlacel P135 and NMP. This was consequently followed by a continuous mechanical stirring of the mixture using a planetary ball mill (NQM-4 model) at 194 rpm for 48 h to ensure proper dispersion of bentonite powder. A relatively low ceramic loading of bentonite (i.e. 18 wt.%) was used as compared to other ceramic loading reported in the literature because naturally bentonite clay swells and exhibits thixotropy in both polar and non-solvent [22–25]. PESf (5 wt.%) was added to the mixture after 48 h to achieve a complete dispersion and binding of the dope suspension for another 48 h. Moreover, the formulation for the preparation of the ceramic dope suspension was adopted based on the work reported by Hubadillah *et al*. [24]. Furthermore, the prepared bentonite dope was degassed on a gentle stirring for 30 min to ensure that any air bubbles trapped are entirely removed from the dope suspension. Table 1 shows the spinning condition employed in this study.

2.4. Fabrication of bentonite hollow fiber membrane

Subsequent degassing of the dope suspension, the prepared bentonite dope suspension was loaded into 200 mL stainless steel syringes. The dope suspension and deionized water were extruded by the syringe pump (PHD2000, Harvard Apparatus) via the spinneret at a constant flow rate of 10 mL/min at room temperature and an air gap of 5 cm as indicated in Figure 1. Before thorough washing of the membrane with water, the membrane precursor undergone a complete immersion in water for 12 h for proper phase inversion. The nascent precursor fibers were cut into 20 cm in length and dried at room temperature for 24 h before sintering between 950°C and 1100°C at an interval of 50°C in a tubular furnace (Model: XL-1700, Magna value). After the initial thermal decomposition of organic solvent and other additives. The sintering process was further increased from 650°C at the rate of 3°C/min for a period of 2 h and followed by a heating rate of 5°C/min for another period of 5 h to the target temperature. Then a gradual drop in the temperature at a cooling rate of 5°C/min. Figure 1 illustrates the process of bentonite hollow fiber ceramic membrane spinning.

2.5. Morphological studies

The membrane was examined on scanning electron microscopy (SEM) HITACHI model: TM3000. The magnification was also varied from 100 – 8,000 to establish their surface and cross-sectional area. The effect of sintering temperature on the membrane morphology was studied at 950°C, 1000°C, 1050°C, and 1100°C.

Table 1
Spinning parameter for bentonite hollow fiber membrane precursors.

Spinning parameters	Values
Spinneret internal diameter (mm)	1.4
Spinneret orifice diameter (mm)	2.8
Dope temperature (ºC)	27.0
Dope extrusion rate (mL/min)	10.0
Bore fluid injection rate (mL/min)	10.0
Air gap (cm)	5.0
Internal coagulant	Deionised water
External coagulant	Tap water
Internal coagulant temperature (ºC)	27.0
External bath temperature (ºC)	27.0

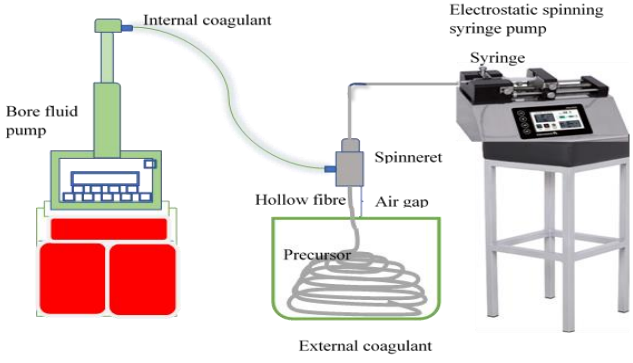


Fig. 1. Schematic representation of bentonite hollow fiber membrane fabrication process.

2.5.1. Chemical resistance, water absorption test and shrinkage analysis

The chemical resistance test was carried out using NaOH (aq) at pH (12) and HCl (aq) at pH (2) solution. The membrane was evaluated after allowing it in the acid and alkaline medium for the weight loss [25]. The weight loss of the membrane was conducted three times for the two aqueous solutions, and the standard error of the mean was determined from the standard deviation using the Eq. (1). While the water absorption of the membrane sintered at 950°C, 1000°C, 1050°C and 1100°C was determined based on ASTM C373-88 method. Both the outer and inner diameters of the unsintered membrane and sintered membrane were determined using micron ruler and on the SEM images, and the Eq. (2) was used to compute the shrinkage percentage.

$$SE = \frac{\sigma_M}{\sqrt{N}} \quad (1)$$

where SE is the standard error, σ_M is the standard deviation and N is the number of samples tested.

$$S = \frac{D_o - D_i}{D_i} \times 100 \quad (2)$$

where *Do* and *Di* are the diameters of membrane before and after sintering.

2.6. Surface properties measurement

Since the contact angle measurement is desirable in wettability studies as it expresses the degree of wetting in a solid and liquid interaction. The measurement of the contact angle was used to quantify the hydrophilicity of the fabrication surface using contact angle goniometer (OCA 15 EC, Dataphysics). About 0.5 µL of deionized water was dispensed on the bentonite membrane surface. In the process, any changes were accessed using a high-resolution camera to obtain an approximate average contact angle. Hence the procedure was repeated severally, and the average contact angles were taken in order to minimize the experimental errors.

2.7. Membrane permeability

The experimental set-up used for the water permeability test was conducted on a dead-end system at room temperature. Bentonite membrane (4 cm) was potted in an adapter and then inserted into filtration column. The feed solution (deionized water) was pumped using a peristaltic pump in tangentially to the membrane. The transmembrane pressure was maintained constantly through the pressure gauge and corresponding regulation valve. Permeate was collected in a measuring cylinder so that the flux can be determined over time at sintering temperature of 950°C, 1000°C, 1050°C, and 1100°C. The pure water flux (J) was determined using Eq. 3:

$$J_w = \frac{V}{A\Delta t} \quad (3)$$

where J_w is the pure water flux (L/ m^2.h), V is the volume of the water permeate through the membrane, A is the effective surface area of bentonite membrane (m^2), Δt is the complete permeation time, and t (h) is the filtration time.

3. Results and discussion

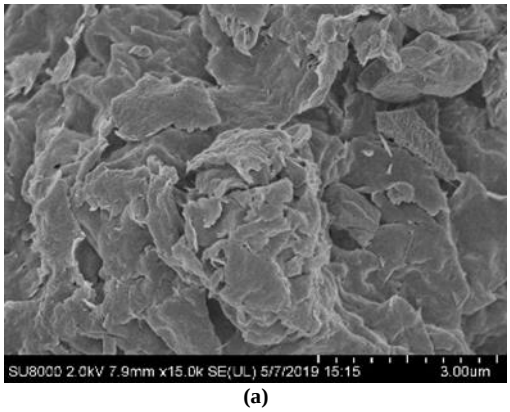
3.1. Bentonite powder analysis

FESEM was used to observe the morphological structure of bentonite powder. Figure 2 (*a* and *b*) revealed flake-like structures of abundant silicates interlayers. The structure contained a compacted interlayer order of heterogeneous surface morphology, which was in accordance with Fauzi *et al*. that the destabilization process contributed to more loosely packed tactoids in bentonite [26]. Similarly, Lui *et al*. also confirmed the existence of lamellar curly in the surface of bentonite [27].

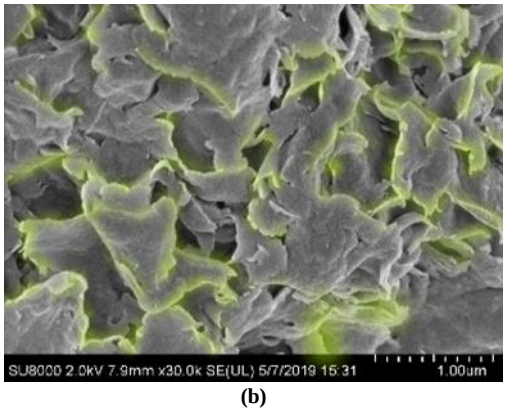
3.2. Morphology studies

Figure 3 depicts the overall SEM images of sintered bentonite hollow fiber ceramic membrane prepared at 18 wt.%. Figure 3 (A, B, C, and D) showed the surface morphological view of the membrane at four different sintering temperatures (950°C, 1000°C, 1050°C; and 1100°C). Figure 3 (A2, B2, C2, and D2) possess macrovoids, which extends across at 70% and 30% sponge-like structure. Figure 3A (950°C) contains less dense, asymmetric, and highly porous structures; and Figure 3B (1000°C) becomes slightly reduced in the asymmetric structures and the sponge-like structures becomes dense. This can be attributed to grain growths during bentonite membrane sintering operation and the compacted interlayer framework of the membrane [28].

Figure 3 (C3 and D3) showed that as the sintering temperature increases from 1050°C to 1100°C, the sponge-like voids tend to become dense and more compacted. Meanwhile, the macrovoids seem to be a standstill and become partly and gradually melted with a rise in sintering temperature. It can also be seen that the membrane sintered at 950°C has the most porous structure as compared to the ones sintered at higher temperatures, for example, at 1100°C. Therefore, with a gradual rise in sintering temperature from 950°C to 1100°C, the pore size of the sponge-like structures of the membranes became reduced, and the pore size then becomes isolated on the surface of the membrane. Meanwhile, the surface structures (Figure 3 A4, B4, C4, and D4) revealed that the membrane possesses sponge-like structures. Apparently, with the rise in the sintering temperature, the sponge-like structures became denser with significant changes (see Figure 3 (A4 - C4)). Conversely, the outer and inner diameters decrease from 1.10 mm to 0.991 and 0.746 mm to 0.667 mm, respectively as the sintering temperature increases from 950°C to 1100°C.



(a)



(b)

Fig. 2. FESEM images of natural bentonite powder. Two magnifications, 15,000x and 30,000x, are shown for the powder.

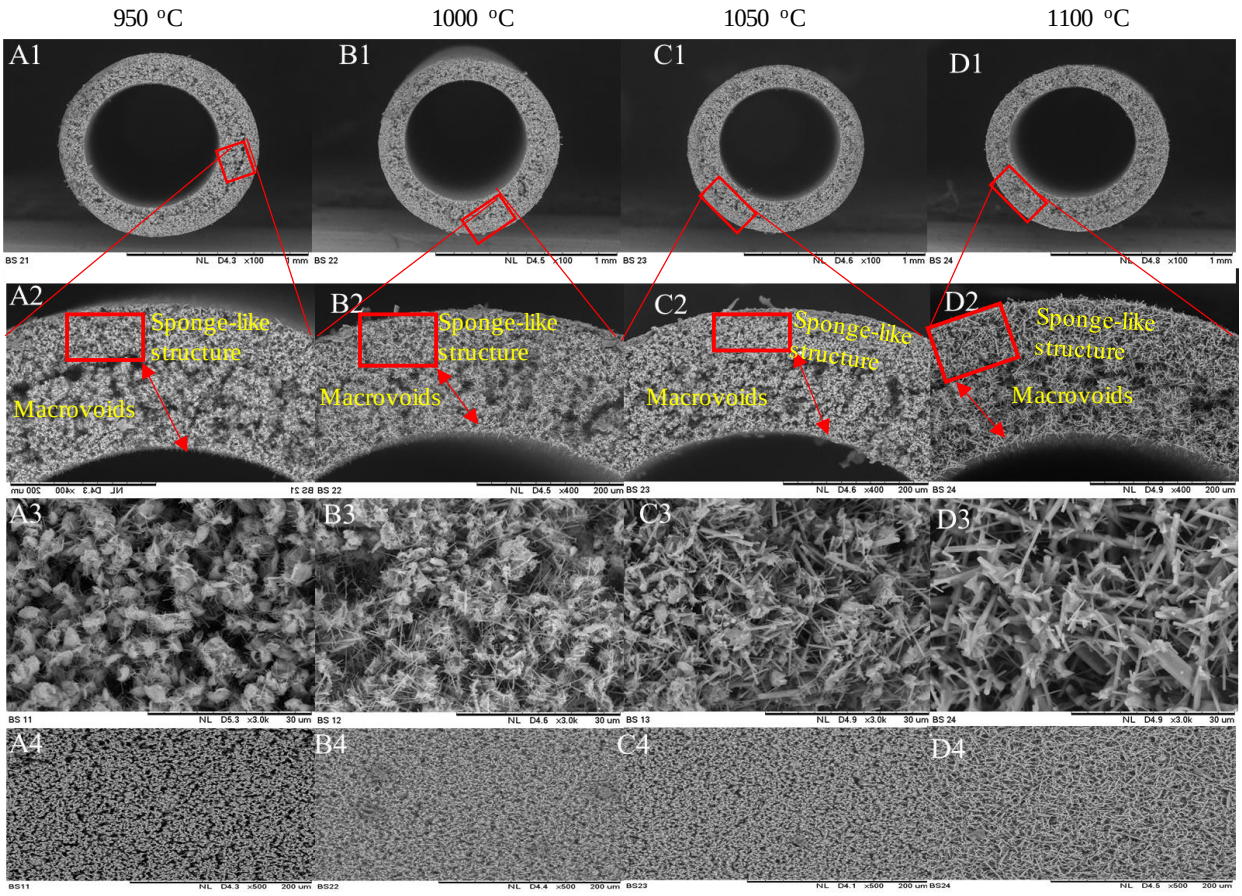


Fig. 3. (1) Overall view; (2) cross-section; (3) porous structures; (4) surface microstructure of BHFC membrane with the loading of 18 wt.% sintered at; (A) 950°C; (B) 1000°C; (C) 1050°C; and (D) 1100°C.

This is in line with similar studies by Kingbury *et al.* [29] that the higher viscosity of the ceramic suspension lowers the formation of finger-like structures in the membrane. The finger-like structures tend to completely diminish if the ceramic dope suspension viscosity attained a specific limit as reported in the work of Hubadillah *et al.* [26] and Othman *et al.* [27]. In this case, the asymmetric for finger-like structure tend to become macrovoid due to low dope suspension viscosity. At higher loading dope suspension becomes highly viscous, which deters the extrusion process. As indicated in Figure 4, the viscosity of bentonite suspensions at the relative shear rates ranges from 5 to 90 s^{-1} contained 18 wt.% in the dope suspension. In addition, the trends can be attributed to the shear-thinning behaviour as the shear rates increase with a decline in the bentonite viscosity. Figure 4 showed a similar pattern with the works reported by [28,29]. The shear viscosity was attained at 950 cp after 5 $s^{-1}$and less than the shear rates and viscosities at 58.7 wt.% alumina and 40 wt.% kaolin content loading, respectively [28,29]. The result from the low viscosity has contributed and exhibited a reduction in the formation of finger-like voids, which has been the controlling factor for finger-like structures.

3.3. Porosity and water absorption

Figure 5 illustrates the variations in porosity and water absorption as a function of sintering temperature. As indicated in the figure, the temperature ranges from 950°C to 1100°C. For the porosity, it decreases from 53 % at 950°C to 43% at 1100°C. The membrane macrovoids become gradually close as the temperature increases from 950°C to 1100°C. This reduction in the membrane macrovoids can be attributed to smaller pores in the membrane densified structures [31], which was in accordance with Figure 3. A similar trend was observed for the water absorptive capacity. The water absorption decreases from 50% at 950°C to 42% at 1100°C. The apparent absorptive behaviour can be attributed to a decrease in the pores [32] and surface structures, as indicated in Figure 3 (B3, C3, and D3).

3.4. Effect of chemical resistance

The effect of alkaline and acidic aqueous solution was studied on the bentonite-based membrane for chemical resistance, as indicated in Figure 6. In the NaOH medium, the membrane experiences high weight loss from 0.35% at 30 mins to 1.5% at 250 mins. The maximum weight loss was obtained at 1.5%. This weight loss can be as a result of an increase in acid concentration in the bentonite compositions as it is known to contain a large proportion of weak metallic oxides and other weak acids [31]. While no significant weight loss was observed in the acidic medium, HCl (aq). 0.05% weight loss at 30 mins was initially obtained with a slight 0.15% increase at 250 mins, but a steady weight loss°Ccurred between 150 mins and 200 mins. The standard errors were presented as an error bar in Figure 6. Hence, the presence of bentonite-based ceramic membrane in HCl (aq) medium does not favour acidic medium, which can lead to low integrity and deterioration of membrane performance.

3.5. Water permeation test

Permeation performance of bentonite hollow fiber membrane was examined based on pure water flux test. Figure 7 illustrates the membrane fabricated from bentonite at different sintering temperatures. The performance of the membrane largely depends on the pore size and porosity. Before the stability of the water flux, all the four sintered membranes showed high fluxes for 40 mins permeation time. Then, after 40 min permeation time, the water flux became stable. The membrane sintered at 950°C reveals highest water flux because of its large pore size and high porosity as also indicated in the SEM image (Figure 3 A3). In addition, the structure became densified and melted at 1050°C and 1100°C, then resulted in low flux during permeation process. The water permeance of bentonite membrane showed a decline in the water flux in the following order of sintering temperature: 950°C (~ 326 $L/m^2.h$) > 1000°C (~ 288 $L/m^2.h$) > 1100°C (~ 78 $L/m^2.h$) > 1050°C (~ 18 $L/m^2.h$). The above order was expected as the SEM displayed the morphological changes due to the heat treatment during the sintering process [33]. Also, FESEM analysis provides the flaky nature of bentonite particles, and the voids formed between particles due to their irregular shapes [33].

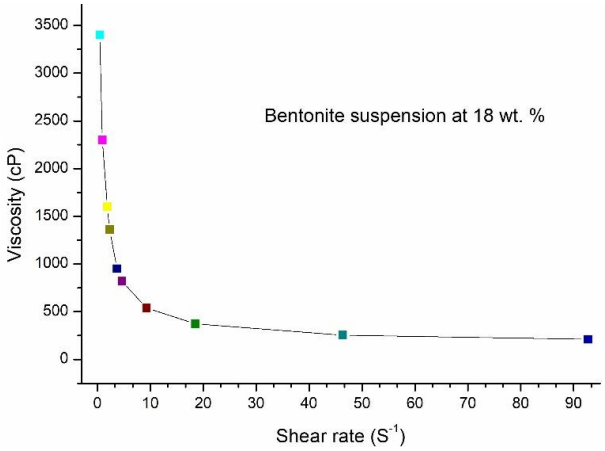


Fig. 4. Viscosity of ceramic suspension at 18 wt.% bentonite content.

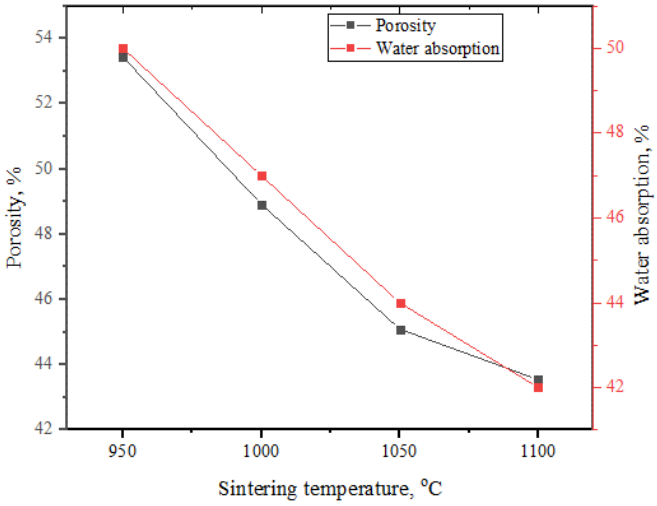


Fig. 5. Effect of sintering temperature on porosity and water absorption.

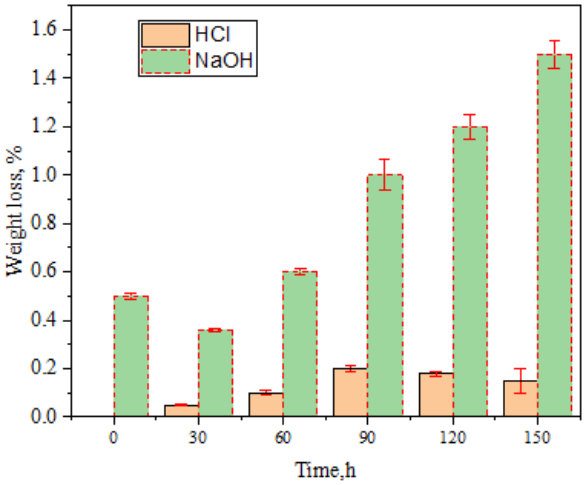


Fig.6. Weight loss of membrane support in alkaline and acidic solution (sample number, N:3).

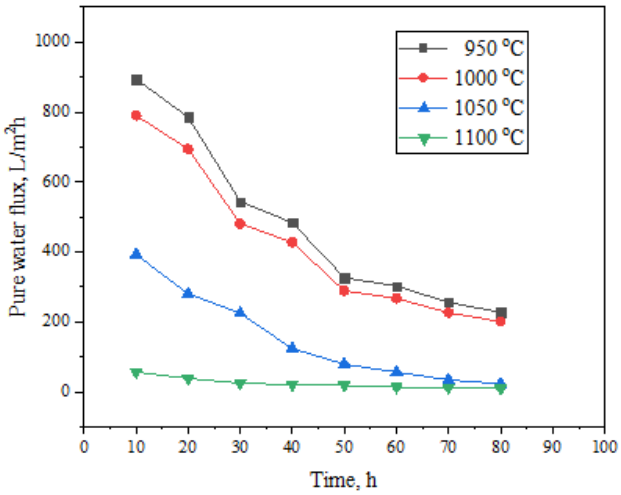


Fig. 7. Variation of water flux of bentonite membrane sintered at 950°C, 1000°C, 1050°C, and 1100°C with filtration time.

Also, the water flux performance of bentonite hollow fiber ceramic membrane was evaluated and compared with other bentonite-based membranes, as indicated in Table 2. It can be observed that bentonite 18 wt.% at 950°C sintering temperature possesses high water flux at 326 L/ m^2.h. It is significant to mention that bentonite hollow fiber membrane was sintered at relatively low sintering temperature. In the work investigated by Eom *et al.* [9] and Chihi *et al*. [4], bentonite membrane fabricated through uniaxial pressing and extrusion were sintered at a higher temperature. Despite having a wide mean pore size of 3.9 µm and pure water flux of 326 L/m^2.h, the flux is still not proportional with the pore compared to low lower pore size and high-water flux as reported by Bouazizi *et al*. [17]. The performance can be attributed to non-pronounced asymmetric (finger-like) structure, as it contributes to high water permeability and small effective surface area of membrane used during the filtration process, as it also provides limited surface interaction with the feed solution [24]. Hence, the bentonite membrane prepared through phase inversion technique and low sintering temperature offers relatively more advantages compared with the other membranes with different configurations. Therefore, bentonite hollow fiber ceramic membrane promises a better alternative in terms of preparation technique, configuration and performance.

4. Conclusions

In the present work, preparation and characterization of bentonite hollow fiber ceramic membrane were carried out. BHFC membrane was successfully sintered at 950°C, 1000°C, 1050°C, and 1100°C at 18 wt.% loading. SEM results showed that the membrane surface was sponge-like, and the cross-sectional was nested – straw-like structure. Lower bentonite loading makes the membrane asymmetric and less dense as a result of low viscous of the dopes suspension. In contrast, higher temperature effect does not favor the surface morphology as the membrane structures tend to melt and shrink, and this may also be attributed to low densification of the membrane. The shrinkage of the membrane is less than 9.91 % and steady in the range from 950 to 1000°C. While the shrinkage increases from 9.91 % to 12.46 % with rising temperature from 1000 to 1100°C. This is attributed to the neck to neck growth between the agglomerates of small particles and larges particles during the sintering process and then resulted in the densification in the membrane structure, as such contributed lower water permeability. However, the temperature at 950°C favors BHFC application in water permeation process as it possessed the highest flux at 326 L/m^2.h.

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Table 2
Comparison between this work and other similar studies.

Ceramic membrane	Membrane module	Fabrication techniques	Sintering Temp.,°C	Mean pore size, μm	Mechanical strength, MPa	Water permeability L/ h.m^2	Removal efficiency, %	Reference
Bentonite clay	Hollow fiber	Phase inversion	950	3.9	NA	326	NA	This work
Bentonite (montmorillite)	Tubular	Extrusion	NA	0.120	NA	160	NA	[15]
Bentonite clay	Flat disk	Uniaxial pressing	950	1.70	22	520	99	[31]
Bentonite clay	Flat	Uniaxial pressing	950	1.8	14.6	725	98	[3]
Bentonite clay	Tubular	Extrusion	1100	1.7	24.06	525	NA	[4]
Calcined diatomite, bentonite	Ceramic	Uniaxial pressing	1000	0.4	32	0.09	92.9	[9]

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