Design and fabrication of ceramic hollow fiber membrane derived from waste ash using phase inversion-based extrusion/sintering technique for water filtration

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Research Article

Keywords: palm oil fuel ash, ceramic hollow fiber membrane, membrane morphology, phase inversion, sintering

Posted Date: August 24th, 2020

DOI: <https://doi.org/10.21203/rs.3.rs-62414/v1>

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Abstract

Low-cost green ceramic hollow fiber membranes (CHFM) were successfully designed and fabricated from the industrial waste ash, palm oil fuel ash (POFA) via phase inversion-based extrusion/sintering techniques for water filter application. The extrusion process parameters such as suspension viscosity, air gap distance, and bore fluid flow rate were systematically explored to produce the membrane with the desired morphology. A high suspension viscosity would result in a small macro-voids structure. Moreover, a high air gap distance would induce fiber with long macro-voids structure, while a low bore fluid flow rate would lead to the formation of distorted lumen structure. The effect of sintering temperature towards CHFM was also studied in detail. An increase in sintering temperature improved the membrane bending strength, but also adversely affected the pure water flux due to lower porosity and higher tortuosity. The developed membranes achieved excellent bending strengths of > 75 MPa at relatively low sintering temperatures than the alternative ceramic counterparts, due to its high potassium oxide, K_2O content, which acted as the low melting point sintering aid. The relatively low sintering temperature of POFA-derived CHFMs could reduce the energy consumption and sintering duration, which could be more economically attractive, as compared to their ceramic counterparts, thus benefiting industrial users.

1 Introduction

Porous ceramic hollow fiber membranes (CHFM) have gained so much interest in water treatment applications in recent years due to several remarkable advantages such as high durability at high operating temperature and pressure, exceptional mechanical strength, excellent resistance against corrosive agents/chemicals, high flux and separation efficiency, as well as easy to clean and regenerate. These advantages allow CHFM to attain long service time without frequent breakdown [1, 2]. Moreover, the hollow fiber membrane configuration consisting of shell and tube sides has high packing density with a large permeation area when it is assembled in a membrane module, offering high productivity at low energy consumption [3, 4]. Today, the majority of CHFM in the market are fabricated from high-purity alumina. In addition, alumina is known for its intrinsic refractory nature, hence, a very high sintering temperature (usually $\geq 1450^\circ\text{C}$) is required to fabricate high-strength alumina membranes from micron-sized alumina precursor powders [5–7]. The uses of costly alumina precursor powders and very high sintering temperatures result in high operational cost of ceramic membrane fabrication. In the last decade, an increasing number of studies have been devoted to the fabrication of CHFM using alternative materials such as natural materials (clays) [8–10] and sustainable solid wastes materials [9, 11, 12]. It is believed that the utilization of these low-cost alternative materials could reduce the membrane fabrication costs, making CHFM more cost-competitive.

Malaysia is naturally endowed with tropical biodiversity such as palm oil. The palm oil industry is considered as one of the world's largest agriculture industries. The global palm oil production volume was over 77 million tons in 2018 [13]. According to the European Palm Oil Alliance [14], palm oil and palm kernel oil account for over one-third of the global vegetable oil production. The rapid development of the palm oil industry leads to the generation of colossal amounts of by-products that could pose threats to

the surrounding environment and local communities. Palm oil fuel ash (POFA) is among the major by-products from the burning of palm kernel shells, palm oil fiber, and palm oil husk. POFA contains a large amount of crystalline silica in the form of quartz [12]. To add its technological values, POFA could be harnessed for the fabrication of low-cost silica-based CHFM for water treatment. Typically, the fabrication of silica-based ceramic membranes involves comparatively low sintering temperature than that of alumina-based membranes [9, 11, 15]. The crystalline silica phase also gives the membranes with better mechanical strength than the amorphous phase [9]. Besides, POFA also has high porosity that is attributed to its composition consisting of various inorganic oxides [12, 16]. The abundant silanol group on the rudimentary porous structure of POFA makes it prone to adsorption, separation, and catalysis processes [17].

Apart from the selection of raw materials, the fabrication cost of CHFM can also be reduced using simplified and efficient fabrication techniques. In general, ceramic membranes for water treatment applications exhibit multilayer asymmetric microstructure, in which the denser layers contribute to the separation mechanisms, whereas the less dense porous layers provide the mechanical support to the membranes. The fabrication of asymmetric structured CHFM typically involves multiple steps that are time and energy-consuming. Recently, the combined phase inversion and sintering methods have attracted popularity for the fabrication of CHFM [7, 18, 19]. This method enables asymmetric structured CHFM to be produced in a single step with one heat treatment session only [3, 20].

Usually, the CHFM prepared via this method has two distinct structures, namely sponge- and finger-like structures. The dense sponge-like layer with the smallest packing pore size acts as the separation layer and contributes to the mechanical strength of the membranes. The finger-like structure reduces the mass transfer resistance, but also detracts the membrane strength [18, 20]. High mechanical strength is particularly crucial for CHFM to ensure the long-term performance stability of the membranes [21, 22]. Past studies have witnessed the manipulation of the phase inversion parameters and sintering temperatures to augment the mechanical strength of CHFM [5, 9]. The variation of these parameters would change the morphological structure of the membrane, and as a result, affecting the mechanical strength of the membranes.

Recently, our group has investigated the feasibility of the green POFA-derived CHFM for oily water separation and heavy metal adsorption [12, 23, 24]. These membranes successfully demonstrated outstanding filtration and adsorption performances. However, a detailed study on the effects of the phase inversion-based extrusion/sintering method conditions towards acquiring POFA-derived CHFM with high strength has not yet been explored. Hence, a thorough investigation of the effects of phase inversion parameters such as suspension viscosity, air gap distance and bore fluid flow rate on the morphological and mechanical properties of membranes was conducted. The effects of sintering temperature on the membrane properties such as microstructure, surface properties, mechanical properties, pore size, porosity, and water permeation performance were also thoroughly examined. This study would expand the understanding of the development of CHFM from the green alternative materials, especially the waste

materials with relatively large particle sizes. In the last part of this study, the properties of the POFA-derived CHFM were compared with other alternative CHFMs reported in the literature.

2 Materials And Procedures

2.1 Materials

The raw POFA was supplied by a crude palm oil mill in Chaah, Johor, Malaysia. The raw ash was first ground into smaller particles using a Waring Model 800G blender, followed by the thermal pre-treatment in a Nabertherm Model LT 5/12 muffle furnace at 700°C for 2 hours with a heating rate of 5°C/min. The pre-treated POFA was further ground by ball-milling at 200 rpm for a week and sieved to the particle sizes of < 36 µm before the membrane fabrication. Table 1 shows the chemical compositions of POFA as determined by X-ray fluorescence (XRF) analysis. N-methyl-2-pyrrolidone (NMP, EMPLURA®, Merck), polyethersulfone (PES, Radel A-300, Ameco Performance), and Arlacel P135 (Polyethylene glycol 30-dipolyhydroxystearate, CRODA) were used as the solvent, binder, and dispersant, respectively for the preparation of the ceramic suspensions. Furthermore, tap water was used as the internal and external coagulants during the phase inversion extrusion process.

Table 1
Chemical compositions of POFA as determined by XRF analysis

Metal oxide	Composition (wt%)
SiO ₂	74.5
K ₂ O	6.4
Al ₂ O ₃	5.2
CaO	3.3
MgO	3.4
Fe ₂ O ₃	3.1
P ₂ O ₅	2.5
SO ₃	1.0
TiO ₂	0.2
Mn ₂ O ₃	0.1

2.2 Preparation of POFA-derived ceramic hollow fiber membrane

The POFA-derived CHFMs were developed via phase inversion-based extrusion/sintering method. Ceramic suspensions with different POFA loadings ranging from 40 wt%–60 wt% were prepared. In the suspension preparation, 1 wt% of Arlacel P135 was first mixed with NMP, followed by the addition of POFA with gentle stirring. The suspension was then subjected to ball-milling in a NQM-2 planetary ball mill system at 190 rpm for 48 hours before the addition of PES. 5 wt% of PES was added to the suspension which was then further ball-milled to acquire a uniform suspension. After 48 hours, the suspension was degassed under vacuum with gentle stirring for 30 minutes to remove the entrained air bubbles that might cause membrane defects. After degassing, the ceramic suspension was immediately transferred to a 100 mL stainless steel syringe and extruded through a tube-in-orifice spinneret (outer diameter: 2.8 mm; inner diameter: 0.5 mm) at an extrusion rate of 10 mL/min into a tap water coagulant bath. The schematic diagram of the tube-in-orifice for single-layer hollow fiber precursors is shown in **Figure A.1** in the **Appendices**. The air gap distance between the spinneret and water bath was varied from 5 to 15 cm. Moreover, tap water was used as the internal coagulant (bore fluid) with flow rates ranging from 6 to 20 mL/min. The hollow fiber precursors obtained from the extrusion process were soaked in tap water for 24 hours to complete the phase inversion process, followed by the overnight drying at room temperature. Finally, the precursors were sintered at the temperature ranging from 1 025°C–1 100°C in an air atmosphere using a XY-1700 Magna tubular furnace. The sintering profile of the POFA-derived CHFMs is depicted in **Figure A.2** in the **Appendices**. The membrane fabrication parameters are summarized in Table 2.

Table 2
Fabrication parameters of POFA-derived CHFM

Sample	POFA loading (wt%)	Suspension flow rate (mL/min)	Bore fluid flow rate (mL/min)	Air gap distance (cm)	Sintering temperature (°C)
C-P40	40	10	9	5	1 050
C-P45	45	10	9	5	1 050
C-P50	50	10	9	5	1 050
C-P55	55	10	9	5	1 050
C-A5	55	10	9	5	1 050
C-A7	55	10	9	7	1 050
C-A10	55	10	9	10	1 050
C-A15	55	10	9	15	1 050
C-B6	55	10	6	5	1 050
C-B9	55	10	9	5	1 050
C-B12	55	10	12	5	1 050
C-B15	55	10	15	5	1 050
C-B20	55	10	20	5	1 050
C-S1	55	10	9	5	1 025
C-S2	55	10	9	5	1 050
C-S3	55	10	9	5	1 075
C-S4	55	10	9	5	1 100

2.3 Characterization of POFA-derived ceramic hollow fiber membrane

The chemical composition of the POFA was determined via XRF analysis using a X-Supreme 8000 Oxford Equipment. The viscosity of the ceramic suspension was analyzed via a Brookfield viscometer at the shear rates ranging from 0.3 s^{-1} to 100 s^{-1} at room temperature. The cross-sectional and surface morphologies of the as-prepared CHFM were observed through scanning electron microscopy (SEM) analysis (Hitachi TM3000) at various magnifications. The membrane samples were coated with platinum under vacuum for several minutes before the SEM analysis. The radial shrinkage of the CHFM was also determined from the SEM images. The flexural strength of the CHFM was determined via a three-point bending test using an Instron 3342 tensile tester. Each measurement was repeated three times ($n = 3$) to ensure the accuracy of the results. The bending strength, σ_F (MPa) was then calculated using **Eq. (1)**.

$$\sigma_F = 8FLD_o / \pi(D_o^4 - D_i^4) \quad (1)$$

where F is the force at which the hollow fiber membrane fractured (N), L is the length of the membrane (m), whereas D_o and D_i are the outer and inner diameters of the membranes (m), respectively. The surface roughness of the CHFM prepared at different sintering temperatures was investigated via atomic force microscopy (AFM) analysis using XE-100 AFM from Park Systems by tip-scanning with a scan size of $10 \mu\text{m} \times 10 \mu\text{m}$. A Micromeritics Autopore IV 9510 mercury porosimeter was used to characterize the pore size distribution (PSD), porosity, and tortuosity factor of the membranes sintered at different temperatures (1025°C – 1100°C). The pure water flux (PWF) performance of the membranes was tested using a homemade crossflow filtration system. The flux performance test was conducted at a pressure of 2 bar at room temperature for one hour. The test was repeated three times ($n = 3$) for each sample. Before starting the experiment, the membranes were stabilized by running the pure water through the membranes at constant pressure for 10 minutes. PWF ($\text{L}/\text{m}^2\text{h}$) was calculated using the following equation.

$$\text{PWF} = V / At \quad (2)$$

where V is the cumulative volume of permeate water collected (L), A is the total permeation area of the membrane (m^2) and t is the time taken to collect V (1 hour).

3 Results And Discussion

3.1 Morphology controls and bending properties of hollow fiber membranes

3.1.1 Effect of POFA loading

The POFA loading of the ceramic suspension was varied to investigate the effect of suspension viscosity on the CHFM morphology. Figure 1 illustrates the rheological behaviors of suspensions with different POFA loadings. All the suspensions demonstrated the shear-thinning behaviors in which the suspension viscosity decreased with increasing shear rate from 5 s^{-1} to 100 s^{-1} (Fig. 1A). These rheological behaviors were typical non-Newtonian flows that are commonly found in many ceramic slurries [25–27]. Figure 1B shows the suspension viscosity at the shear rate of 25 s^{-1} . It could be seen that the suspension viscosity increased slowly with the increase in POFA loading for the suspensions with low POFA loading ($< 55 \text{ wt}\%$). However, the suspension viscosity increased drastically with a further increase in POFA loading when the POFA loading exceeded the critical value ($55 \text{ wt}\%$). When the POFA loading was beyond the critical value, there was limited free liquid (solvent) and the particles could easily mingle with each other in the suspension creating frictional interactions, which resulted in a sharp increase in suspension viscosity [28]. A similar trend was also observed by Liu et al. [29] who reported that a sharp increase in

nickel oxide/yttria-stabilized zirconia ceramic suspension viscosity occurred with further increase in solid loading when the solid loading exceeded the critical value of 75.6 wt%.

Past studies have witnessed that suspension viscosity is the dominating factor that affects the morphology of the CHFM prepared via phase inversion technique [18, 29, 30]. Often, suspension viscosity can also affect the spinnability of the suspension [31]. In this part of the study, CHFMs were prepared with different POFA loadings ranging from 40 wt% to 55 wt%. These membranes are denoted as C-P40, C-P45, C-P50, and C-P55 based on the POFA loading used. Meanwhile, the suspension with 60 wt% POFA loading was too viscous to extrude through the spinneret during the hollow fiber forming process.

Figure 2 shows the SEM images of the cross-sections and surface morphologies of the CHFM prepared with different POFA loadings that sintered at 1050°C. C-P40 derived from the lowest solid loading exhibited a sandwich structure, which was a thin central sponge-like layer between two finger-like structures, as shown in **Fig. 2A2**. The thicknesses of the finger-like near the inner and outer regions were approximately 52% and 42% of the overall wall thickness, respectively. When the POFA loading was increased to 45 wt%, the finger-like structure near the outer region disappeared, while the finger-like macro-voids layer near the inner region extended to 53% of the overall wall thickness. However, the thickness of the finger-like macro-voids layer decreased to 51% and 39% of the overall wall thickness when the POFA loading was increased to 50 wt% and 55 wt%, respectively. Additionally, the width of the finger-like voids also decreased with increasing POFA loading.

The morphological difference in CHFM derived from different POFA loading can be explained with a viscous fingering mechanism. As the fiber left the spinneret to travel in the 5 cm air gap, the exposure of the fiber to the atmosphere resulted in the simultaneous solvent evaporation and non-solvent (moisture in the atmosphere) condensation, causing a rapid increase in the local viscosity at the outer surface of the fiber before immersion in the coagulant bath [18, 32]. The increase in the viscosity of the outer fiber surface inhibited the viscous fingering phenomenon and the subsequent growth of the finger-like voids at the outer region of the fiber. Based on Fig. 1, the suspension viscosity was higher with increasing POFA loading, which consequently suppressed the viscous fingering phenomenon. As a result, the finger-like structure was not seen in the outer region for the CHFM derived with the POFA of 45 wt% and above.

The thickness of the finger-like structure near the inner region of C-P45 increased to 53% of the overall wall thickness as the invading micro-streams from the inner surface experienced less momentum loss when the finger-like formation at the outer surface was prevented. According to García-Fernández et al. [5], the invading non-solvent micro-streams would lose momentum when two sets of micro-streams met from the opposite directions, resulting in thinner finger-like structures originating from both surfaces with a sponge-like layer sandwiched in between them. Meanwhile, the thickness of the finger-like structure near the inner region decreased when the POFA loading was further increased from 45 wt% to 55 wt%, which was due to higher suspension viscosity that posed greater resistance and friction against the invading non-solvent micro-streams. Simultaneously, the increase in the POFA loading also led to a lower polymer/POFA particle ratio which reduced the polymer coverage of the POFA particle in the suspension.

Consequently, the POFA particle might aggregate and hinder the consolidation of the finger-like micro-channels into larger finger-like macro-voids [29]. Therefore, the width of the finger-like voids decreased with increasing POFA loading.

Interestingly, the sponge-like layer of the POFA-derived CHFM was relatively less dense as compared to other CHFM derived from pure metal oxides [5, 29, 31]. Such a loosely packed sponge-like structure is commonly found in the CHFM derived from waste ash due to large particle sizes [11, 33]. Generally, the waste ash can exhibit the particle sizes of larger than 10 μm , whereas the sizes of pure metal oxide particles are much finer (usually about 1 μm) [5, 18, 34, 35]. The particle size distribution curve in Fig. 3 shows that the majority of the POFA particles exhibited the sizes ranging from 2.8 μm to 25.8 μm with a mean size of 8.0 μm . The large particle size of POFA resulted in a less dense sponge-like structure due to longer interparticle distance. Moreover, the notable content of residual organic matters such as carbon could also be a reason that led to the less dense sponge-like structure in the POFA-derived CHFM. During sintering, some voids were created in the membrane structure due to the oxidation of the organic matters which resulted in larger membrane pore size [12].

Table 3 shows the bending strength of the CHFM derived from different POFA loadings. The bending strength of the membranes increased from 59.7 MPa to 98.1 MPa when the POFA loading was increased from 40 wt% to 55 wt%. C-P40 exhibited the lowest bending strength due to the extensive finger-like structures near the inner and outer regions of the membranes with only a thin sponge-like layer sandwiched in between them. The sponge-like layer was thicker for the CHFM derived from higher POFA loading which bestowed them with enhanced mechanical strength. In addition, the increase in the mass of POFA particles at higher loading also contributed to ameliorating the bending strength properties of CHFM. This can be explained by the mass transport mechanism during the sintering process [19]. As commonly known, the sintering of ceramic particles commenced at the contact areas of the particles [36, 37]. At higher POFA loading, the particles were more densely arranged and had more contact points with neighboring particles which prompted more extensive neck growth between the particles. This created bonds between the particles that consolidated the strength of the POFA particles, thereby enhancing the mechanical properties of CHFM.

Table 3
Bending strength of the CHFMs derived from different POFA loading (n = 3)

Membrane	Bending strength (MPa)
C-P40	59.7 $\pm$ 3.3
C-P45	75.5 $\pm$ 1.7
C-P50	83.9 $\pm$ 2.2
C-P55	98.1 $\pm$ 1.8

3.1.2 Effect of air gap distance

In this part of the study, POFA-derived CHFMs were fabricated using 55 wt% POFA loading due to the highest bending strength achieved in the previous section. High mechanical strength is essential for CHFM to ensure that the membranes can be assembled and packed into the modules without breaking [2]. The literature has revealed that the extremely low air gap distance, such as 0 cm, leads to the formation of a sandwich structure with a thin central sponge-like layer in between finger-like structures originating from both inner and outer surfaces [18, 29]. As discussed in the section earlier, the local viscosity at the outer surface of the fiber increases when traveling through the air gap before contacting the coagulant bath. When no air gap is applied, the fiber falls directly into the coagulant bath without experiencing an increase in local viscosity at the outer surface of the fiber, which favors the growth of a finger-like structure near the outer region of the fiber. The extensive finger-like structure at both inner and outer regions of the membrane is detrimental to the mechanical strength of CHFM. Hence, in this section, the fibers were spun with air gap distances of 5 cm, 7 cm, 10 cm, and 15 cm, followed by sintering at 1050°C; they are represented as C-A5, C-A7, C-A10, and C-A15, respectively depending on the air gap distance applied. It is noted that C-A5 is the same as C-P55 in the previous section.

Based on **Fig. 4A2 to D2**, the thickness of the finger-like macro-voids structure increased with increasing air gap distance. The thickness of the finger-like macro-voids structure slightly increased from 39–41% when the air gap distance was increased from 5 cm to 7 cm. C-A15 exhibited the thickest finger-like macro-voids structure with it sprawling across 71% of the overall wall thickness. Moreover, the width of the finger-like macro-voids also increased with the increase in air gap distance. As displayed in **Fig. 4A1 to D1**, the outer diameter and wall thickness decreased with increasing air gap distance. The diameter of CHFM decreased from 1.46 ± 0.02 mm to 1.25 ± 0.06 mm when the air gap distance was increased from 5 cm to 10 cm. C-A15 which was fabricated with the longest air gap distance demonstrated the smallest external diameter of 1.16 ± 0.04 mm. Furthermore, the membrane wall thickness also decreased from 210 μ m to 149 μ m when the air gap distance was increased from 5 cm to 15 cm.

The variation in the length of the finger-like macro-voids structure originating from the inner surface was caused by the difference in the contact period between the bore fluid and hollow fibers. Longer air gap distance provided more time for the invading micro-streams to penetrate further into the fiber, resulting in a thicker finger-like structure [7, 18]. The fiber also experienced the circumferential stress exerted by the bore fluid for longer duration at higher air gap distance, thereby causing the width of the finger-like macro-voids to increase [19]. The long exposure to the circumferential stress by the bore fluid also reduced the membrane wall thickness. Concurrently, at higher air gap distance, the fiber also faced greater longitudinal stress by gravity due to long distance between the spinneret and coagulant bath, which resulted in the reduction in wall thickness and external diameter of the membrane, as indicated in **Fig. 4A1 to D1** [38, 39].

Table 4 summarizes the bending strength of the POFA-derived CHFM spun at various air gap distances. The bending strength of the membranes decreased with increasing air gap distance. C-A5 had the strongest bending strength with a value of 98.1 MPa. The bending strength dropped to 88.5 MPa when the air gap distance was increased to 7 cm. A significant decrease in bending strength to 66.6 MPa was

observed when the air gap distance was further increased to 10 cm. The bending strength then slightly reduced to 64.5 MPa for the CHFMs spun at a 15 cm air gap distance. The decrease in bending strength of CHFMs with increasing air gap distance was most likely attributed to the formations of thicker finger-like macro-voids structure and thinner membrane wall.

Table 4
Bending strength of the CHFMs spun at different air gap distance (n = 3)

Membrane	Bending strength (MPa)
C-A5	98.1 ± 1.8
C-A7	88.5 ± 1.6
C-A10	66.6 ± 0.5
C-A15	64.5 ± 1.4

3.1.3 Effect of bore fluid flow rate

To study the effect of bore fluid flow rate, POFA-derived CHFMs precursors were prepared using the suspensions with 55 wt% POFA loading and spun at the air gap distance of 5 cm with different bore flow rates of 6 mL/min, 9 mL/min, 12 mL/min, 15 mL/min and 20 mL/min. The precursors were then sintered at 1050°C to acquire CHFMs. These membranes are denoted as C-B6, C-B9, C-B12, C-B15, and C-B20 based on the bore fluid flow rates applied for spinning. Again, it is noted that C-B9 is the same as C-A5 in the previous section, which demonstrated the strongest bending strength.

Based on **Fig. 5A1**, the deformation of the lumen could be observed when the bore fluid flow rate of 6 mL/min was used. During phase inversion, the rate of solvent diffusing out of the ceramic suspension is faster than that of coagulant (water) diffusing into the ceramic suspension, which results in the shrinkage of hollow fiber [40]. The shrinkage of the hollow fiber creates a large inward radial force towards the lumen side. At the same time, the ballooning effect of the bore fluid in the lumen helps to retain the uniform shape of the lumen. In this study, the lumen deformed when a low bore fluid flow rate (6 mL/min) was applied due to insufficient hydrodynamic force generated by the bore fluid against the relatively large inward radial force [7]. In addition, the de-mixing process was also afflicted by the low bore fluid flow rate which led to the partial solidification of polymer near the inner surface of the fiber. The partially solidified polymer was not strong enough to withstand the inward radial force, thereby resulting in the deformation of the lumen [7]. With increasing bore fluid flow rate to 9 mL/min, a more rigid lumen surface was formed due to the rapid de-mixing process and complete solidification of polymer near the inner surface of the fiber. As a result, the uniform round-shaped lumen was obtained as illustrated in **Fig. 5B1**.

The lumen diameter of CHFMs increased gradually from 980 µm to 999 µm when the bore fluid flow rate was increased from 9 mL/min to 15 mL/min (**Fig. 5B1** and **D1**). Furthermore, C-B20 exhibited the largest lumen diameter of 1.53 mm (**Fig. 5E1**). Meanwhile, there was no obvious increase in the external diameter

of CHFM when the bore fluid flow rate was increased from 9 mL/min to 15 mL/min, but it increased substantially at the bore fluid flow rate of 20 mL/min. Besides, the membrane wall thickness also decreased from 210 μm to 175 μm with the increase of bore fluid flow rate from 9 mL/min to 20 mL/min. This could be explained by the large hydrodynamic force exerted by the bore fluid that pushed the ceramic suspension outward radially. As a result, the membrane acquired a larger lumen diameter with a thinner membrane wall when a higher bore fluid flow rate was applied. For C-B20, the bore fluid flow rate was too high that the bore fluid pushed the ceramic suspension outward rapidly, which resulted in the formation of the extremely large lumen and external diameter with a thin membrane wall.

As shown in the higher magnification (400x) SEM images, the thickness of the finger-like macro-voids structure near the inner surface increased with increasing bore fluid flow rates (**Fig. 5A2 to E2**). The finger-like macro-voids structure increased in thickness and occupied 59% of the overall wall thickness when the bore fluid flow rate was increased from 9 mL/min to 15 mL/min. The membrane prepared with the bore fluid flow rate of 20 mL/min possessed the largest finger-like macro-voids structure of approximately 60% of the overall membrane wall thickness. At high bore fluid flow rate, the fast-moving bore fluid helped to remove the exchanged solvent accumulated near the coagulant-ceramic suspension interface at the lumen side. The removal of the accumulated exchanged solvent could maintain the initial concentration gradient of the solvent across the coagulant-ceramic suspension interface, which enhanced the solvent and non-solvent exchange rate, thereby resulting in thicker finger-like macro-voids structure [5].

As expected, the bending strength of the CHFM decreased with increasing bore fluid flow rates as shown in Table 5. The bending strength of the membranes decreased from 98.1 MPa to 61.9 MPa when the bore fluid flow rate was increased from 9 mL/min to 20 mL/min. Similarly, the reduction in the bending strength of the membranes was mainly due to the increase in finger-like structure thickness and thinner membrane wall. Interestingly, despite having a thicker membrane wall, C-B6 exhibited a bending strength of 81.8 MPa, which was lower than that of C-B9. This could be due to the deformed lumen structure that afflicted the membrane mechanical properties.

Table 5
Bending strength of the CHFMs prepared at different bore fluid flow rates (n = 3)

Membrane	Bending strength (MPa)
C-B6	81.8 ± 0.7
C-B9	98.1 ± 1.8
C-B12	75.6 ± 1.2
C-B15	69.1 ± 1.3
C-B20	61.9 ± 1.1

3.2 Effect of sintering temperature on membrane properties

3.2.1 Microstructure and surface properties

The hollow fiber precursors were prepared using the suspensions with 55 wt% POFA loading and spun at 5 cm air gap distance and 9 mL/min bore fluid flow rate. As indicated in Table 1, POFA contained a substantial amount of K_2O (6.4 wt%). The melting point of K_2O is 740°C, which is much lower than the sintering temperature of POFA, hence, it could act as an intrinsic liquid phase sintering aid to improve the sintering behaviors of POFA. Hence, a small range of sintering temperatures of 1 025°C to 1 100°C was selected for this part of the study. The membranes were sintered at 1 025°C, 1 050°C, 1 075°C, and 1 100°C, which are labeled as C-S1, C-S2, C-S3, and C-S4, respectively.

Figure 6 displays the cross-section and outer surface morphologies of the CHFM sintered at different temperatures. As mentioned earlier, the POFA used in this study demonstrated a broad particle size distribution ranging from 2.80 μm to 25.8 μm . At a low sintering temperature of 1 025°C, neck formation only occurred between small grains, whereas the large grains did not experience any observable neck growth (Fig. 5.6A1). As the sintering temperature was increased to 1 050°C, significant grain growth occurred between the large grains (Fig. 5.6B1 and B2). At this temperature, the grains grew and consumed the adjacent grains to form larger grains, which resulted in a decrease in grain number. A network of interconnected grains was formed when the sintering temperature was increased to 1 075°C (Fig. 5.6C1). The neck growth and grain diffusion were more vigorous for the membrane sintered at 1 100°C as could be seen from the interconnected grain structure with larger grain size and thicker neck at the membrane cross-section (Fig. 5.6D1).

The rapid change in the microstructure of POFA-derived CHFM at a small sintering temperatures range of 1 025°C to 1 100°C was attributed to the presence of K_2O which acted as an intrinsic liquid phase sintering aid. In our previous work, the SEM images demonstrated that the surface of the POFA particles started to become smooth and the particle grew larger when the pre-treatment temperature was increased to 700°C [12]. The findings suggested that K_2O had melted at the temperature of 700°C and facilitated the diffusion between particles. During the membrane sintering process at elevated temperatures, the POFA grains were enveloped by the liquid phase K_2O . The K_2O liquid could adhere to the adjacent grains via capillarity which increased the contact area between the grains, thereby facilitating mass transport and densification [41, 42].

The liquid phase aided membrane mass transport process can be explained by three mechanisms, namely contact flattening, dissolution of small grains, and solid-state bonding [43]. In the contact flattening mechanism, the grains were pulled together due to the capillary pressure and surface tension of the K_2O liquid. Due to the capillary stress at the contact area between the grains, the solid dissolved in the liquid and was transported to the surface of the neck [43, 44]. The contact flattening tended to occur at small grains due to large contact stresses, which also explained the neck growth at the small grains at the sintering temperature of 1 025°C. At the temperature of 1 050°C, the mass transport in the way that the small grains dissolved in the liquid K_2O and reprecipitated on the large grains. As a result, large grains

grew larger while the small grains were eliminated, as depicted in **Fig. 6B1**. The solid-state bonding mechanism might occur when the sintering temperature was further increased to 1 075°C and 1 100°C. This mechanism involved the grain growth by diffusion along the liquid wetted grain boundary [45, 46]. At this stage, the membrane structure experienced densification and shrinkage.

From the SEM image in **Fig. 6A2**, it could be seen that the outer surface of the membrane was rough after sintering at 1 050°C. This result was further confirmed by the AFM image that showed a mass of small peaks (brighter region) and valleys (darker region) with a mean surface roughness (R_a) of 0.340 μm (**Fig. 7A**). It is noted that the black spots in the AFM images indicate the membrane pores. The number of black spots (pores) on the membrane surface decreased for the membranes sintered at higher temperatures, signifying lower surface porosity which was consistent with the SEM images of the outer membrane surface (**Fig. 6A2 to D2**). The R_a value increased considerably to 0.379 μm when the sintering temperature was increased to 1 050°C. According to Talebi et al. [47], the mean surface roughness of the film would increase when the grain growth had started. In their study, the R_a values of the yttria-stabilized zirconia electrolyte film decreased from 45.3 to 26.7 nm when the sintering temperature was increased from 1 300°C to 1 400°C. However, due to the grain growth phenomenon, the R_a value bounced to 48.7 nm for the film sintered at 1 500°C. In this study, significant grain growth between particles of different sizes started to occur at 1 050°C, which has been indicated in **Fig. 6B1**. However, the surface of the membrane sintered at 1 075°C showed a lower R_a value of 0.336 μm due to the more rapid effective migration of surface grains by diffusion, leading to a smoother membrane surface. A further increase of sintering temperature to 1 100°C caused the membrane surface to become very smooth and almost dense with very few pores (**Fig. 6D2**), resulting in the lowest R_a value of 0.303 μm (**Fig. 7D**).

3.2.2 Radial shrinkage and bending properties of membranes

Figure 8 illustrates the overall morphology of CHFM before and after sintering at various temperatures. As compared to the hollow fiber precursor, the overall diameter of CHFM decreased after sintering due to the thermal/oxidative decomposition of the membrane precursor during the sintering process. Moreover, the radial shrinkage of CHFM also increased with increasing sintering temperature. Based on Table 6, C-S1 experienced a radial shrinkage of 14.4% compared to that of the hollow fiber precursor. The radial shrinkage of CHFM increased to 16.0% and 18.2% for C-S2 and C-S3, respectively. The radial shrinkage of CHFM was the highest when the sintering temperature was increased to 1 100°C, with a value of 22.9% due to the rapid membrane densification as indicated in **Fig. 6D**.

The bending strength of CHFM increased with the increase in sintering temperature (Table 6). The bending strength was the lowest for the CHFM sintered at 1 025°C with a value of 75.9 MPa. Based on **Fig. 6A1**, the neck formation only occurred among the small particles, whereas the large particles had yet to undergo any observable neck growth, which afflicted the mechanical properties of CHFM. The grain growth started to occur at the sintering temperature of 1 050°C, which led to an increase in the bending

strength to 98.1 MPa. A further increase of sintering temperature to 1 075°C promoted the densification of the membrane, consequently provided an enhanced bending strength. C-S4 demonstrated the highest bending strength of 112.5 MPa due to the most densified membrane structure (**Fig. 6D1 and D2**), which bestowed the membrane with enhanced mechanical properties.

Table 6
Radial shrinkage and bending strength of the CHFMs after sintering at different temperatures (n = 3)

Membrane	Radial shrinkage (%)	Bending strength (MPa)
C-S1	7.2 ± 0.9	75.9 ± 4.0
C-S2	8.6 ± 0.7	98.1 ± 1.8
C-S3	18.2 ± 0.2	102.7 ± 1.7
C-S4	22.9 ± 0.4	112.5 ± 1.8

3.2.3 Overall pore size distribution, porosity, tortuosity, and permeation performance

The overall PSD, porosity, and tortuosity factor of the CHFM sintered at different temperatures were determined via MIP. Based on Fig. 9A, the PSD curves show bimodal peaks which indicated the asymmetric structure of finger-like macro-voids and sponge-like layers. For C-S1, the two significant peaks at 1.05 µm and 2.09 µm were attributed to the sponge-like pores and finger-like macro-voids. The pore size of the sponge-like layer increased from 1.05 µm to 1.33 µm with increasing sintering temperature from 1 025°C to 1 100°C. The increase in sintering temperature accelerated the neck formation between the large grains, which prevented the large pore from collapsing [7]. Simultaneously, the small pores were eliminated due to the dissolution of small grains in the liquid K₂O to reprecipitate on the larger grains. A similar trend was also observed by Li et al. [7] who reported that the pore size of the alumina-mullite composite CHFM increased with increasing sintering temperature. Based on the PSD results, the CHFM developed in this study can be categorized as microfiltration membranes. Hence, these membranes can be used for microfiltration applications, such as oil/water separation and removal of bacteria from water.

C-S1 exhibited a high porosity of 39.2% and the porosity gradually decreased to 36.5% for C-S2 (Fig. 9B). A drastic drop in porosity occurred when the sintering temperature was 1 075°C. C-S4 demonstrated the lowest porosity of 16.4% due to the densest membrane structure after sintering. From Fig. 9B, it could be seen that the tortuosity factor increased for the CHFM sintered at higher temperatures which suggested that higher sintering temperature induced the formation of more complex pore channels. An abrupt surge in tortuosity factor from 2.06 to 2.14 was observed when the sintering temperature was increased from 1 050°C to 1 075°C. The vigorous neck growth and grain diffusion phenomena during the sintering process altered the pore channels which resulted in more tortuous pore structures.

As shown in Fig. 10, the PWF of the CHFM was influenced by the membrane porosity and tortuosity. The low porosity would limit the passage of the mass transfer across the membrane, while high tortuosity would create a long path and high resistance against the mass transport. C-S1, which had the most porous membrane structure and the lowest tortuosity, demonstrated the highest flux of 289.6 L/m²h at 2 bar operating pressure. Meanwhile, grain growth started to occur at the sintering temperature of 1050°C, which reduced the membrane porosity and increased the tortuosity. As a result, the PWF performance of C-S2 was afflicted and reduced drastically to 123.2 L/m²h. The PWF further dropped to 59.4 L/m²h and 12.1 L/m²h for the CHFM sintered at 1075°C and 1100°C, respectively. The findings of the PWF performance suggested that the CHFM sintered at 1050°C exhibited the optimum properties in terms of excellent mechanical strength of close to 100 MPa with high PWF.

3.3 Properties comparison with other alternative ceramic hollow fiber membranes

Table 7 summarizes the properties of POFA-derived CHFM and other alternative CHFM reported in the literature. The POFA-derived CHFM developed in this study showed relatively low PWF compared to alternative counterparts mainly due to the lower membrane porosity. Moreover, it could also be observed that the POFA-derived CHFM obtained high bending strengths of ≥ 75.9 MPa, which was higher than the silica-based CHFM reported in the literature, such as sugarcane bagasse ash, rice husk ash, corncob ash, ball clay, waste fly ash, kaolin, natural zeolite and corncob ash/metakaolin (Table 7). Interestingly, the POFA-derived CHFM also acquired such high bending strengths at comparatively low sintering temperatures than the high-strength bauxite and metakaolin CHFM, which required higher sintering temperatures of $\geq 1200^\circ\text{C}$ to attain high bending strength. For POFA-derived CHFM, the relatively low requirement of sintering temperature was attributed to the notable K₂O content (6.4 wt%) that acted as an intrinsic sintering aid. At elevated sintering temperature, the liquid phase K₂O promoted the neck and grain growth phenomena which contributed to lower the sintering temperature [42]. The relatively low sintering temperature of POFA-derived CHFM is advantageous in minimizing the energy consumption and sintering duration, which could make it more economically attractive compared to its CHFM counterparts.

Table 7

Comparison between the properties of POFA-derived CHFMs with other alternative CHFMs from literature

Membrane	Sintering temperature (°C)	Pore size (μm)	Porosity (%)	Bending strength (MPa)	PWF (L/m ² h)	Reference
POFA	1 025–1 100	1.05–2.09	16.4–39.2	75.9–112.5	12.0–289.6	This work
Bauxite	1 200–1 350	0.7–0.9	–	24.0–188.3	–	[7]
Metakaolin	1 200–1 500	0.007–0.15	9.2–27.2	117.7–187.9	–	[48]
Kaolin/alumina	1 400	0.29	61.8	87.4	–	[49]
Sugarcane bagasse ash	1 000–1 300	1.85–3.87	42.3–68.3	44.1–68.1	457–540	[11]
Rice husk ash	1 200–1 400	1.63–3.70	62.1–66.3	42.2–90.3	300–330	[9]
Corncob ash	800–1 100	–	–	9.9–31.4	–	[50]
Ball clay	1 250	0.61	50.5	55.8	1 286	[10]
Alumina dross	1 250–1 300	0.49–0.55	4.2–25.0	25.7	17.6–84.0	[51]
Alumina/waste of quartzite	1 100–1 500	–	6.6–27.3	25.0–82.6	4.4–263.2	[52]
Waste fly ash	1 150–1 400	–	–	13.0–128.0	51.0–478.0	[53]
Kaolin	1 200–1 500	0.21–1.51	7.8–69.1	5.6–61.9	–	[8]
Natural zeolite	900–1 150	–	–	10.3–63.5	228.1–828.1	[54]
Corncob ash/metakaolin	1 200	10.38	62.0	41.6	1 159	[34]

4 Conclusions

In this study, low-cost green CHFM were successfully fabricated from POFA and designed in a predetermined manner via phase inversion-based extrusion/sintering techniques for water filtration applications. The morphology structures of CHFM were studied by manipulating the phase inversion parameters such as suspension viscosity, air gap distance, and bore fluid flow rate. Particularly, the finger-like macro-void structure diminished when the suspension viscosity increased due to greater resistance and friction against the invading micro-streams. The length of the finger-like structure could be increased by increasing the air gap distance as it provided longer contact time between the hollow fibers with the

bore fluid before the fibers were being submerged into the coagulant bath. In addition, higher air gap distance also caused the hollow fiber to experience larger circumferential stress caused by the bore fluid, as well as greater longitudinal stress by the gravitational pull when falling from the spinneret into the coagulant bath, resulting in the thinner wall and smaller fiber diameter. Moreover, the bore fluid flow rate could also affect the morphology of CHFM. A lower bore fluid flow rate resulted in the deformation of the fiber lumen, whereas a higher bore fluid flow rate led to a larger lumen diameter due to larger hydrodynamic force against the ceramic suspension during spinning. It was also found that the membrane porosity decreased while the membrane pore size and tortuosity increased with an increase in sintering temperature. Due to lower porosity and higher tortuosity, the CHFM sintered at higher temperatures demonstrated lower PWF. The CHFM fabricated with a POFA loading of 55 wt%, air gap distance of 5 cm, bore fluid flow rate of 9 mL/min and sintering temperature of 1050°C showed the optimum properties for water filtration applications with an excellent mechanical strength of 98.1 MPa and high PWF of 123.2 L/m²h.

Lastly, the properties of the POFA-derived CHFM were compared with other alternative CHFM reported in the literature. The POFA-derived CHFMs demonstrated excellent bending strengths of > 75 MPa at comparatively low sintering temperatures. This was attributed to the significant amount of K₂O in the POFA composition which acted as the low melting point sintering aid. The relatively low sintering temperature of POFA-derived CHFM would reduce the energy consumption and sintering duration, which could potentially be more economically attractive compared to their ceramic counterparts.

Declarations

Acknowledgments

The authors gratefully acknowledge the financial support from the Ministry of Education Malaysia under the Higher Institution Centre of Excellence Scheme (Project Number: R.J090301.7846.4J193) and Long-Term Research Grant Scheme (Project Number: R.J130000.7809.4L895), and also Universiti Teknologi Malaysia under the Transdisciplinary Research Grant (Project number: Q.J130000.3509.05G75), Malaysia Research University Network (MRUN) Grant (Project number: R.J130000.7809.4L867), Higher Institution Centre of Excellence (HICoE) Research Grant (Project number: R.J090301.7851.4J426), and UTM GUP Tier 1 (Project number: Q.J130000.2546.19H18). The authors would also like to thank the Research Management Centre (RMC), Universiti Teknologi Malaysia for the technical support and services provided.

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Figures

Figure 1

Rheological properties of POFA suspensions: (A) viscosity versus shear rate; (B) viscosity versus POFA loading at the shear rate of 25 s^{-1}

Figure 2

(1) Overall morphology (80x magnification) and (2) cross-sections (400x magnification) of (A) C-P40, (B) C-P45, (C) C-P50 and (D) C-P55 where the area bounded by yellow lines refers to finger-like structure (air gap distance: 5 cm; bore fluid flow rate: 9 mL/min; sintering temperature: 1 050°C)

Figure 3

Particle size distribution of POFA

Figure 4

(1) Overall morphology (80x magnification) and (2) cross-sections (400x magnification) of (A) C-A5, (B) C-A7, (C) C-A10 and (D) C-A15 where the area bounded by yellow lines refers to finger-like structure (POFA loading: 55 wt%; bore fluid flow rate: 9 mL/min; sintering temperature: 1 050°C)

Figure 5

(1) Overall morphology (60x magnification) and (2) cross-sections (400x magnification) of (A) C-B6, (B) C-B9, (C) C-B12, (D) C-B15 and (E) C-B20 where the area bounded by yellow lines refers to finger-like structure (POFA loading: 55 wt%; air gap distance: 5 cm; sintering temperature: 1 050°C)

Figure 6

(1) Cross-sections (1,800x magnification) and (2) outer surface (2,000x magnification) of (A) C-S1, (B) C-S2, (C) C-S3 and (D) C-S4 (POFA loading: 55 wt%; air gap distance: 5 cm; bore fluid flow rate: 9 mL/min)

Figure 7

AFM images of the outer surface of (A) C-S1, (B) C-S2, (C) C-S3 and (D) C-S4

Figure 8

Overall morphology (60x magnification) of (A) hollow fiber precursor, (B) C-S1, (C) C-S2, (D) C-S3 and (E) C-S4 (POFA loading: 55 wt%; air gap distance: 5 cm; bore fluid flow rate: 9 mL/min)

Figure 9

(A) PSD and (B) porosity and tortuosity factor of the CHFM sintered at different temperatures

Figure 10

PWF of the CHFM sintered at different temperatures (Operating pressure: 2 bar, n=3)