

Fabrication of Low Cost Alumina Tube through Agar Gelcasting for Membrane Microfiltration

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ABSTRACT

Tubular Al₂O₃ membranes for microfiltration were successfully fabricated by combined steps of agar gelcasting and then acetone-assisted drying. Firstly, Al₂O₃ slurry and agar solution were separately prepared prior to being mixed together at a constant temperature of 70°C. Subsequently, the warm mixtures were poured into assembled glass mold and then rapidly transformed into gel with tubular shape. The as-gelled tubes were demolded and then soaked in acetone for 50 h allowing rapidly drying of the gel tubes after removal from acetone due to a lot of evaporation of acetone in air atmosphere at room temperature. The tubular Al₂O₃ membranes were prepared from Al₂O₃ powder with the significantly different particle sizes of ~5 μm and 0.167 μm at different proportions of 0.167 μm Al₂O₃ powder from 5 to 20 wt%. The tubular Al₂O₃ membranes possessed the range of linear drying and firing shrinkage of 8.5–11.5 and 0–0.75%, respectively. After sintering at 1300°C, all the tubular Al₂O₃ membranes showed pore diameters ranging from 0.1 to 10 μm. The small Al₂O₃ powder at 20 wt% content demonstrated the considerable potential as membrane due to the beginning of connected pore network from their sintering. The method of agar gelcasting combined with the acetone-assisted drying offered a new alternative for forming the tubular Al₂O₃ membranes with simplicity and economy replacing the use of expensive extruder together with particularly suitable binder.

Keywords: Agar, gelcasting, microfiltration, alumina, drying

1.0 INTRODUCTION

Porous tubular ceramic membrane has a wide range of applications in microfiltration and ultrafiltration particularly for regeneration and processing in food and biotechnology industries as well as wastewater treatment [1]. Although the ceramic membrane is quite expensive compared with polymeric membrane, it is highly demanded for the industrial

uses due to its irreplaceable properties, i.e. resistance to high thermal and severe acidic/basic conditions [2-5]. In addition, for industrial applications the tubular ceramic membrane is an effective geometry due to its capability to be arranged in a compact module, sealed effectively, and operated at higher pressure-cross flow compared with flat sheet membrane [6].

In order to fabricate the porous ceramic membrane tube, the extrusion

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method is typically used in pragmatic approach. With the extrusion method, a sophisticated extruder is needed for fabricating the defect-free tubular ceramic membrane as well as careful selection of binder and extruder operating conditions [7]. As a result, the extruded tubular ceramic membrane is mainly subject to the availability of sophisticated-system extruder with high cost. In order to tackle the issue of expensive extruder dependence, new fabrication method of tubular ceramic membrane through agar gelcasting is proposed in this work as an alternative using only assembled glass mold for shaping.

Gelcasting method is a near-net-shape forming of complex-shaped, advanced ceramic [8]. Only a small amount of polymers as binder is needed for gel-network creation strong enough to hold all the ceramic particles together. In pioneering work of gelcasting, acrylamide-based binder is preferable to other monomers. However, with its neurotoxic effect other potential monomers with low toxicity have thoroughly been investigated [9]. In order to avoid the health risk, agar being a biopolymer is applied in this work for gelcasting to fabricate the tubular ceramic membrane. One of challenging problems for applying agar in gelcasting process is the drying of as-gelled tube without both deformation and crack [10–15].

From the literature reviews, an osmotic drying liquid, i.e. polyethylene glycol (PEG) has widely been known to be suitable for drying the gelcast ceramics [10, 12, 14–16]. The solution of PEG with high molecular weight of 8000 offered more effective drying than that with low molecular weight of 1000 [14]. There was no penetration of PEG molecules with the high molecular weight through the pores within gel network allowing actively

water removal and then creating denser, homogeneous microstructure of ceramic parts.

Parameters of importance for the osmotic drying liquid included gelcast thickness and ceramic loading [15]. Both the parameters had direct effect on drying rate. Moreover, it was suggested that water removal from only some part of the gelcast body occurred. With the partial removal of water, the osmotic liquid drying was particularly used for pre-drying stage in food industries [17–22]. In order to make the drying of gelcast ceramic body completed in one step, new drying liquids have been investigated. Our investigations indicated that agar-gelcast ceramic with small thickness of ~ 2 mm was able to be dried when it was soaked in acetone [23]. However, the parameters of interest in the acetone drying, for example, the effects of gelcast thickness and agar solution concentration on acetone-soaking time, etc. needed to be thoroughly examined later.

In order to apply the new drying technique to agar gelcasting in this work, the tubular Al₂O₃ membranes were initially fabricated by agar gelcasting and the technique of acetone-assisted drying was subsequently employed. The different Al₂O₃ particle sizes of ~5 and 0.167 μm were employed using the proportions of smaller Al₂O₃ particles from 5 to 20wt% for preparing the gelcasting mixtures. The proposed proportions might offer the advantages of strength from the much smaller Al₂O₃ particles at low sintering temperature as well as the microfiltration pore size from the much larger Al₂O₃ particles. The effects of large-to-small proportions of Al₂O₃ particles on shrinkage, microstructure and pore size distribution of the agar-gelcast tubes were studied. A set of assembled glass mold was adapted for

transformation from the mixture into the gelling state of Al_2O_3 tube. The as-gelled tube was soaked in acetone at a certain period of 50 h ensuring an inter-diffusion equilibrium between acetone and water. After the as-gelled tube was removed from acetone, it was rapidly dried and then rigid due to inherently acetone evaporation at room temperature. The dried Al_2O_3 tube was sintered at 1300°C to reach a mechanical strength enough for pressure-driven membrane applications.

2.0 EXPERIMENTAL

2.1 Materials

Al_2O_3 powder with the average particle size of $\sim 5 \mu\text{m}$ (SRM 30, Hindalco Industries Limited, India) was used for preliminary experiment on fabricating the Al_2O_3 tube. Additionally, Al_2O_3 powder with the average particle size of $0.167 \mu\text{m}$ (TMDAR, Taimicron, Japan) was used for forming the tubular Al_2O_3 membrane with a useful mechanical strength. Agar powder from Chile was employed as gelling agent. Sodium salt of polyacrylic acid (Na-PAA, Sigma-Aldrich) was used as dispersant. Acetone was used as liquid drying agent.

2.2 Agar Gelcasting Combined with Drying Assisted by Soaking in Acetone

In the preliminary experiment, the Al_2O_3 powder SRM 30 was used for preparing Al_2O_3 slurry at the total solid loading of 80wt%. The dispersant (Na-PAA) was used at 0.7wt% of total solid loading. The starting materials were first ball milled in HDPE bottle for 2 h to prepare well-dispersed Al_2O_3 slurry prior to being warmed at a constant temperature of 70°C . In the subsequent preparation of agar solution

at 2wt% concentration, agar powder was dispersed in RO water for 5 min using magnetic stirrer in order to help rapidly and completely dissolve agar powder prior to boiling in closed system of beaker to prevent water evaporation.

After that, the hot agar solution was mixed together with the warm Al_2O_3 slurry using the magnetic stirrer. The mixture of Al_2O_3 slurry and agar solution was maintained at 70°C to prevent unintentional transformation of agar from solution to gel. The adding amount of agar solution was based on the final agar content at 0.5wt% of the total solid loading of the Al_2O_3 slurry. The warm Al_2O_3 mixture was poured into assembled, warm glass mold, as shown in Figure 1, to shape into the tube of Al_2O_3 mixture. Shortly after cooling the tubular Al_2O_3 mixture to room temperature, the mixture completely became gel with an enough strength to be demolded.

Finally, the as-gelled Al_2O_3 tube was soaked in acetone for 50 h, appropriate period of soaking for this work, to create inter-diffusion between the water in agar-gel network structure and the surrounding acetone. In the step of soaking, the outer glass mold was removed and then the inner glass mold with the coverage of gelling Al_2O_3 tube was put into acetone contained in glass cylinder with tight sealing of parafilm to prevent acetone evaporation as shown in Figure 2. In the closed-system soaking, acetone diffused into the agar gel structure and water diffused out of the gel structure due to concentration gradient. The mechanism of acetone-water inter-diffusion leading to drying in this work differed from that of osmotic drying in food drying technology [21-22]. After the removal of gel tube from acetone, the acetone-rich gel tube was rapidly pulled out from the inner glass mold and then left to be dried for ~ 15 min to

rapidly evaporate acetone. While the acetone was evaporating in the early 15 min, the gel Al_2O_3 tube remained able to be straightened due to highly

flexible feature of agar gel. Dried Al_2O_3 tube was eventually formed without cracking.

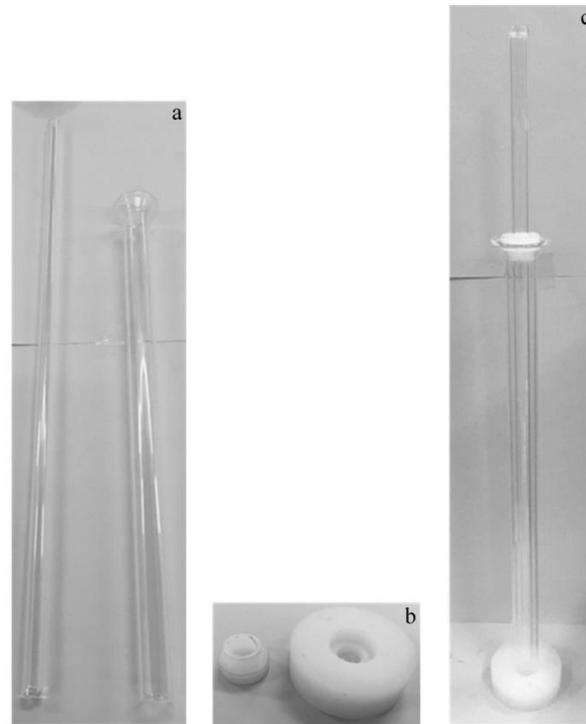


Figure 1 Images indicating (a) glass molds for agar gelcasting of both inside (left) and outside (right), (b) Teflon for covering the glass molds at both top (left) and bottom (right), and (c) assembled glass mold for gelcasting the Al_2O_3 mixtures into tube

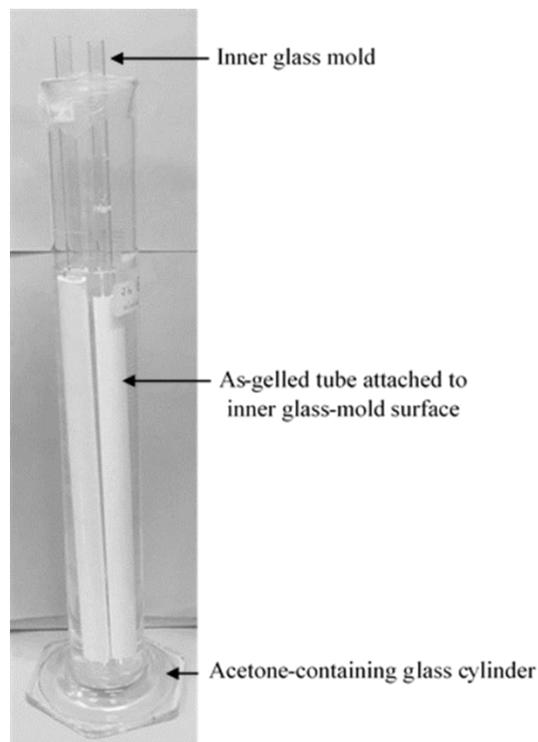


Figure 2 Water-acetone inter-diffusion drying through soaking the as-gelled Al_2O_3 tube in acetone contained in the glass cylinder

2.3 Fabrication of Tubular Al₂O₃ Membranes

All the processes of forming the tubular Al₂O₃ membranes were the same as those of forming the Al₂O₃ tube, except for the adding amount of Na-PAA and the kind of Al₂O₃ powder. Na-PAA was added at 1wt% of the total solid loading. The Al₂O₃ powders SRM 30 and TMDAR was mixed together at various proportions as named in Table 1. The tubular Al₂O₃ membranes were named 5SA, 10SA, 15SA, and 20SA following the adding amounts of the Al₂O₃ powder TMDAR (0.167 μm). The powder SRM 30 with 5 μm average particle size was used at much higher proportion to form the pore sizes in the range of microfiltration membrane—0.1 to 10 μm. Since the powder SRM 30 was unable to be sintered at low temperature the powder TMDAR with 0.167 μm average particle size was used to strengthen the gelcast tubes. After all the Al₂O₃ tubes were completely dried by the acetone evaporation, the green Al₂O₃ tubes were sintered at 1300°C for 1 h to obtain the tubular Al₂O₃ membranes with an optimum strength.

Table 1 Nomenclature of gel-casting mixtures following the proportions of used Al₂O₃ particle sizes

Sample	Proportions of Al ₂ O ₃ Powder (%)	
	TMDAR (0.167 μm)	SRM 30 (5 μm)
5SA	5	95
10SA	10	90
15SA	15	85
20SA	20	80

2.4 Characterizations

Linear drying and firing shrinkages of all the Al₂O₃ tubes (3 samples for each

composition) were calculated to obtain net dimension. The porosity of sintered tubes were determined from Archimedes' method according to ASTM C373-88. The microstructure of sintered tubes were observed with SEM (SEM-JSM5800LV, JEOL). The pore diameter of membrane tubes was measured by mercury intrusion porosimetry (Poremaster, Quantachrome Instruments) method.

3.0 RESULTS AND DISCUSSION

3.1 Preliminary Fabrication of Tubular Al₂O₃ Membrane

The Al₂O₃ tubes were fabricated by the alternative processes of gelcasting using the set of assembled glass tubes as the mold and then drying assisted by the acetone diffusion-evaporation. The different results of tube shape between the drying by virtue of putting in air at room temperature and soaking in acetone was shown in Figure 3a on right and left sides, respectively. The air drying provided the Al₂O₃ tube with cracking; however, the acetone-assisted drying provided the Al₂O₃ tube with no defect. It should be noted that the air-dried tube was dried on the inner glass mold since it needed a support to keep the circular shape of tube cross section; otherwise, without the glass support it deformed before drying completely. Figure 3b and c indicated the dried Al₂O₃ tubes with 16 cm in length and 1 cm in outer diameter, respectively.

The soaking in acetone was the critical step of successful fabrication without deformation and cracking. However, there were still a number of emerging issues related to the acetone-assisted drying needing to be explored such as the allowable thickness of agar gelcasting ceramics for thoroughly acetone diffusion into the agar gel

structure, the appropriate final content of agar in the gelcasting mixture, effective technique to control the diffusion rate of acetone replacing the

water in the agar gel structure and unequal density along the tube length. The emerging issues were not investigated yet.

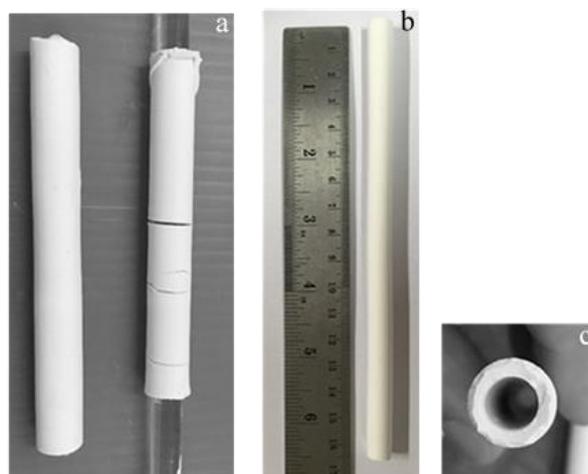


Figure 3 Photos of (a) green tubes after (left) drying by pre-soaking in acetone and (right) drying by putting in air at RT without removal from the inner glass mold to prevent the collapse of as-gelled tube, (b) sintered tubes drying by pre-soaking in acetone and (c) cross section of sintered tubes drying by pre-soaking in acetone

The acetone-assisted drying was simpler than the conventional drying of acrylamide-based gelcasting. The new drying only required the closed system of soaking in acetone for attaining acetone-water inter-diffusion equilibrium, whereas the conventional drying needed to carefully control humidity to prevent non-uniform shrinkage [8]. Moreover, the new drying was not the same as the osmosis-assisted drying in ceramic gelcasting [10, 12, 14-16] and food [21-22] processing. The osmosis-assisted drying relied on the movement of water from the gelled samples to directly a surrounding liquid such as PEG due to osmotic phenomenon.

3.2 Physical Properties of Agar Gelcast Membrane Tube

Physical property in terms of shrinkages both after drying and sintering was shown in Figure 4 (a).

The drying shrinkage was in the range between 8.5 and 11.5% and the firing shrinkage was rather small ranging from 0 to 0.75%. Therefore, the respective total shrinkage would be ~ 8.5, 12.5, 10.75 and 10.5% when the particles TMDAR increased from 5 to 20wt%, respectively. The behavior of shrinkages was governed by the drying stage of gelcasting. Moreover, sample 10SA showed the highest drying shrinkage of 11.5% and sample 15SA showed the highest firing shrinkage of 0.75%. The results of shrinkage implied that for the each individual composition, the drying shrinkage was influenced by the dispersion of Al_2O_3 particles in agar gel network; while, the firing shrinkage was controlled by the sintering mechanism at the different dispersion of Al_2O_3 particles.

Figure 4(b) showed bulk density and water absorption of all the sintered Al_2O_3 tubes. The bulk density increased with increasing the Al_2O_3

particles TMDAR from 5 to 15wt% and subsequently decreased when those particle amounts reached 20wt%. The result suggested that the sintering creating connected pore network occurred at 20wt% content of the Al_2O_3 particles TMDAR. Therefore, the sample 20SA might begun to possess the enough strength for practical use as membrane.

Furthermore, the water absorption of all the sintered Al_2O_3 tubes decreased with increasing the Al_2O_3 particles TMDAR. The linearly decreasing trend of water absorption confirmed that the small Al_2O_3 particles TMDAR were inserted among the large Al_2O_3 particles SRM 30.

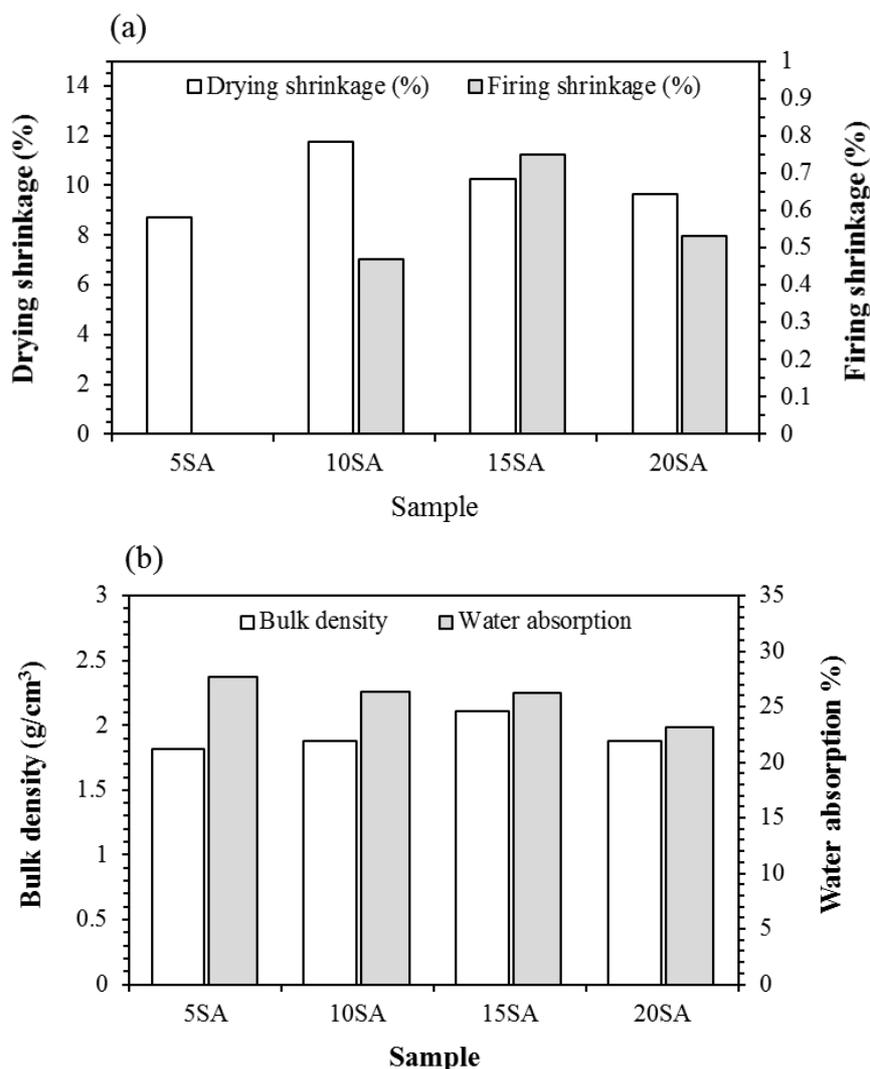


Figure 4 (a) Percentages of drying and firing shrinkage of dried and sintered tubes, respectively, and (b) bulk density and water absorption of sintered Al_2O_3 tubes with different ratios of small Al_2O_3 particles

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3.3 Membrane Microstructure

Figure 5 displayed all the microstructure of gelcast Al_2O_3 tubes after sintering at 1300°C . The images clearly showed that the sintering leading to the connected pore network occurred when the Al_2O_3 particles TMDAR reached 20wt%. Whereas, the too small amount of well-dispersed

Al_2O_3 particles TMDAR from 5 to 15wt% resulted in the gap or separation of particles larger during sintering and then the connected pore network did not occurred. It was observed that the microstructures of all the gelcast Al_2O_3 tubes corresponded with the trend of firing shrinkage and bulk density. It meant that the sintering leading to the connected pore network occurred for only the sample 20SA resulted in the decreases in firing shrinkage and bulk density. In other words, the sintering was associated with the combination of the too small Al_2O_3 particles TMDAR and then became the new larger particles of Al_2O_3 . The larger particles were also connected with each other and then creating the pore network. Therefore, the amount and dispersion of the particles TMDAR of the sample 20SA were responsible for the microstructure suitable for use as the membrane.

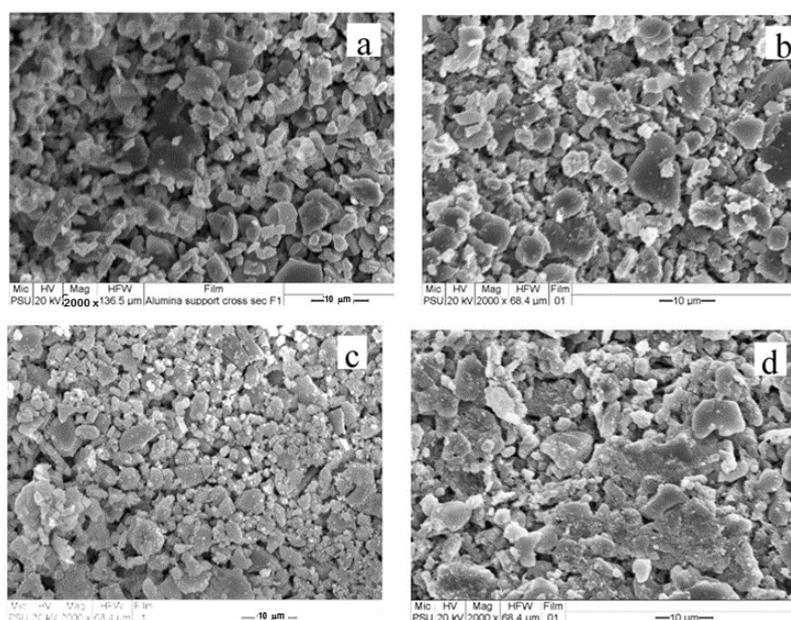


Figure 5 SEM images of sintered Al_2O_3 tubes for samples a) 5SA, (b) 10SA, (c) 15SA and (d) 20SA

3.4 Pore Size Distribution of Gelcast Membrane Tube

The pore size distribution of all the gelcast Al_2O_3 tubes after sintering at

1300°C was shown in Figure 6. The pore sizes were able to be divided into three main ranges: range of $0.02\text{--}0.03\ \mu\text{m}$, $0.1\text{--}10\ \mu\text{m}$ and $10\text{--}100\ \mu\text{m}$. The pore sizes between 0.1 and $10\ \mu\text{m}$ was

considered to be the range of microfiltration membrane created by all the gelcast Al_2O_3 tubes. On the other hand, the other two ranges were noticeably shown from only the sample 15SA. For the range of 0.02–0.03 μm , it occurred from the thorough dispersion and close contact of the Al_2O_3 particles TMDAR prior to sintering. Therefore, the quite small pore sizes were formed due to the existence of voids among the Al_2O_3 particles TMDAR sintered.

For the large pore sizes between 10–100 μm , it was attributed to defect being air bubbles trapped in the gelcast mixtures during the gelcasting. It was considered that the defect resulting from air bubbles occurred, especially for the sample 15SA since the mixture 15SA might possess the highest viscosity compared with the other mixtures. Therefore, the sample 20SA had the most potential to be used as the microfiltration membrane.

Furthermore, it was noticed that over the range of 0.1–10 μm the shape of peaks tended to distort only at the smaller pore size range of 0.1–1 μm . Therefore, it was noteworthy for focusing on the range of pore sizes as

shown in the inserted image of Figure 6. The order of decreasing the amount of the pore sizes over the range of 0.1–1 μm was found to be 5SA > 15SA > 20SA > 10SA. The result of pore size order confirmed that sample 15SA possessed the larger amount of small pores than sample 20SA. The order was directly connected with the sintering leading to connected pore network for 20wt% small Al_2O_3 particles of sample 20SA, corresponding to the results of firing shrinkage and microstructure.

From the results of pore sizes, it pointed out that using the Al_2O_3 particles with significantly different sizes of ~5 and 0.167 μm created the equal pore sizes of 0.1–10 μm governed by the size of large Al_2O_3 particles. The equal pore sizes were parallel to a report indicating that the small addition of medium and/or fine Al_2O_3 powder to the coarse one showed the hardly changed pore size distribution of microfiltration membrane supports [24]. However, the report suggested that the addition of medium Al_2O_3 particles was beneficial to their bending strength.

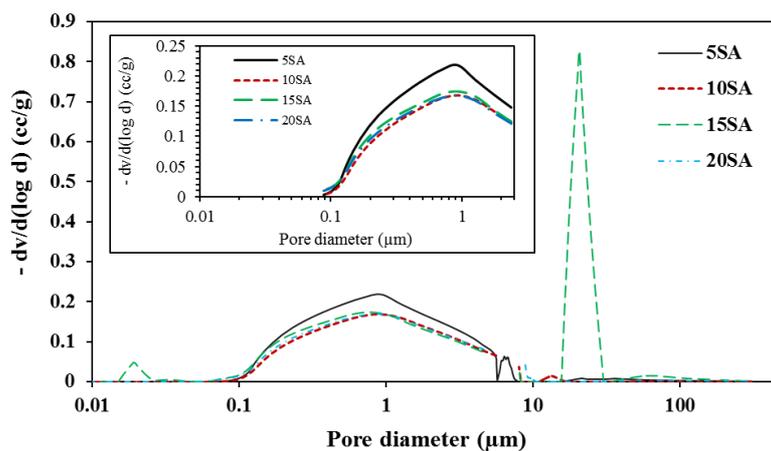


Figure 6 Pore size distribution of sintered Al_2O_3 tubes prepared with different ratios of small Al_2O_3 particle sizes from 5 to 20 wt%, inserting the image of pore size distribution between 0.1 and 1 μm

4.0 CONCLUSION

The tubular Al₂O₃ membrane for using in microfiltration was fabricated through the combination of agar gelcasting into glass mold and acetone-assisted drying. The drying by soaking the as-gelled tubes in acetone for 50 h was successfully used. The Al₂O₃ membrane tubes with no deformation and cracking were formed. The use of small (0.167 μm) to large (~5 μm) ratio of Al₂O₃ particles at 20:80 for gelcasting showed the potential for applying in microfiltration with pore sizes in the range of 0.1–10 μm. The ratio of small-to-large particles offered the pore network that was thoroughly connected in the Al₂O₃ tubes. However, the bending strength needed to be further tested prior to the permeation test.

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