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The effect of kaolin addition on the characteristics of a sintered diatomite composite support layer for potential microfiltration applications

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Abstract

Porous ceramic membranes have lately become a subject of special interest due to their outstanding thermal and chemical stability. We prepared a sintered diatomite composite support layer with the addition of kaolin, which enhanced the mechanical strength of the diatomite composite matrix while retaining acceptable permeation properties. We also discussed whether the sintered diatomite composite support layer could serve as a separation layer to minimize processing difficulties as well as the feasibility of using a sintered diatomite composite layer without a separation layer as a porous ceramic membrane for microfiltration. The pore characteristics of the sintered diatomite specimens were studied by scanning electron micrography, mercury porosimetry, and capillary flow porosimetry.

Keywords: A. Sintering; Porous materials; Microstructure

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1. Introduction

Porous ceramics are increasingly important [1] as researchers seek to exploit their unique properties, such as high wear resistance [2], low density levels [3], low thermal conductivity [4–6], and a low dielectric constant [7]. Notably, porous ceramic membranes [8–12] are among most feasible applications of porous ceramics. The driving force for the development of porous ceramic membranes is mainly the need to produce membranes with greater thermal and chemical stability, as most polymeric membranes cannot withstand operating temperature above 200 °C or exposure to organic solvents such as benzene and toluene [13].

It is generally thought that the most important features of a porous ceramic membrane are its permeation and separation properties. Therefore, precise control of the average pore size, the largest pore size, and the permeation properties, while retaining acceptable mechanical properties is important. A challenging area in ceramic membranes application is how to control, tailor, and characterize pore characteristics. Various approaches that afford control over the microstructural features of a separation layer with or without a support layer, which ultimately determines the permeation properties of porous ceramic membranes, have been assessed.

Although processing routes to produce porous ceramics are extensively documented in the literature [1], the relationship between the pore characteristics and the membrane properties of porous ceramic membranes has yet to be established. Ceramic membranes are usually composites consisting of several layers of one or more different ceramics. A porous ceramic membrane is for this reason typically fabricated in multiple steps. A support layer is initially to provide mechanical strength for the membrane, if needed, followed by the coating of one or more intermediate layers on the support layer, after which a final separation layer is deposited. Each step involves a high-temperature sintering process, making the ceramic membrane fabrication procedure expensive and difficult. If the support layer can also serve as a separation layer, it becomes possible to simplify the process while also reducing the time and cost.

Applications and separation mechanisms pertaining to porous ceramic membranes depend on the pore size of the membranes. Because it is difficult to create a support layer

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with microporous or dense materials due to their low permeation properties, it is worthwhile to investigate porous ceramic membranes with a sub-micron pore size range that consist of a single support layer. Among reverse osmosis, nanofiltration, ultrafiltration, and microfiltration methods, we focused on microfiltration in this study, as the largest and smallest pore sizes of membranes for microfiltration are roughly several μm and 0.1 μm, respectively.

Recent developments in porous ceramic membranes have heightened the need to investigate mass transport through a separation layer deposited on a support layer, as the overall permeation property and pore size reliability of the separation layer are critical to porous ceramic membrane applications. In general, the permeation properties of a separation layer on a support layer govern the overall permeation properties of the membrane. However, we attempted to prepare a well-defined support layer that also serves as a separation layer. Therefore, the permeation and separation property of the support layer are important in this study.

Although there have been various reports on commonly used materials for ceramic membranes, including γ-Al_2O_3 [11,14], α-Al_2O_3 [10,15], TiO_2 [16,17], ZrO_2 [18], SiO_2 [19], and composites of these materials [20,21], there have been few studies on porous starting particles such as diatomite. Diatomite is a sedimentary rock originating from the siliceous fossilized skeletons of diatoms, which are composed of rigid cell walls known as frustules. As documented in the literature [22–24] and in preliminary works, a diatomite support layer can be prepared such that it has both permeation and separation properties, by maintaining the sintering temperature in the range of 900 °C to 1200 °C. However, notwithstanding the outstanding properties of a diatomite support layer for potential microfiltration applications, the mechanical strength of a diatomite support layer should also be considered with regard to its use as a free-standing membrane. Although there are many complicated porosity parameters affecting the overall mechanical strength of a ceramic support layer, essentially, as the porosity of a ceramic support layer increases linearly and enhances the permeation property, the mechanical strength decreases exponentially [25]. In addition, in terms of the separation property, if there are no other processing variables excluding the sintering temperature, it is difficult to control factors affecting the separation characteristics, such as the largest pore size.

To alleviate these problems, in this study, we introduced kaolin to tailor the pore characteristics of a diatomite support layer. There are two key reasons for applying this approach. First, kaolin consists mostly of kaolinite ($Al_2Si_2O_5(OH)_4$), similar to the composition of diatomite [26], which is mainly silica (SiO_2) and a small amount of alumina (Al_2O_3) as an impurity. Second, the phase transformation and growth of mullite ($3Al_2O_3 \cdot 2SiO_2$) in kaolin appears at around 1100 °C during the sintering process [27,28]. As this mullite crystal shows a plate-like morphology and because the particle size of kaolin is smaller than that of diatomite, we speculated that kaolin particles may act as a girder between the inter-particle voids of diatomite particles. Consequently, the mechanical strength of a diatomite support layer may be increase. However, when more than a certain amount of kaolin is added, the clogging of pore channels in the diatomite matrix by kaolin particles can severely degrade the permeation properties. Nevertheless, to date, no detailed studies of the effect of an addition of kaolin on the properties of a diatomite separation layer have been published.

By taking into account the aforementioned factors, in this study we focused on a sintering temperature of 1200 °C for the diatomite composite support layers because we wanted to enhance the mechanical strength of the diatomite composite support layers such that they retain acceptable air permeation properties. In the initial stage of the experiments, we sought to rule out the processing conditions that may lead to negligible permeation properties due to an excessive amount of added kaolin, by measuring the air permeation properties. Therefore, to study the general trend of the permeation properties of diatomite composite layers with kaolin in amounts ranging from 0 wt% to 100 wt%, sintering was performed in a range of 900–1200 °C. When a certain amount of kaolin in which reasonable permeation properties were retained was determined, for further detailed study, the sintering temperature of the diatomite composite support layer was confined to 1200 °C, which was the highest practicable temperature as regards the permeation properties.

The present study investigated the several important aspects. First, we discussed whether we could enhance the mechanical strength of a diatomite composite support layer with minimum loss of the permeation properties, for application as a free-standing support layer by strengthening the diatomite matrix with additions of kaolin ranging from 0 wt% to 100 wt% and with different sintering temperatures ranging from 900 °C to 1200 °C. Second, we investigated whether a diatomite composite support layer could simultaneously serve as a separation layer by measuring the pore characteristics of the diatomite composite support layer, specifically the largest pore size and the average pore size.

2. Material and methods

Diatomite (Celite 499, Celite Korea Co. Ltd., Korea) was used for the preparation of the sintered diatomite specimens. The average particle size of the as-received diatomite and as-received kaolin were 12.79 μm and 1.53 μm, respectively. To enhance the sinterability of the diatomite particles, the average particle size of diatomite was reduced by ball-milling. Distilled water was used as a solvent and the slurry was ball-milled for 24 h with a alumina ball-to-powder volume ratio of 2:1. To incorporate kaolin into the diatomite matrix, diatomite particles at quantities ranging from 0 wt % to 100 wt% of Kaolin (Kaolin, Sigma-Aldrich, USA.) were mixed for 3 h by ball-milling with a ball-to-powder volume ratio of 0.5:1. With polyethylene glycol as a binder, the diatomite composite specimens were dry-pressed at 18.7 MPa and finally sintered at 900–1200 °C for 1 h. The flexural strengths of the sintered diatomite specimens were measured by a three-point bending test (Instron 4206, Instron, USA.) and bulk densities were calculated by measuring the dimensions and weights. The pore characteristics of the diatomite were investigated by scanning electron micrographs (JSM-5800, JEOL, Japan), and mercury porosimetry (Autopore IV 9510, Micromeritics, USA.). The air flux and pure water flux were measured by capillary flow porosimetry (CFP-1200-AEL, Porous Materials Inc., USA.). A sintered diatomite composite specimen (diameter 4 cm and thickness 0.4 cm) fitted between the O-rings in the bottom of the chamber and the bottom of the chamber insert in the capillary flow porosimetry. And then, the flux was measured by sensors automatically while incrementing the diameter of the motorized valve and the pressure of the regulator.

3. Results and discussion

The particle size distributions of diatomite ball-milled for 24 h and the as-received kaolin are shown in Fig. 1(a). The average particle sizes of the diatomite ball-milled for 24 h and the as-received kaolin were 7.43 μm and 1.53 μm, respectively. As the average particle size of kaolin is around 20% of that of diatomite, we hypothesized that plate-like kaolin particles could strengthen the roughly connected pore channels induced by irregular diatomite particles or act as a girder between inter-particle voids of diatomite particles. Thus, the mechanical strength of the diatomite support layer may be increased.

To validate this hypothesis, we initially measured the flexural strength of the diatomite composite layers. When the sintering temperature is 900 °C or 1000 °C, the flexural strength is proportional to the sintering temperature regardless of the amount of kaolin, as shown in Fig. 1(b). However, when the sintering temperature is greater than 1000 °C, the flexural strength sharply increases past this point as both the sintering temperature and the amount of kaolin are increased. This suggests that the preparation of a diatomite composite support layer with high mechanical strength is possible at a sintering temperature of 1200 °C or higher, as a slight increase in the sintering temperature dramatically increases the mechanical strength.

The flexural strength of a typical ceramic microfiltration membrane based on alumina [8] or silicon carbide [29] is often

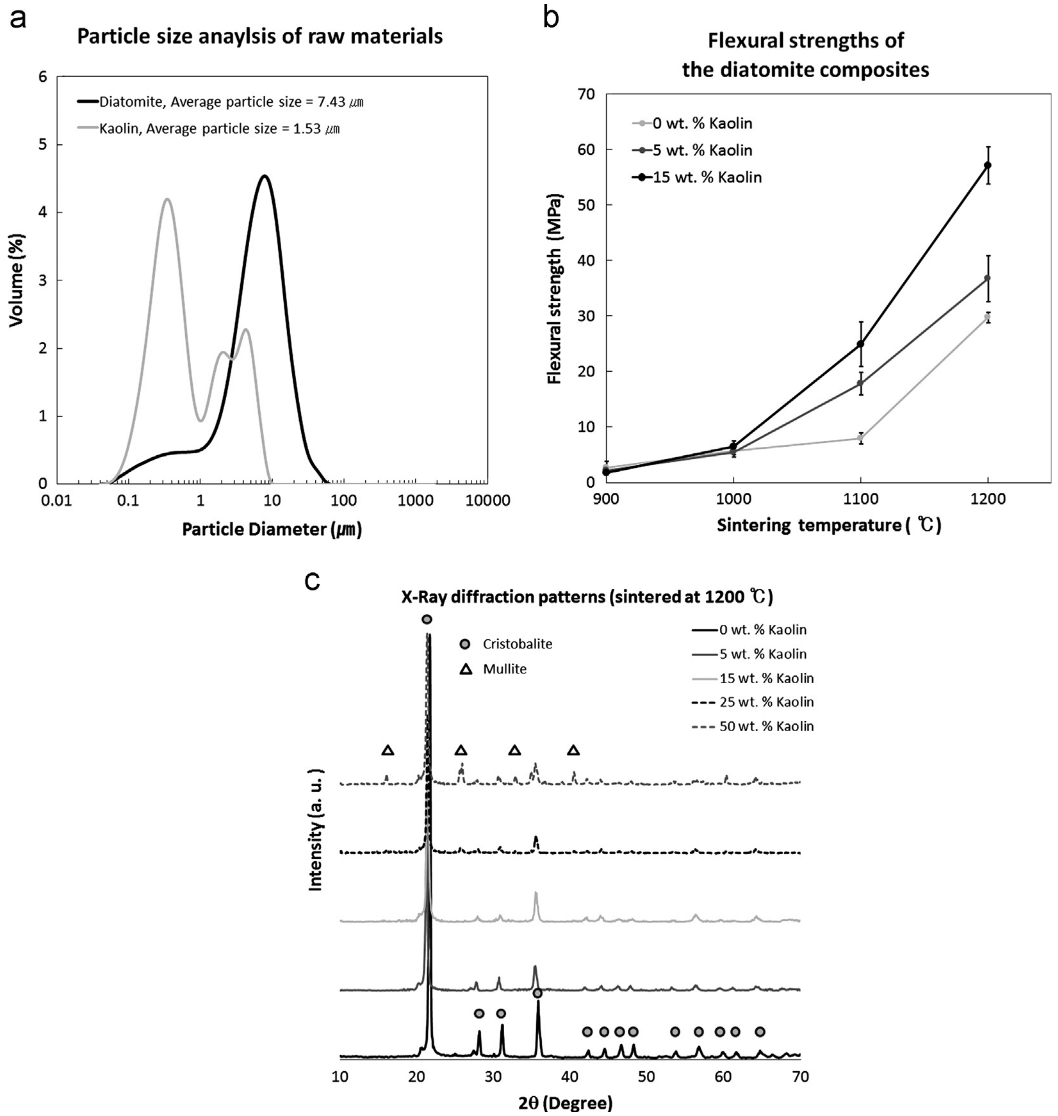

Fig. 1. (a) Particle size distributions of diatomite ball-milled for 24 h and as-received kaolin, (b) flexural strengths of diatomite support layers with addition of 0 wt % kaolin, 5 wt% kaolin, and 15 wt% kaolin sintered at temperatures ranging from 900 °C to 1200 °C for 1 h, and (c) X-ray diffraction patterns of diatomite support layers while varying the amount of kaolin when sintered at 1200 °C for 1 h.

above 50 MPa, and the flexural strengths of clay materials such as mullite [30], kaolin [31], and diatomite [32] are usually below 30 MPa. In this study, although, the sintered diatomite composite support layer with no kaolin addition had the lowest flexural strength, the flexural strength of diatomite composite support layers sintered at 1200 °C for 1 h with an addition of 15 wt% kaolin eventually reached 60 MPa. Considering the high permeation properties, the low sintering temperature, and the low cost of the raw material, the flexural strength of a sintered diatomite composite support layer is feasible for microfiltration applications.

Taking into consideration the findings of the preliminary experiments and those in the literature [2,24], we found that sintered diatomite had similar microstructures up to 1200 °C. In addition, we could not expect a further enhancement of the permeability of the diatomite composite support layer by increasing the average pore size when the sintering temperature was higher than 1200 °C. Therefore, the sintering temperature of the diatomite composite support layer was determined to 1200 °C.

Fig. 1(c) depicts X-Ray Diffraction (XRD) patterns of diatomite composite support layers with varying amounts of kaolin and different sintering temperatures. When a diatomite composite support layer with a low amount of kaolin was sintered at 1200 °C for 1 h, cristobalite was noted to be the major phase. In addition, the mullite phase begins to appear as the amount of kaolin reaches 25 wt%. Consequently, we ruled out a phase transformation of the kaolinite to the mullite phase to explain the strengthening of the diatomite support layer with less kaolin added, such as 5 wt% and 15 wt%, as shown in Fig. 1(b). To identify the needle-like mullite or the plate-like kaolinite phase in the diatomite composite support layers, typical Scanning Electron Microscope (SEM) images of diatomite composite support layers sintered at 1200 °C for 1 h with additions of 0 wt% kaolin, 50 wt% kaolin, and 100 wt% kaolin are shown in Fig. 2(a), (b), and (c), respectively. With an addition of 50 wt% of kaolin, the microstructure of the diatomite composite support layer shows intermediate microscopic features between irregular diatomite and plate-like kaolinite. Furthermore, the predicted needle-like interlocking structure of the mullite phase or plate-like girders of kaolinite inside the pore channels are not found in the diatomite composite matrix. This may have resulted because the sintering temperature was not high enough to induce the phase transformation of kaolin to mullite to a degree that would be clearly visible.

Fig. 3(a) presents the densities of diatomite composite support layers sintered at from 900 °C to 1200 °C, showing that these values are proportional to the amount of kaolin added and the sintering temperature. Although the general trend of the density change of the diatomite composite support layers follows the rule of mixture well, the mercury intrusion pore volume decreases abruptly as the amount of kaolin added increases. The mercury intrusion pore volumes of diatomite

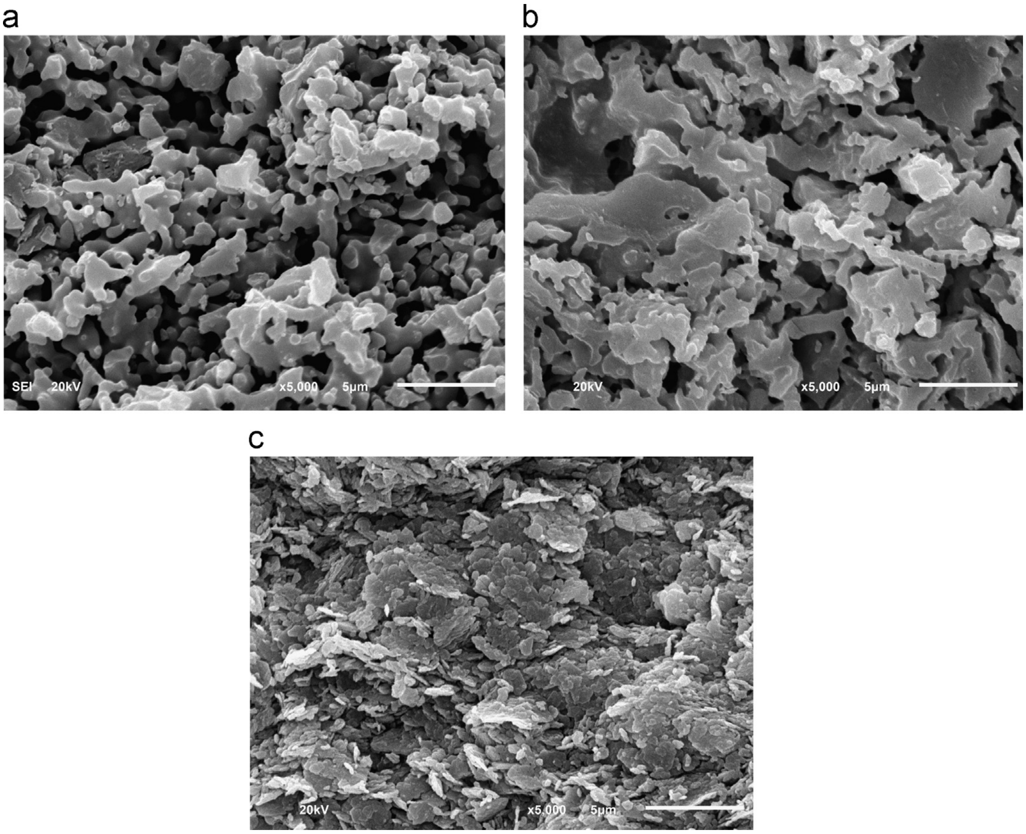

Fig. 2. Typical SEM images of the diatomite composite support layers sintered at 1200 °C for 1 h, with (a) 0 wt% kaolin; (b) 50 wt% kaolin; and (c) 100 wt% kaolin.

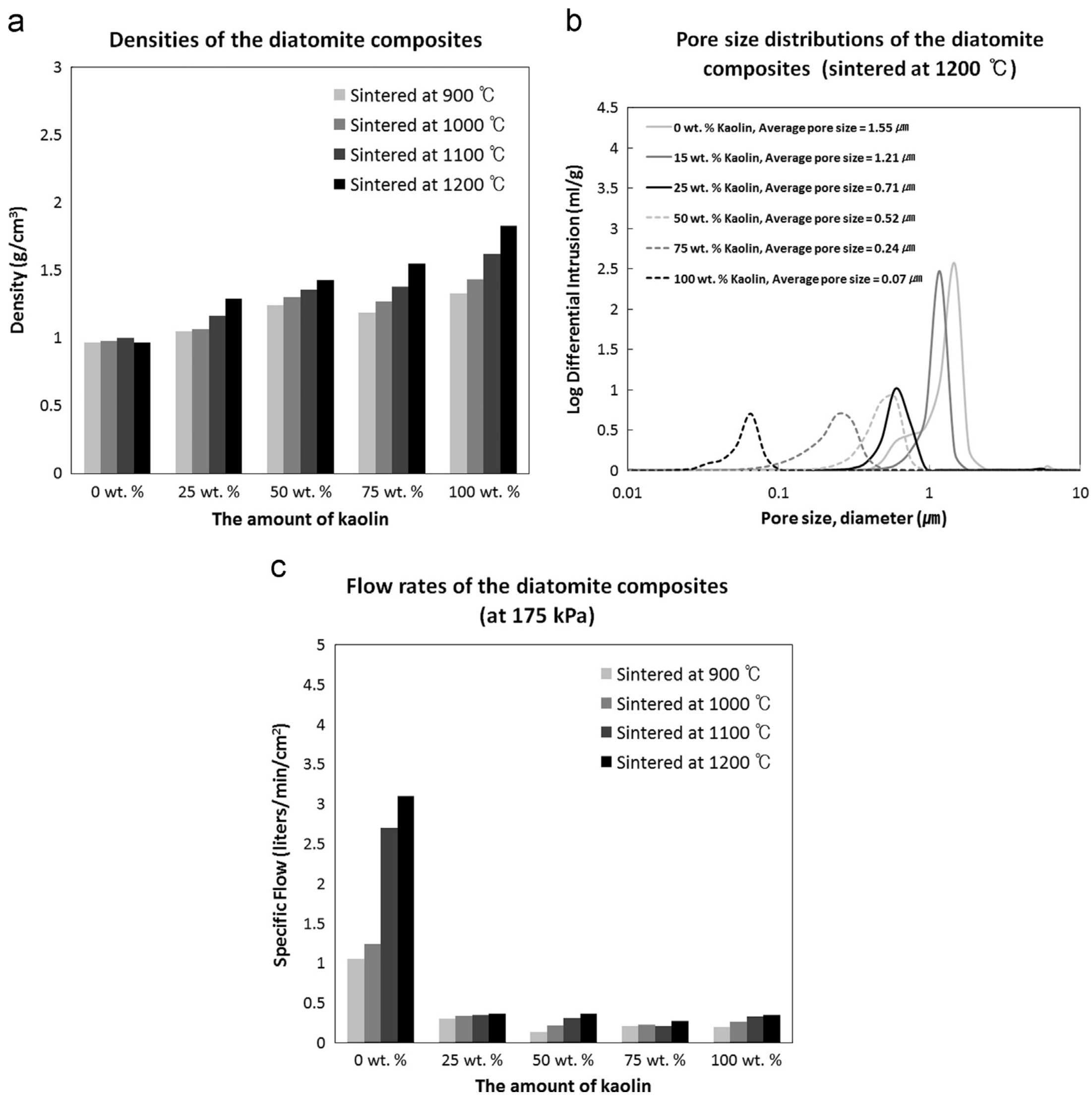

Fig. 3. (a) Densities of diatomite composite support layers sintered at 900 °C to 1200 °C for 1 h, (b) pore size distributions of the diatomite composite support layers sintered at 1200 °C for 1 h, and (c) the air permeation properties of the diatomite composite support layers sintered at 900 °C to 1200 °C for 1 h.

composite support layers sintered at 1200 °C for 1 h with additions of 0 wt% kaolin, 5 wt% kaolin and 15 wt% kaolin are 0.6588 ml/g, 0.5396 ml/g, and 0.3686 ml/g, respectively. In addition, the mercury intrusion pore volume of 100 wt% kaolin, eventually reaches 0.1447 ml/g. Also, the average pore sizes decrease as the amount of added kaolin increases, as shown in Fig. 3(b).

Fig. 3(c) presents the air permeation properties of diatomite composite support layers sintered at temperature ranging from 900 °C to 1200 °C, showing that the properties increased near linearly with the sintering temperature. Although there is no abrupt increase in the density, the air permeation properties of the diatomite support decrease exponentially. The strengthening effect induced by the added kaolin, with sintering at 1200 °C, may therefore be explained by the decrease in the pore volume and pore size, respectively, and the clogging of the pore channels (a decrease in the permeation property) in the diatomite composite matrix. Hence, the abovementioned pore characteristics strongly affect the overall properties of the diatomite composite support layers.

The one aspect that remains to be clarified is the largest pore size of the diatomite composite support layer. If a sintered porous ceramic is used as a support layer of a surface membrane, the largest pore size of the sintered layer is inconsequential. The important factors that must be considered for sintered porous ceramic support layers are the mass transfer resistance, mechanical strength, and chemical resistance. However, in this study which assesses the feasibility of a diatomite composite support layer without a separation layer as a porous ceramic membrane, this parameter is an important matter. Furthermore, one of the main specifications of a porous ceramic membrane is that the largest size of the solute that can pass through it is determined by the largest pore size.

Additional experiments were performed with the amounts of kaolin controlled from 0 wt% to 15 wt% to evaluate the permeation properties of diatomite composite support layers.

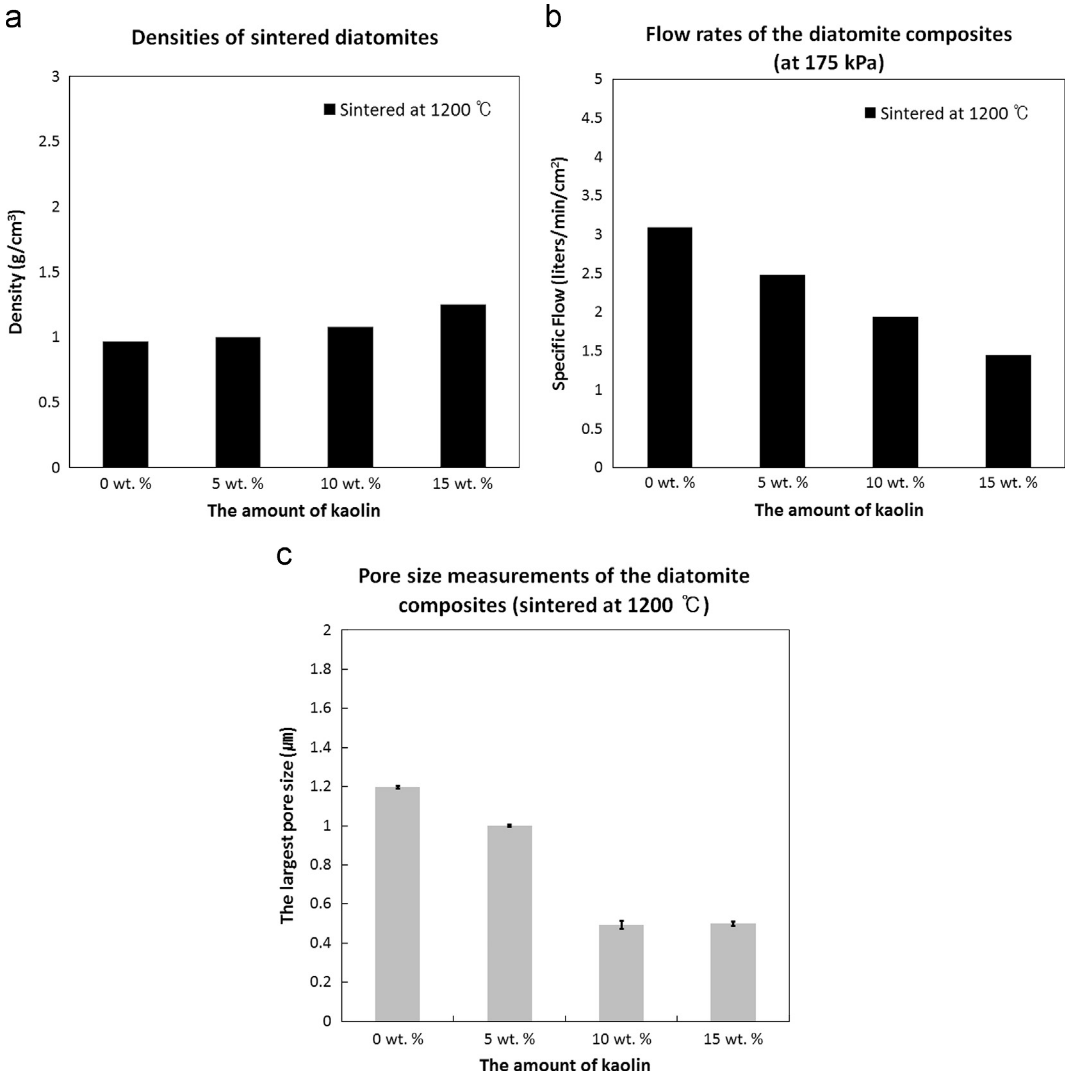

Fig. 4. (a) Densities, (b) the air permeation properties, and (c) the largest pore size of the diatomite composite support layers while varying with the amount of kaolin up to 15 wt% and sintering for 1 h at 1200 °C.

Fig. 4(a) presents the densities of diatomite composite support layers sintered at 1200 °C, showing that they are proportional to the amount of kaolin added. Moreover, the air permeation properties of the diatomite composite support layers sintered at 1200 °C, which are inversely proportional to the amount of kaolin addition, are shown in Fig. 4(b). Fig. 4(c) shows the variation of the largest pore size of sintered diatomite composite support layers with a sintering temperature of 1200 °C, as measured by the bubble point method. The bubble point method is the most widely used approach for evaluating the pore size, and it is capable of determining the largest pore size of a membrane as well. It is based on the feature, for a given fluid and pore size with a constant wetting, that the pressure required to force an air bubble through the pore is inversely proportional to the size of the pore. The largest pore sizes of sintered diatomite composite support layers could be controlled from around 0.4–1.2 μm with negligible standard error by changing the amount of kaolin that was added. With an addition of more than approximately 20 wt% kaolin, the measurement of the largest pore size will be imprecise in practice because the presence of open pore channels with certain degrees of permeation is the main assumption of the bubble point method. Fig. 4(b) and (c) show that the largest pore size of the sintered diatomite composites coincide with the permeability of the sintered diatomite composites. As shown in Fig. 3(b) and (c) earlier, the permeability of the sintered diatomite composites decreases abruptly as the amount of added kaolin increases, whereas the average pore size of the sintered diatomite composites decrease linearly. This occurs because the average pore size as measured by mercury porosimetry contains all open pores regardless of the pore type, including blind pores, cross-linked pores, and through pores [33], whereas, in principle, the largest pore size measured by capillary flow porosimetry ensures that the pores are inter-connected and acts as pore channels.

We measured the air permeation properties of sintered diatomite support layers. Therefore, to compare our results with the liquid permeation properties of typical ceramic microfiltration membranes reported in the literature, the pure water flux of the diatomite support layer sintered at 1200 °C was measured by capillary flow porosimetry. The pure water flux of the diatomite support layer sintered at 1200 °C was $6.3 \times 10^4\ \mathrm{L\ m^{-2}\ h^{-1}\ bar^{-1}}$. In the literature, the pure water flux of typical ceramic microfiltration membranes based on spherical fly ash [34], γ-alumina [11], and zirconia [35] are $1.6 \times 10^4\ \mathrm{L\ m^{-2}\ h^{-1}\ bar^{-1}}$, $\sim 1.0 \times 10^3\ \mathrm{L\ m^{-2}\ h^{-1}\ bar^{-1}}$, and $1.6 \times 10^3\ \mathrm{L\ m^{-2}\ h^{-1}\ bar^{-1}}$, respectively. Thus, the permeability of the diatomite membrane exhibited in this study may be good enough for cross-flow microfiltration. In addition, while we focused on the air permeation properties of sintered diatomite supports, the liquid permeation properties of these supports represent an important issue for future research.

4. Conclusion

In summary, the mechanical strengths of sintered diatomite composite support layers were found to be proportional to the increase in both the amount of kaolin and the sintering temperature up to 1200 °C. In addition, the air permeation properties of sintered diatomite composite support layers were proportional to the decrease in the average pore size and the pore volume induced by the addition of kaolin. The largest pore sizes of sintered diatomite composite support layers varied in a range of 0.4–1.2 μm with negligible standard error with an addition of kaolin up to 15 wt%. It was difficult to identify the presence of a phase transformation to the mullite phase from the kaolinite phase with a low amount of kaolin added at a sintering temperature of 1200 °C.

It is noteworthy that the mechanical strengths of the sintered diatomite composite support layers could be enhanced, while retaining acceptable permeation properties, by adding kaolin, which provides an effective means of tailoring the pore characteristics of the diatomite composite matrix. Furthermore, the largest pore sizes of the sintered diatomite composite support layers could be controlled in the sub-micron size range. These findings show the feasibility of using a sintered diatomite composite layer without a separation layer as a porous ceramic membrane for microfiltration.

Acknowledgments

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