



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.

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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 $\gamma\text{-Al}_2\text{O}_3$ [11,14], $\alpha\text{-Al}_2\text{O}_3$ [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 ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), 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 ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) 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

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