



The effects of diatomite addition on the pore characteristics of a pyrophyllite support layer

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Abstract

Recently, porous ceramic membranes have become a subject of special interest due to their outstanding thermal and chemical stability. To alleviate the manufacturing cost issues of these porous ceramic membranes, recent research has been focused on the utilization of low cost natural materials. Therefore, in this paper, we report the results of our efforts to determine whether we could utilize diatomite as a pore former and whether we could prepare a pyrophyllite–diatomite composite support layer that could effectively control the largest pore size and permeability. The pore characteristics of the specimens were studied by scanning electron micrography, mercury porosimetry, and capillary flow porosimetry. © 2015 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

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1. Introduction

Porous ceramics have lately attracted considerable interest [1] as researchers have endeavored to exploit their unique properties, such as low density [2], low thermal conductivity [3,4], and a low dielectric constant [5]. In particular, ceramic membranes [6–8] are among the most feasible applications of porous ceramics. Therefore, the precise control of the average pore size, the largest pore size, and the permeability of the porous ceramic membranes while minimizing the processing cost is important.

Because there are limitations on the reduction of the processing cost simply by altering the process conditions while utilizing commonly used, expensive high-purity ceramic materials, such as alumina and silicon carbide, in this study, we utilized porous ceramic membranes prepared from pyrophyllite as a matrix and diatomite as a pore former, as both of these materials are low-cost and natural.

Pyrophyllite is basically a natural, environmentally friendly clay material that is abundant in the Jeollanam-do Province of

Korea. Pyrophyllite is a hydrated aluminosilicate with a composition of $\text{Al}_2\text{O}_3 \cdot 4 \text{SiO}_2 \cdot \text{H}_2\text{O}$. The crystals of the pyrophyllite possess a plate-like form and are close to talc in structure. As the Al–OH bonds in the pyrophyllite might also possess the capacity for fluoride adsorption [9], pyrophyllite was recently considered to be one of the promising new candidate materials for utilization in porous ceramic membranes. However, there have only been a few studies on the pyrophyllite support layer [10,11].

Diatomite is a sedimentary rock resulting from the siliceous fossilized skeletons of diatoms, which are composed of rigid cell walls called frustules [12–15]. Previously, we reported possible approaches for the fabrication of a diatomite-based support layer that allowed for control over the pore characteristics, such as the largest pore size and the high mechanical strength, while an acceptable level of permeability was retained. These approaches that enabled the enhancement of the permeability of the diatomite support layer beyond what was previously achieved were as follows: by controlling the sintering temperature [16–18], the reduction of the largest pore size of the diatomite support layer was achieved through the formation of a diatomite–kaolin composite support layer [19], through the formation of a diatomite–kaolin composite coating

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layer [20], and the deposition of an alumina coating layer [21,22].

In this study, to further utilize the unique shapes of diatomite beyond its use as a porous matrix, we investigated the feasibility of diatomite as a pore former. It was expected that diatomite might act as a pore former due to the inter-particle voids induced by the irregular shapes of the diatomite particles and the inherent pores inside of the diatomite particles. The main advantages that were expected by substituting the conventional sacrificial polymer template with diatomite were as follows. The first advantage is that the micro-cracks, which are an escape path for the gas phase that is generated during the pyrolysis of the sacrificial polymer beads, within microstructure can be avoided. Another advantage is that the sintering time, which is required for complete pyrolysis, can be reduced [1]. The final advantage is that the cost can be mitigated by the elimination of the expensive sacrificial polymer template, which inhibited the utilization of low-cost pyrophyllite.

The present study investigated several important aspects. First, it was determined whether the pore characteristics of a pyrophyllite support layer with different sintering temperatures ranging from 1200 °C to 1400 °C could be controlled. Secondly, we investigated whether the pore characteristics of the pyrophyllite–diatomite composite support layers could be controlled by tailoring the pyrophyllite matrix through the addition of diatomite as a low cost pore former. Third, to support the investigation of the feasibility of diatomite as a pore former, kaolin–diatomite composite support layers were also prepared and characterized.

2. Material and methods

Pyrophyllite (Korea Powder Co. Ltd., Korea) was used for the preparation of the pyrophyllite support layers. The average particle sizes of the as-received pyrophyllite, the as-received diatomite (Celite 499, Celite Korea Co. Ltd., Korea), and the as-received kaolin (Kaolin, Sigma-Aldrich, U.S.A.) were 6.95 μm, 7.43 μm, and 1.53 μm, respectively. And the typical technical properties of the as-received diatomite are as follows: the color is white, the appearance is fine powder, and the origin is plankton marine diatomite. Distilled water was used as a solvent, and the slurry was ball-milled for 24 h with an alumina ball-to-powder volume ratio of 2:1. After ball-milling, the slurry was dried at room temperature for 24 h.

To incorporate diatomite into the pyrophyllite matrix, pyrophyllite and diatomite were mixed for 3 h by ball-milling with a ball-to-powder volume ratio of 0.5:1. With the use of 2 wt% of polyethylene glycol as a binder, the pyrophyllite, the diatomite, or the pyrophyllite–diatomite mixtures (the pyrophyllite with a variation in the amount of diatomite from 0 wt% to 100 wt%) were dry-pressed at 18.7 MPa and sintered between 1200 °C and 1400 °C for 1 h. For comparison, kaolin–diatomite support layers were prepared by following the aforementioned procedures. The pore characteristics of the diatomite were investigated through scanning electron micrographs (JSM-5800, JEOL, Japan), and mercury porosimetry (Autopore IV 9510, Micromeritics, U.S.A.).

The permeability was measured by capillary flow porosimetry (CFP-1200-AEL, Porous Materials Inc., U.S.A.). A sintered diatomite composite specimen (diameter of 4 cm and a thickness of 0.4 cm) was inserted between the O-rings in the bottom of the chamber, and the bottom of the chamber was inserted into the capillary flow porosimeter. The flux was then measured by sensors automatically while the diameter of the motorized valve and the pressure of the regulator were increased incrementally.

The largest pore size of the diatomite membrane was measured by the bubble point method. The bubble point method is the method that is the most widely used for pore size determination, and it is capable of determining the largest pore size of a membrane. The method is based on the fact that, for a given fluid and pore size with constant wetting, the pressure required to force an air bubble through the pore is inversely proportional to the size of the pore.

3. Results and discussion

X-ray diffraction (XRD) patterns of the pyrophyllite support layers sintered at 1200 °C, 1300 °C, and 1400 °C for 1 h are shown in Fig. 1(a). When a pyrophyllite support layer was sintered at 1200 °C for 1 h, the SiO₂ (quartz) phase was observed to be the major phase. When a pyrophyllite support layer was sintered at 1300 °C for 1 h, the Al₆Si₂O₁₃ (mullite) phase, as well as the SiO₂ (cristobalite) phase were observed. As the sintering temperature of the pyrophyllite support layer reached 1400 °C, the peak intensity of the SiO₂ (quartz) phase significantly decreased. The observed phase transformation phenomenon of the pyrophyllite with the increase in the sintering temperature was well-matched with what had been reported in the literature [23,24].

To determine the microstructure of the pyrophyllite support layers, typical Scanning Electron Microscope (SEM) images of the pyrophyllite support layers sintered at 1200 °C, 1300 °C, and 1400 °C for 1 h are shown in Fig. 1(b)–(d), respectively. In Fig. 1(b), the plate-like pyrophyllite phase in the microstructure was easily identified. As the sintering temperature of the pyrophyllite support layer increased from 1200 °C to 1400 °C, the gradual phase transformation to the mullite phase was found in Fig. 1(b)–(d), as was expected from the observations made from Fig. 1(a).

As presented in Fig. 2(a), the average pore size of the pyrophyllite support layers increased as the sintering temperature increased up to 1400 °C. These trends were in good agreement with the experimental results that focused on different porous ceramics, such as diatomite [15], silicon carbide [25,26], alumina [27], corundum–mullite [28] and zirconia [29], with different temperature ranges. Although the increase of the average pore size is generally explained by pore coarsening [27] or densification [26], the relationship between the average pore size and the sintering temperature has not yet been established.

Fig. 2(b) presents the permeabilities and the largest pore sizes of the pyrophyllite support layers prepared at different sintering temperatures ranging from 1200 °C to 1400 °C. The

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