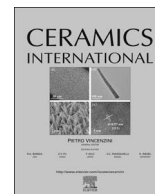




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# The preparation and characterization of alumina-coated pyrophyllite-diatomite composite support layers

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## ABSTRACT

Porous ceramic membranes are a current research focus because of their outstanding thermal and chemical stabilities. Recent research has utilized inexpensive natural materials such as pyrophyllite and diatomite to reduce the expense of these porous ceramic membranes. However, insufficient data exist for microfiltration applications using pyrophyllite-based membranes. We compared the measured membrane properties of alumina-coated alumina support layers and alumina-coated pyrophyllite-diatomite composite support layers. These experiments were used to determine whether we could effectively reduce the average pore size with acceptable water permeability by controlling the thickness of the alumina coating layer. The membrane properties of the alumina-coated alumina support layers and alumina-coated pyrophyllite-diatomite composite support layers were examined using scanning electron microscopy, mercury porosimetry, and a dead-end microfiltration system.

## 1. Introduction

Porous ceramics have received constant attention and they were recently reviewed [1], because of their unique properties, such as low densities [2], low thermal conductivities [3,4], and low dielectric constants [5]. Porous ceramic membranes [6,7] are the most feasible application for porous ceramics. Therefore, it is important to control the average pore size, mechanical strength, and water permeability of porous ceramic membranes and to minimize the material processing costs. Altering the process conditions and using standard high-purity ceramic materials, such as  $\alpha$ -alumina, zirconia, and silicon carbide, results in an expensive process.

We used a pyrophyllite-diatomite composite support layer as a feasible substitute for typical ceramic support layers because pyrophyllite and diatomite are inexpensive, natural, and abundant.

Pyrophyllite is 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 pyrophyllite possess a plate-like form and are close to talc in structure. The Al–OH bonds in the pyrophyllite might possess the capacity for fluoride adsorption [8], and pyrophyllite was recently considered as a promising new candidate material for utilization in porous ceramic membranes. However, there have only been a few studies on the pyrophyllite support layer [9,10].

Diatomite is a sedimentary rock resulting from the siliceous

fossilized skeletons of diatoms, which are composed of rigid cell walls called frustules [11–14]. We used diatomite as a pore former for the pyrophyllite support layer because the permeability of a pyrophyllite support layer is not acceptable as a membrane for water treatment applications. Previously, we reported on the fine-tuning of the membrane properties of a pyrophyllite-based support layer [15,16].

There are direct comparisons between a typical alumina-based membrane and a novel pyrophyllite-based membrane. We previously discussed the presence of a coating layer over the pyrophyllite-based support layers by analyzing the pore size distributions and the pore structure [17]. The thickness of the coating layer is an important factor because the thickness of the coating layer affects the permeability and average pore size of the porous ceramic membrane. Only a few studies have examined the thickness of the alumina coating layer deposited on the pyrophyllite-diatomite composite support layers.

We determined how the thickness of the alumina coating layer deposited over the pyrophyllite-diatomite composite support layer was controlled by changing the dip-coating conditions. We also investigated whether the water permeability of the pyrophyllite-diatomite composite support layers was acceptable after depositing an alumina coating layer compared to the alumina support layer.

## 2. Materials and methods

$\alpha$ -Alumina (AM-210, Sumitomo Chemical Co. Ltd., Japan), pyr-

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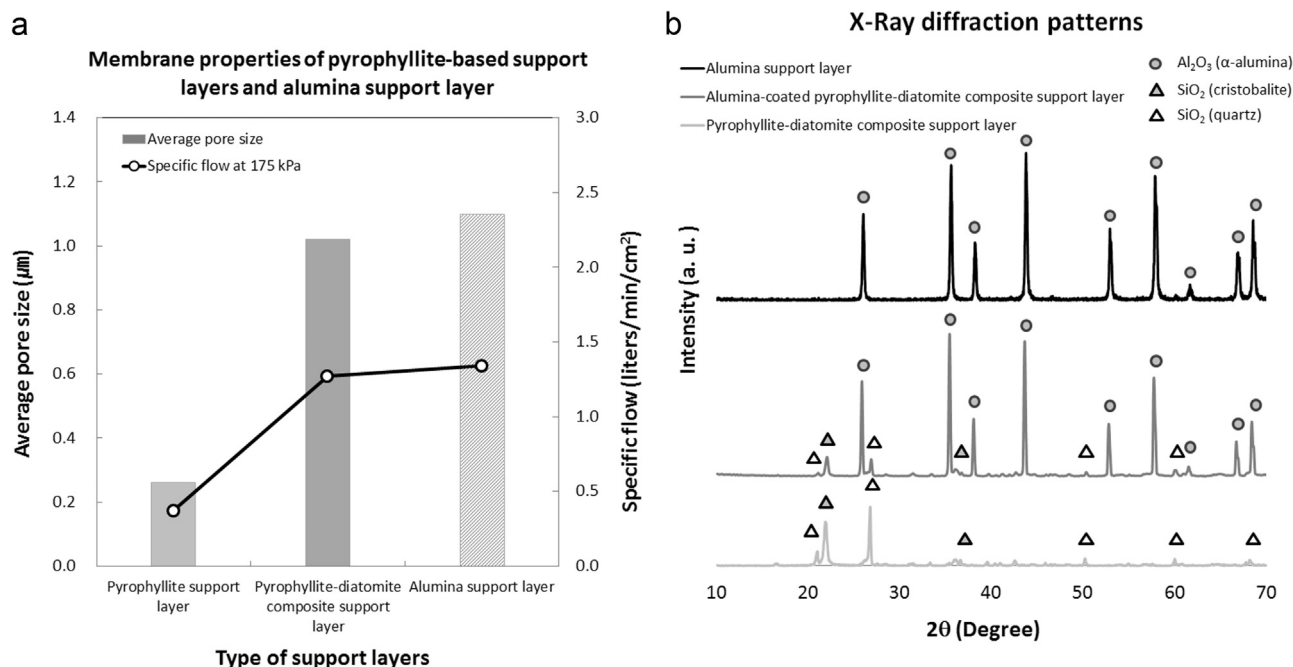
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**Fig. 1.** (a) Average pore sizes (left vertical-axis, bar graph) and specific flow (right vertical-axis, line graph) for a pyrophyllite support layer sintered at 1200 °C for 1 h, a pyrophyllite-diatomite composite support layer sintered at 1200 °C for 1 h, and an alumina support layer sintered at 1600 °C for 1 h; (b) X-ray diffraction patterns of an alumina support layer, an alumina-coated pyrophyllite-diatomite composite support layer, and pyrophyllite-diatomite composite support layer.

ophyllite (Korea Powder Co. Ltd., Korea), and diatomite (Celite 499, Celite Korea Co. Ltd., Korea) were used for the preparation of the porous ceramic support layers. The average particle sizes of the starting powders were determined using a particle size analyzer (LSTM 13 320 MW, Beckman Coulter, USA). The average particle sizes of the as-received alumina, pyrophyllite, and diatomite were 4.80  $\mu\text{m}$ , 6.95  $\mu\text{m}$ , and 7.43  $\mu\text{m}$ , respectively. 73.5 wt% Pyrophyllite, 25 wt% diatomite, and 1.5 wt% sodium borate (Shinyo Pure Chemical Co. Ltd., Korea) were used for the preparation of the pyrophyllite-diatomite composite support layers.

Methyl cellulose (17 wt%, Sigma-Aldrich, USA) (binder) and 20 wt % distilled water (solvent) were added to extrude the alumina support layers. Methyl cellulose (17 wt%, binder) and 50 wt% distilled water (solvent) were added to extrude the pyrophyllite-diatomite composite support layers.

The mixed slurry was aged for 48 h at room temperature and extruded using a double screw extruder (KTE-50S, Kosentech, Korea). The extruded alumina or pyrophyllite-diatomite composite support layers had flat tube-type dimensions (a width of 50 mm, height of 4 mm, and length of 200 mm) and 16 inner holes (a width of 2 mm and height of 2 mm). After extrusion, the specimens were dried for 24 h at room temperature. The dried specimens were heat treated at 400 °C for 1 h to burn-off the binder and sintered at 1200 °C for 1 h (for pyrophyllite-diatomite composite support layers) or 1600 °C for 1 h (for alumina support layers).

A conventional dip-coater (table-top dip-coater, E-flex, Korea) was used to deposit an alumina coating onto the alumina support layer and the pyrophyllite-diatomite composite support layer. For the coating process, 8 wt% alumina (AKP-30, Sumitomo Chemical Co. Ltd., Japan), 33 wt% 2-propanol (Sigma-Aldrich, USA), 57 wt% distilled water, and 2 wt% PVA binder (Polyvinyl alcohol 500, Junsei Chemical, Japan) were mixed and ball-milled with alumina media balls for 0–2 h, dip-coated for 10 s, retracted at a withdrawal speed of 1 mm/s, dried at room temperature for 24 h, and heat-treated at 1200 °C for 1 h (for pyrophyllite-diatomite composite support layer) or 1300 °C for 1 h (for alumina support layers).

The pore characteristics of the specimens were investigated using scanning electron micrographs (JSM-5800, JEOL, Japan) and mercury

porosimetry (Autopore IV 9510, Micromeritics, USA).

The flexural strength of the alumina support layer, and the pyrophyllite-diatomite support layer were measured using a four-point bending test on an Instron 4206 testing system. For the flexural strength test, 3×4×25 mm specimens were machined and tested on 20 mm outer span four-point fixtures.

The air permeability was measured using capillary flow porosimetry (CFP-1200-AEL, Porous Materials Inc., USA). A specimen (4 cm in diameter and 0.4 cm thick) was fitted between the O-rings and inserted in the capillary flow porosimeter located in the bottom of the chamber. Finally, the flux was automatically measured via sensors while the diameter of the motorized valve and the pressure of the regulator were incrementally changed.

The water permeability of the alumina support layer and the pyrophyllite-diatomite composite support layer were measured using a dead-end microfiltration system (MTS2000, Sam Bo Scientific, Korea) equipped with particle counters (PAMAS-4132, PAMAS, Germany). The operating trans-membrane pressure was maintained at 2.5 bar.

### 3. Results and discussions

Fig. 1(a) depicts the air permeability and the average pore size of the pyrophyllite support layer, the pyrophyllite-diatomite composite support layer, and the alumina support layer. The air permeability and the average pore size of the pyrophyllite-diatomite composite support layer sintered at 1200 °C for 1 h shifted toward those of the alumina support layer when diatomite was introduced to the pyrophyllite support layer. The flexural strength of the alumina support layer (77.9 MPa) sintered at 1600 °C for 1 h was four times higher than the pyrophyllite-diatomite composite support layer (16.3 MPa) sintered at 1200 °C for 1 h. However, it was worthwhile to investigate the feasibility of the pyrophyllite-diatomite composite support layers when operated under low load-bearing conditions because of the low sintering temperature (1200 °C), the similar average pore size (1.02  $\mu\text{m}$ ), and the low cost of the pyrophyllite-diatomite composite support layer. These findings were partially reported [16]. Although we demonstrated that the addition of diatomite provided an effective

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