



The effect of an alumina coating on the pore characteristics of a diatomite–kaolin composite support layer

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Abstract

Porous ceramic membranes have recently attracted great interest due to their outstanding thermal and chemical stabilities. In this paper, we report the results of our efforts to determine whether we could prepare an alumina coating to be deposited over a diatomite–kaolin composite support layer that could control the largest pore size and permeability. In addition, we determined under what conditions such an alumina coating over a diatomite–kaolin composite support layer could be prepared with minimum micro-crack generation during heat-treatment. The pore characteristics of an alumina coating on a diatomite–kaolin composite support layer were studied by scanning electron microscopy and capillary flow porosimetry.

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1. Introduction

Porous ceramics have recently attracted great interest [1] as researchers seek to exploit their unique properties [2–7]. Notably, membranes [8–12] are among the most feasible applications of porous ceramics.

It is generally believed that the most important features of a porous ceramic membrane are its permeation and separation properties. Therefore, precisely controlling pore characteristics while retaining an acceptable level of permeability and strength is very important. One challenge associated with the application of porous ceramic membranes is determining how to control, tailor, and characterize pores, and various pore shapes have been well discussed in detail [13,14].

Much research has been dedicated to the study of materials commonly used for ceramic membranes [10,11,15–20], but there have been only a few studies on ceramic membranes prepared from natural clays such as diatomite or kaolin [21–23].

Previously, we reported on three types of possible approaches for fabricating a sintered diatomite membrane that

allow for control over membrane characteristics (e.g., largest pore size and mechanical strength) while retaining an acceptable level of permeability.

The aim of the first approach is to enhance the permeability of the diatomite support layer beyond that achieved by controlling the sintering temperature. To this end, spherical macro-pores were induced in the diatomite support layer by incorporating a sacrificial polymer template as a pore former [24]. In the second approach, to reduce the largest pore size of the diatomite support layer, kaolin was added to form a diatomite–kaolin composite support layer [25]. In the third approach, the focus was on determining how to decrease the largest pore size of the diatomite membrane even further while maintaining an acceptable level of permeability. Therefore, kaolin was selected to form a diatomite–kaolin composite coating [26].

However, there are practical limitations in tailoring the pore characteristics of a sintered diatomite membrane by using only natural clay composites such as a diatomite–kaolin composite coating or a diatomite–kaolin composite support layer, due to the coarse particle size, inherent pores inside particles, and the irregular shape of diatomite particles. Moreover, to the best of our knowledge, it has not yet been investigated whether an

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alumina coating can be deposited on a diatomite-based support layer. If a low-cost sintered diatomite membrane can be coated with minimum use of an expensive high-purity alumina, then it is possible to apply a vast database of alumina membranes to diatomite membranes as further coatings in catalytic or filtration applications.

Therefore, in a fourth approach (in this study), the main focus was to elucidate the feasibility of coating an alumina layer on a sintered diatomite membrane. To these ends, several important aspects of our ultimate goals were investigated. First, we determined whether we could control the pore characteristics of a sintered diatomite membrane, with minimal crack formation or delamination, by adopting an alumina coating prepared under different processing conditions. We also investigated whether we could overcome the difference in the degree of shrinkage between an alumina-based coating and a diatomite-based support layer.

2. Material and methods

Diatomite (Celite 499, Celite Korea Co. Ltd., Korea) and kaolin (Kaolin, Sigma-Aldrich, U.S.A.) were used to prepare a diatomite–kaolin composite support layer. The average particle sizes of the starting powders were determined by a particle size analyzer (LSTM 13 320 MW, Beckman Coulter, USA). The average particle sizes of the as-received diatomite, as-received kaolin, and as-received alumina (AKP-30, α -alumina, Sumitomo Chemical, Japan) were 12.79 μm , 1.53 μm , and 0.45 μm , respectively. To enhance the sintering of the diatomite particles, the average particle size of the diatomite was reduced to 7.43 μm by ball-milling. 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. To incorporate kaolin into the diatomite matrix, diatomite particles were mixed with 10 wt% kaolin for 3 h by ball-milling with an alumina ball-to-powder volume ratio of 2:1. With polyethylene glycol as a binder, the diatomite–kaolin composite specimens were dry-pressed at 18.7 MPa, and sintered at 1200 °C for 1 h.

To investigate the effect of the interface between an alumina coating and an diatomite–kaolin composite support layer, diatomite particles were mixed with 5 wt% kaolin and 50 wt% alumina for 3 h by ball-milling with a ball-to-powder volume ratio of 2:1. With polyethylene glycol as a binder, the diatomite–kaolin–alumina composite specimens were dry-pressed at 18.7 MPa and sintered at 1000–1400 °C for 1 h.

A dip-coating process was used to deposit an alumina coating onto the diatomite–kaolin composite support layer. For the coating process, alumina, distilled water, an organic binder (HS BD-25, San Nopco Korea, Korea), and an inorganic binder (AS-40, Sigma-Aldrich, USA) were mixed, dip-coated on a pre-sintered diatomite–kaolin composite support layer, dried at room temperature for 24 h, and then heat-treated at 500–1200 °C for 1 h.

The pore characteristics of the diatomite membranes were examined by scanning electron microscopy (JSM-5800, JEOL, Japan). The air flux was measured by capillary flow porosimetry (CFP-1200-AEL, Porous Materials Inc., U.S.A.). All specimens

(diameter 4 cm, and thickness 0.4 cm) were fitted between the O-rings in the bottom of the chamber and the bottom of the chamber inserted for capillary flow porosimetry. The flux was then measured automatically by sensors while incrementing the diameter of the motorized valve and the pressure of the regulator.

The largest pore size of the diatomite membrane was measured by the bubble point method. The bubble point method is the most widely used for pore size determination and 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

Typical scanning electron microscope (SEM) images of diatomite–kaolin composite support layers sintered at 1000, 1100, and 1200 °C for 1 h with additions of 10 wt% kaolin are shown in Fig. 1(a), (b), and (c), respectively. We prepared diatomite–kaolin composite support layers with the addition of 10 wt% kaolin due to its appropriate permeability and mechanical strength. As shown in Fig. 1(a)–(c), the inter-connected pore structure of the diatomite–kaolin composite support layer was gradually shaped and defined, as the sintering temperature increased from 1000 to 1200 °C.

In a previous report [25], the sintering temperature of diatomite–kaolin composite support layers was confined to 1200 °C to yield an appropriate permeability and mechanical strength. Moreover, the effect of kaolin addition on the pore characteristics of the diatomite–kaolin composite support layers was mainly discussed.

Based on the results of the aforementioned report, in this study, the amount of kaolin addition to the diatomite–kaolin composite support layers was confined to 10 wt%, and then, the effect of sintering temperature on the pore characteristics of the diatomite–kaolin composite support layers was investigated. Because the diatomite–kaolin composite support layers would be exposed to a heat-treatment temperature again, after the deposition of an alumina coating, the sintering temperature of the diatomite–kaolin composite support layers was not confined to 1200 °C.

Fig. 1(d) shows that the diatomite–kaolin composite support layer with 10 wt% kaolin, sintered at 1200 °C for 1 h, exhibits both higher permeability and higher flexural strength than the layers sintered at 900, 1000, and 1100 °C for 1 h, as expected from the pore structures shown in Fig. 1(a)–(c). In addition, the average pore sizes of the diatomite–kaolin composite support layers sintered at 900, 1000, 1100, and 1200 °C for 1 h with the addition of 10 wt% kaolin were 0.48 μm , 0.64 μm , 0.75 μm , and 0.99 μm , respectively. The permeability of the diatomite–kaolin composite support layers can be explained by these values, which are proportional to the sintering temperature.

The flexural strength of a typical ceramic membrane based on alumina [8] or silicon carbide [27] is often above 50 MPa, and the flexural strengths of natural clays such as mullite [28],

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