Development of porous ceramic membrane from natural zeolite – clay for microfiltration

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Development of porous ceramic membrane from natural zeolite – clay for microfiltration

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Abstract. Indonesia has a large amount of natural zeolite and clay. These materials are potential for ceramic membrane production. The aims of this study were to describe the characteristic of ceramic membrane development from natural zeolite and clay. With an addition of TiO₂, ceramic membrane was produced by dry process by pressing at 5 tons for 30 minutes. From the results of the investigation, it can be concluded that the natural zeolite and clay from Banyumas may be utilized for obtaining ceramic membranes for microfiltration at low sintering temperature of 900°C. Optimum addition of TiO₂ was 5%wt. At this condition, the densities of ceramic membranes were 1.44 gr/cm³, 1.8 gr/cm³, and 1.5 gr/cm³. The porosities of ceramic membranes were 53.29%, 31.099%, and 35.42%. The water absorption values of ceramic membranes were 36.83%, 17.06%, and 23.61% for natural zeolite, clay, and natural zeolite – clay respectively.

1. Introduction
Membrane separation process widely used for many purposes. There are two types of membrane that are organic (polymeric membrane) and non-organic membrane (metal and ceramic membranes). Each membrane has advantages and weaknesses. The ceramic membranes have many advantages such as high thermal and chemical stability, pressure resistance, long lifetime, good resistance to fouling, and easy to clean [1]. The weakness of the ceramic membrane is fragile and high-cost production due to the materials used. Materials commonly used for ceramic membranes are Al₂O₃, TiO₂, ZrO₂, SiO₂ etc. or a combination of these materials [2].

Several researchers used the natural materials for ceramic membrane development. The aim of utilization of natural material is to decrease the production cost while good characteristics are also achieved. For instance, kaolin-clay ceramic membrane for microfiltration is used in [1], apatite material for microfiltration and ultrafiltration is used in [3], clay for microfiltration of oily waste water is used in [4] and fly ash for microfiltration is used in [5].

The characteristics and properties of the ceramic membranes are mainly determined by their composition, the pore-former content and the sintering temperature. Based on pore of membrane, membranes can be classified into 2 main groups, porous and non-porous membranes. Separation mechanism in porous membranes is by molecular sieving. Mechanism of transport in non-porous membranes involve permeating molecules dissolve into a membrane, diffuse, and desorb from the membrane [6].
The definition of porous membranes is more in agreement with the definitions adopted by the International Union of Pure and Applied Chemistry (IUPAC). The pore size classification given refers to pore diameter: (i) Macropores > 50 nm. (ii) Mesopores 2 nm < pore size < 50 nm. (iii) Micropores < 2 nm [7]. Transport occurs through porous membranes rather than the dense matrix and ideal gas separation membranes possess a high flux and a high selectivity. Indonesia has a large of natural zeolite and clay. These materials are potential for ceramic membrane production. The aims of this study were to describe the characteristic of ceramic membrane developed from natural zeolite and clay, with an addition of TiO$_2$.

2. Materials and methods

This section explains the materials and methods used in this research. The materials, methods, and characterizations are elaborated in the following subsections.

2.1. Materials

Natural zeolite and starch were purchased from local market at Purwokerto, Indonesia. Clay was obtained from Kebumen district, Indonesia. TiO$_2$ was purchased from Merck.

2.2. Methods

Natural zeolite and clay were crushed to the size of < 45 µm. The materials were mixed with TiO$_2$ at various composition. The composition of mixture is given in table 1. The mixture was then placed at O-ring casting with the diameter of 3.2 cm and the thickness of 1 cm and pressed at 5 tons for 30 minutes. The mixture was then calcinated at the temperature of 900°C for 6 hours.

| Sample | Natural zeolite (%w) | Clay (%w) | TiO$_2$ (%w) |
|--------|---------------------|-----------|---------------|
| 1      | 100                 | 0         | 0 – 5 – 10    |
| 2      | 0                   | 100       | 0 – 5 – 10    |
| 3      | 50                  | 50        | 0 – 5 – 10    |

2.3. Characterizations

The composition of raw materials was analyzed using X-Ray Diffraction (XRD). Morphology of ceramic membrane was analyzed using Scanning Electron Microscopy (SEM). Bulk density, porosity, and water absorption of membrane was analyzed using gravimetric methods.

3. Results and Discussion

The discussion of the result is explained in the following subsections.

3.1. Materials characterization

XRD was used to determine the characteristic of natural zeolite and clay. The Crystallinity of natural zeolite and clay were 84.6354% and 54.41% respectively. Figure 1 shows the XRD pattern of natural zeolite and clay. SiO$_2$ content in natural zeolite is 75.62% while in clay is 53.07%.

3.2. Ceramic membrane characterization

The characteristic of ceramic membrane is explained in the following subsections.

3.2.1. Bulk density. The characteristics of ceramic membrane are described into three parameters such as bulk density, porosity and water absorption. Figure 2 shows the effluence of addition of TiO$_2$ on bulk density of ceramic membrane. Ceramic membrane developed from clay has higher bulk density, while from natural zeolite has lower bulk density. Addition of TiO$_2$ will increase the bulk density of ceramic
membrane. The optimum addition of TiO2 is 5% w. At this condition, the densities of ceramic membranes are 1.44 gr/cm³, 1.8 gr/cm³, and 1.5 gr/cm³ for natural zeolite, clay, and natural zeolite–clay respectively. The density of ceramic membrane developed from natural zeolite was 1.86 g/cm³ at temperature calcination 900°C according to the report in [8].

![Figure 1. XRD pattern of materials: (a) natural zeolite, (b) clay.](image)

![Figure 2. The effluence of TiO₂ concentration on bulk density.](image)

3.2.2. Porosity. Figure 3 describes the porosity of the ceramic membrane. Porosity of Ceramic membrane developed from clay is higher than the other. Addition of TiO₂ will increase the porosity of ceramic membrane. The porosities of ceramic membranes are 53.29%, 31.099%, and 35.42% for natural zeolite, clay, and natural zeolite–clay respectively at addition 5% of TiO₂. The porosities of ceramic membranes are 53.29%, 31.099% and 35.42%. 
3.2.3. Water absorption. Figure 4 describes the porosity of the ceramic membrane. Porosity of Ceramic membrane developed from clay is higher than the other. Addition of TiO$_2$ will increase the porosity of ceramic membrane. The water absorption values of ceramic membranes are 36.83%, 17.06%, and 23.61% for natural zeolite, clay, and natural zeolite – clay respectively at addition 5% of TiO$_2$.

3.3. Morphology of membrane
Figure 5 shows the morphology of ceramic membrane developed. Ceramic membrane from natural zeolite shows more pores. The pore size of ceramic membrane from clay has good unity of pore size.
4. Conclusion
From the results of the investigation, it can be concluded that the natural zeolite and clay from Banyumas may be utilized for obtaining ceramic membranes for microfiltration at low sintering temperature of 900°C. Optimum addition of TiO$_2$ is 5%wt. At this condition, the densities of ceramic membranes were 1.44 gr/cm$^3$, 1.8 gr/cm$^3$, and 1.5 gr/cm$^3$, the porosities of ceramic membranes were 53.29%, 31.099%, and 35.42%, the water absorption values of ceramic membranes were 36.83%, 17.06%, and 23.61% for natural zeolite, clay, and natural zeolite – clay respectively.

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