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Synthesis of a self-supporting faujasite zeolite membrane using geopolymer gel for separation of alcohol/water mixture

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A self-supporting faujasite (FAU) zeolite membrane was synthesized by *in situ* hydrothermal method at 90 °C for 15 h from a Na_2O–$3SiO_2$–Al_2O_3 geopolymer gel that was prepared from the raw materials metakaolin and sodium silicate solutions (molar ratio of $SiO_2/Na_2O=1$). The results revealed that the BET surface area and the compressive strength of the FAU zeolite membrane were 63.31 m^2/g and 19.6 MPa, respectively. The pervaporation study of its separation performance in a C_2H_5OH/H_2O mixture demonstrated that the permeate flux and separation factor can reach 1.41 kg/(h · m^2) and 16.8, respectively, at a feed temperature of 50 °C and an alcohol composition of 70 wt%, making it an effective pervaporation membrane for the separation of alcohol/water mixtures.

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1. Introduction

Geopolymers, first identified by Joseph Davidovits in 1978, are a class of novel, rapidly developed aluminosilicate inorganic polymer materials that exhibit a three-dimensional network. Geopolymers are prepared using natural minerals or the solid waste from the polymerization of silicon–oxygen tetrahedral and aluminum–oxygen tetrahedral structures [1]. In fact, previous studies have indicated that the structures of geopolymers are zeolite-like [1–3], and our previous work demonstrated that the composition and structure of geopolymers are similar to those of zeolites. Moreover, the investigated geopolymers were able to convert to zeolite molecular sieves under appropriate hydrothermal conditions [4,5].

In recent decades, zeolite membranes have attracted considerable research attention due to their separation performance [6–8]. To be used as a separation filter, zeolite membranes are typically required to possess sufficient mechanical strength and no macroscopic defects, making two types of zeolite membranes viable candidates for filtration applications: (1) thick zeolite polycrystalline films (self-supporting) and (2) thin zeolite films supported by a porous material (*e.g.*, alumina, silica, and stainless steel) [9,10]. Kita et al. [11] through secondary hydrothermal method on porous alumina ceramic membrane synthesized high performance faujasite zeolite (NaX and NaY zeolite) membrane, for the ethanol content of 10% at 373 K, the permeation flux and separation factor can achieve 4.5 kg/(h · m^2) and 30,000, respectively. Jeong et al. [12] prepared NaY zeolite membrane on the same supports and this NaY zeolite membrane have a good selectivity and separation effect on benzene in benzene/alkane system. However, few self-supporting faujasite zeolite membranes have been developed, and their separation performances have not been intensively studied [13].

In this study, a thick film (self-supporting faujasite zeolite membrane with 10 mm thickness) was synthesized from Na_2O–$3SiO_2$–Al_2O_3 geopolymers by *in situ* hydrothermal growth method. Compared with traditional supported zeolite membranes, the self-supporting zeolite membrane developed in this study can eliminate defects and the mismatch between the zeolite layer and the support material as well as reduce the difficulty of the preparation procedure.

2. Experimental procedure

A sketch map of the self-supporting faujasite zeolite membranes synthesized by the *in situ* hydrothermal method is displayed in Fig. 1. In this study, an Al_2O_3–$3SiO_2$–Na_2O geopolymer was prepared using metakaolin and sodium silicate (molar ratio of $SiO_2/Na_2O=1$) as the raw materials. The composition of the geopolymer was selected according to the FAU zeolite (X-type) composition (Si/Al/Na atom ratio of 3:2:2), the geopolymer slurry was cast into molds, and finally the molds were sealed and cured at 60 °C for 24 h. Then the solid geopolymer gel molds and 100 ml deionized water were transferred into 150 ml Teflon-lined hydrothermal reservoirs and kept at 90 °C for 6–15 h. The FAU zeolite membranes were tested for their pervaporation performance, as depicted in Fig. 3a. During pervaporation, the disc-shaped

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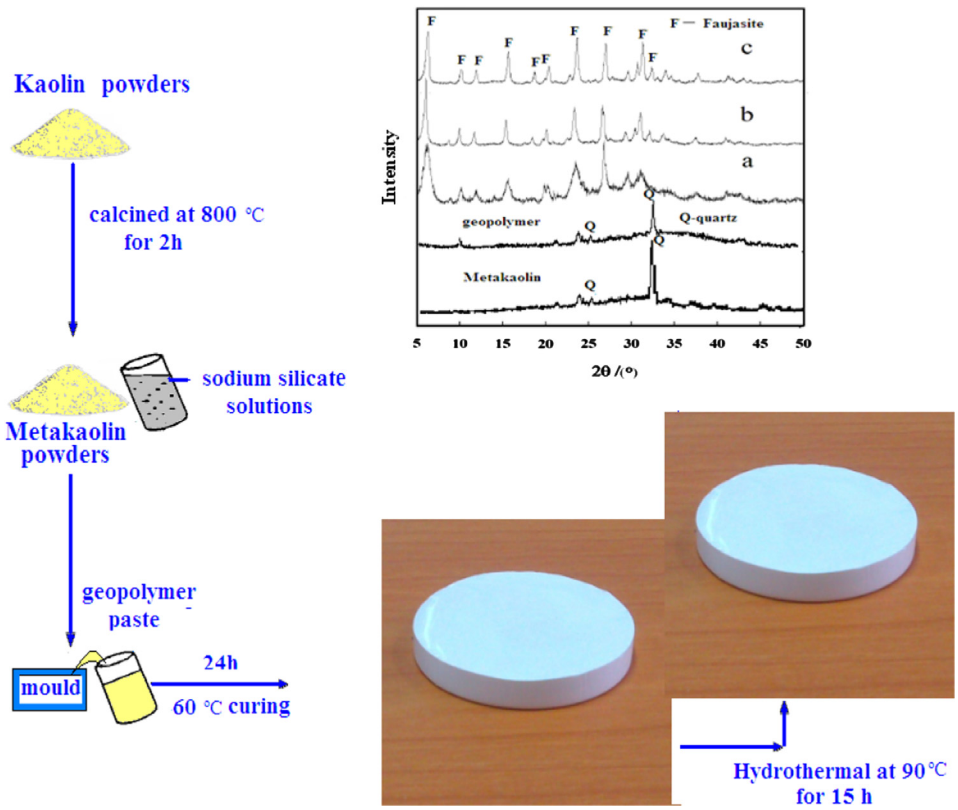

Fig. 1. The processing schematics of FAU zeolite membrane synthesis and the XRD patterns of metakaolin, the geopolymer and the FAU zeolite membranes following varying hydrothermal treatment times: (a) 9 h, (b) 12 h, and (c) 15 h.

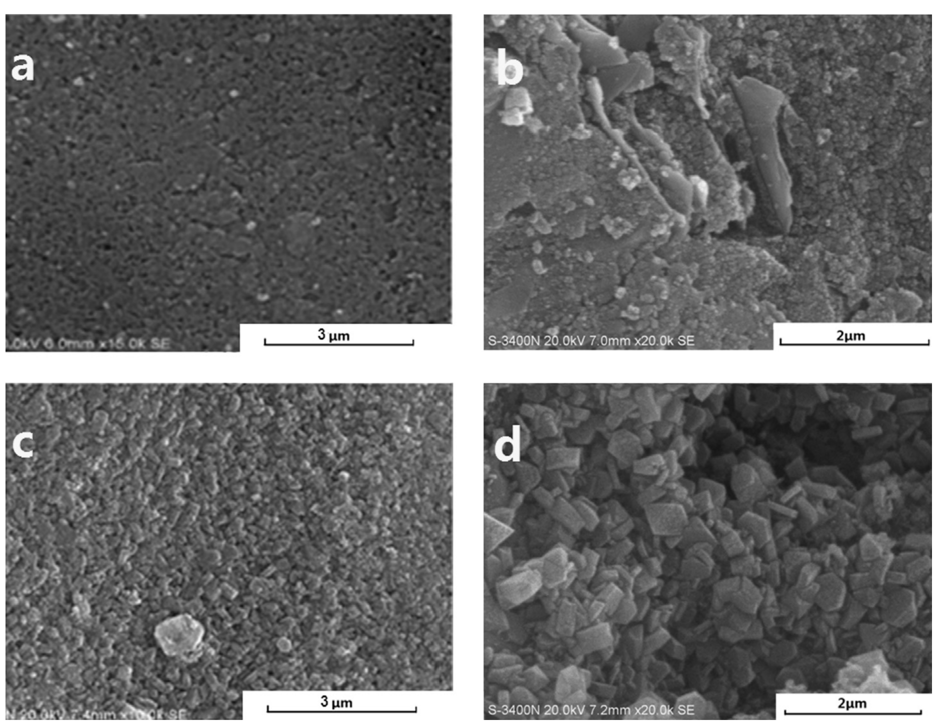

Fig. 2. SEM images of the Na_2O–$3SiO_2$–Al_2O_3 geopolymers and the self-supporting FAU zeolite membrane: (a) the surface of the Na_2O–$3SiO_2$–Al_2O_3 geopolymer, (b) the cross section of the Na_2O–$3SiO_2$–Al_2O_3 geopolymer, (c) the surface of the FAU zeolite membrane, and (d) the cross section of the FAU zeolite membrane.

FAU zeolite membrane (diameter = 40 mm; thickness = 10 mm) was sealed in a membrane fixture. A vacuum was applied to the downstream side and the permeation pressure was maintained below 0.1 kPa. The pervaporation performance was characterized by the permeation flux J and pervaporation separation index P_{SI} [14], using

$$J = m/At \tag{1}$$

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