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Morphological study of yttria-stabilized zirconia hollow fibre membrane prepared using phase inversion/sintering technique

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Abstract

This work describes the preparation of yttria-stabilized zirconia (YSZ) hollow fibre membranes using the phase inversion and sintering process. The study aims to provide generic information on the effects of membrane preparation on the morphology and mechanical properties of YSZ hollow fibre membranes. In this work, ceramic suspensions were prepared by mixing YSZ particles, dispersant, polymer binder and organic solvent using a planetary ball milling machine. This process was followed by extrusion into a coagulation bath via an air gap, drying and sintering process at temperatures ranging from 1250 °C to 1400 °C. The results show that by varying the YSZ loading and YSZ/PESf ratio, different morphologies of ceramic hollow fiber membranes can be obtained due to variations in the viscosities of ceramic suspensions. Similarly, air-gap length between the spinneret and coagulant surface was found to affect the growth of the finger-like structures and sponge-like regions. Varying these parameters also gave significant effects on the mechanical strength of the ceramic membrane due to the change in membrane thickness and compactness. The sintering process had insignificant effects on the membrane morphology but the process can be used to enhance the mechanical strength of ceramic hollow fiber membranes. The optimum temperature of the sintering process was identified. It was found that increasing the sintering temperature further caused a reduction in the mechanical strength due to crack formation in the ceramic hollow fiber membrane. The preliminary performance tests showed that the ceramic hollow fiber membrane sintered at 1300 °C has a pure water flux of 118.4 L/m^2 h. It also has a high PEG rejection with molecular weight cut off (MWCO) of 60 kDa.

Keywords: Yttria-stabilized zirconia; Hollow fiber membrane; Phase inversion; Ceramic suspension; Sintering process

1. Introduction

Ceramics materials are well known for their superior mechanical properties, and high chemical and thermal stability. These properties have resulted in ceramic membranes to have excellent mechanical strength, able to be used in harsh environments and withstand high-pressure operations for backwashing. Their superior thermal, chemical and mechanical properties have widened their range of applications, making ceramic membranes suitable to be used as membrane reactors [1,2], gas separators [3] and fuel cells [4]. These properties have not only contributed in enhancing their lifespan, but also in reducing their maintenance, thereby enhancing the reliability of ceramic membranes.

Preparation of ceramic hollow fibre membranes using the phase inversion technique is applied from the fabrication method of polymeric membranes [5–7]. However, due to the great difference between the polymer system and ceramic system, extensive studies are needed in implementing the phase inversion for ceramic membrane fabrication. Li et al. have documented works on the phase inversion of ceramics [8–11] where they suggested an interesting mechanism they termed as hydrodynamically unstable viscous fingering to explain the formation of macrostructures in a ceramic membrane.

The ceramic membrane obtained through this approach has distinct morphologies, comprising of both sponge-like and

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finger-like structures. The relative length of the finger-like structure and sponge-like region occupies the membrane's cross section, affecting the properties of the ceramic membrane. Typically, the amount of polymer added during the ceramic suspension preparation is relatively low, where the ratio of ceramic to polymer is 10. Kingsbury and Li [8] studied the effect of the viscosity of ceramic suspensions using water as a viscosity enhancer onto the morphology of Al_2O_3 hollow fibre membranes. The results showed sponge-like structures growing from the outer region of the hollow fibre while finger-like structures formed from the lumen. Although a study by Othman et al. [12] also showed a similar pattern, the amount of viscosity enhancer and growth of the length of the sponge-like and finger-like structures were different due to the different ceramic particles used. A study by Liu et al. [9] reported that an increase in the mechanical strength of Al_2O_3 hollow fibre membranes due to an increase in the Al_2O_3 content in the ceramic suspension. The study also concluded that the particle size distribution influenced the mechanical strength of ceramic hollow fibre membranes.

In the spinning process, Kingsbury and Li [8] reported that the variation in the length of air-gaps affected the morphological formation of Al_2O_3 hollow fibre membranes. The membranes were filled by the finger-like structures approximately 50–80% of the cross section of the fibre when the air-gap length varied from 20 cm to 15 cm respectively. A variation in the internal coagulant during the spinning process also affected the membrane morphology as reported by Tan et al. [13] and during the fabrication of LSCF hollow fibre membranes as observed by Yin et al. [14] for YSZ hollow fibre membranes. Another study by Zhang et al. [15] also showed a relative change in the morphology of ceramic membranes when different types of external coagulant were used.

The sintering process is the final stage in the preparation of ceramic membranes. Various studies reported that this process may affect the morphology of the ceramic membrane. As reported by Liu and Li [16] who prepared SCYb hollow fibre membranes, the surface of the ceramic membranes changed with an increase in the sintering temperature from 600 °C to 1600 °C. The ceramic particles started to form grains as the temperature was increased and became completely dense at 1600 °C. Wei et al. [11] indicated that the surface morphology of YSZ hollow fibre membranes became fully dense at a sintering temperature of 1500 °C. The mechanical strength of this ceramic membrane was affected by the sintering process. Liu et al. reported that the mechanical strength of SCYb hollow fibre membranes decreased gradually from >350 MPa to <100 MPa as the sintering temperature was increased from 1350 °C to 1550 °C [17]. A study by Liu et al. [18] also showed a similar trend. However, the highest mechanical strength of SCYb hollow fibre membrane obtained was 60 MPa at a sintering temperature of 1475 °C, which can be considered low. The differences in mechanical strength may be due to the selection of ceramic materials and may also be caused by the preparation steps.

Ceramic membranes have been reported to have a number of advantages in various applications. However, ceramic membranes have their own respective desired morphology depending on the applications of ceramic membranes. From the aforementioned literature, it can be deduced that different ceramic materials used for the membrane fabrication will give ceramic hollow fibre membranes with different morphological structures and mechanical properties. Therefore, the main objective of this study is to provide generic information on the preparation of YSZ hollow fiber membranes and the effects of the preparation procedures on the morphology and mechanical properties of the membranes. YSZ has been chosen in this study due to its hardness, which can enhance the mechanical strength of ceramic hollow fiber membranes. According to Piconi and Maccauro, the maximum bending strength of this material can reach up to 1200 MPa [19]. This will give an advantage when assembling the membranes into membrane modules. Typically, YSZ has been used widely as an electrolyte material for solid oxide fuel cell (SOFC) due to its ability to transport cations at higher temperatures. Additionally, a recent study by Rahman et al. showed that YSZ hollow fibre membranes can also be used as a ceramic support for catalytic reactions of hydrogen production [1]. Compared to alumina, YSZ is an inert material which gives insignificant influence to ethanol steam reforming in the production of H_2. Although ceramic membranes have been demonstrated to offer several benefits, the membrane morphological study is still limited. Thus, this study will correlate a number of preparation parameters, namely, ceramic loading, air-gap length, and sintering temperature, with the morphology of YSZ hollow fibre membranes and their final mechanical properties. Performance tests, which will assist in determining suitable applications for ceramic hollow fibre membranes, will also be carried out.

2. Methodology

2.1. Materials

Commercially available 8 mol% yttria-stabilized zirconia (YSZ) powder with a particle size of 0.3 μm ($d_{50}=0.3$ μm) purchased from Fuel Cell Material was used as the ceramic particles. Polyethersulfone (PESf) (Radel A300, Ameco Performance, USA) was used as the polymer binder, while Arlacel P135 (Polyethyleneglycol 30 Dipolyhydroxystrearate, CRODA) and N-methylpyrrolidone (NMP, QRëC™) were used as the dispersant and solvent, respectively. Tap water was used as the internal and external coagulants.

2.2. Preparation of ceramic suspension

The YSZ powder and PESf were dried and the Arlacel P135 gel was melted in an oven overnight at 60 °C to make sure that there was no moisture trapped. Then, Arlacel P135 was first dissolved in NMP by stirring prior to the addition of the YSZ powder. The dispersion was then milled in NQM-2 planetary ball mill for 48 h with 20 mm agate mill balls to ensure that the ceramic particles, solvent and additive were mixed well. The milling was continued for another 48 h after the addition of PESf to ensure that the polymer binder did not form large clumps and was fully dissolved. Next, the ceramic suspension