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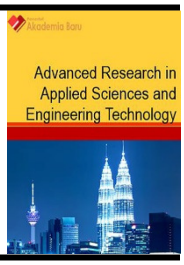

Effect of dipping number on the fabricated silica ceramic membranes via sol dip-coating method

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ABSTRACT

Membrane technologies have received high interest in the separation gas mixture. The ceramic inorganic membranes have possessed high permeability, excellent thermal, chemical and mechanical stabilities compared to conventional polymer membranes. This work presents the fabrication of silica ceramic membrane by sol dip-coating method. The tubular support was dipped into the solution of tetrethylorthosilicate (TEOS), distilled water and ethanol with the addition of nitric acid as a catalyst. The fabricated silica membrane was then characterized by (Field Emission Scanning Electron Microscope) FESEM and (Fourier transform infrared spectroscopy) FTIR to determine structural and chemical properties at different dipping number. FESEM images indicate that the silica has been deposited on the surface fabricated ceramic membrane and penetrate into the pore walls. However, number of dipping did not affect the intensity peak of FTIR analysis.

1. Introduction

In this recent time, the interest in application of membranes for various gas separation has increased significantly in term of economic importance as compared to other conventional techniques, membrane separation is an alternative energy saving [1]. Due to the increase of environmental regulation, membranes is favourable as it reduces waste disposal expenditure and allow the recovery and recycling of the materials which resulting in economic advantage [2]. Membranes categories can be divided into organics, inorganics and mixed matrix membranes [3].

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Inorganic membranes have received great attention as it overcomes the limitation of organic membranes in the term of chemical and thermal resistance [4]. Inorganic membranes divided into two categories, which are porous and nonporous [5]. The porous inorganic membrane is favourable as they can tolerate at higher temperature and limit the connection between permeability and selectivity [6]. Ceramic inorganic membranes possessed the characteristic of high stability, resistance to chemicals and solvents, long term durability and high mechanical strength [2].

There are various ways to prepare ceramic membrane based on material used and required characteristic (porosity, pore size, thickness) [5]. Ceramic membrane can be fabricated using methods such as solid state, tape casting, slip casting and sol-gel [7] Sol-gel is the most preferable process for ceramic membrane fabrication as it possessed many advantages, such as preparation of materials with exceptional purity and homogeneity, excellent BET ((Brunauer, Emmett and Teller) surface area and also well-defined distribution of pore size [8]. Sol dip-coating method is one of the sol-gel technique. Sol dip-coating method is used to fabricate ceramic membrane when reduction of pore size is in needed [5]. Sol dip-coating method is preferable because it offers advantages such as the simplicity of its technique, produce a product with uniform surface and the ability to control the structure of the membranes pore [9]. Example of ceramic membrane that can be fabricated using the sol - gel method is silica membrane, which then resulting in greater surface area and microporosity [5].

In this paper, silica ceramic membranes are fabricated using sol dip-coating method. The supports were dipped in the prepared silica solution by performing a different number of dipping that are one, two and three dipping. Then, the fabricated membranes with different number of dipping were characterized in order to study the effect of number of dipping on its morphology and chemical properties.

2. Research Methodology

A. Preparation of ceramic membrane

The membranes were fabricated using silica sol dip-coating method. The solution was prepared by mixed TEOS, distilled water and ethanol together at 298 K with vigorous stirring. 12 ml of nitric acid was added into the solution as a catalyst. Three supports (10 inch Doulton OBE) were used in this experiment. The supports were dipped into the solution and the dry at room temperature for 24 hours. Then the samples were calcined at 773 K with holding time of three hours. The dipping, drying and calcine process was repeated for second dipping for two of the samples, and third dipping for only one of the samples.

B. Membrane characterization

The morphology of the membrane surface and support were determined using the field emission scanning electron microscope (FESEM) (JSM 6700F, JEOL). The Fourier transform infrared spectroscopy (FTIR) (Perkin Elmer System spectrum 100) was employed in determining the functional group of the membrane.

3. Results and Discussion

The silica ceramic membrane was fabricated in order to increase separation and permeation characteristics. The major aims of the fabrication to modify the pore size of the membrane by silica sol. Fabrication of the ceramic membrane was done through silica sol dip-coating method, by the deposition of silica-based solution on the surface of a porous ceramic support. This technique was performed under neat and dust-free environment at 773 K.

Figure 1 (a) shows the FESEM image of surface of the ceramic support 1000x magnification, while Figure 1 (b), (c) and (d) show the surface image for different dipping number which are first, second and third dipping, respectively. FESEM analysis was employed in order to explore and study the surface morphology of the support and the fabricated silica ceramic membranes produced. Based on FESEM images, it shows that the surface of the support has clear surface. However, after the dipping, there are foreign substances on the membrane surface which proves that the silica already penetrate onto the surface of the fabricated membrane.

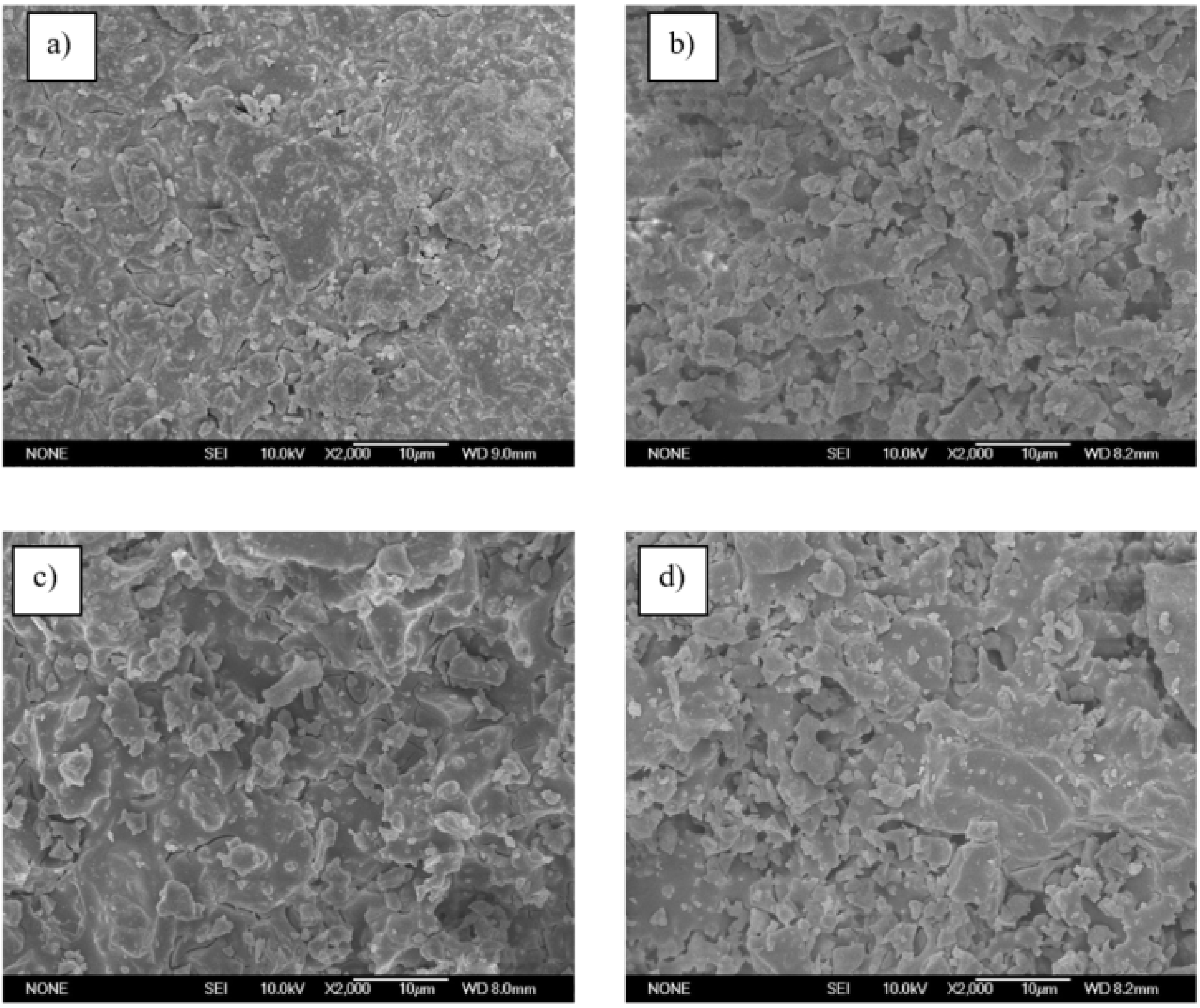

Fig. 1. FESEM image of a) support and fabricated silica ceramic membrane via sol dip-coating method at different dipping number; b) first dipping; c) second dipping; d) third dipping

Figure 2 shows FTIR adsorption spectra of fabricated silica ceramic membranes at different dipping number. It was discovered that the major peak in FTIR adsorption spectra of the fabricated membrane with different number of dipping is around 1060 to 1090 cm-1. The broad absorption band in the region around 1060 to 1090 cm-1 correlated to the Si-O-Si bond of mesoporous silica, which then verify the existence of silica in the fabricated membrane. However, increasing in number of dipping did not affect the intensity of the FTIR peak as shown if the FTIR result.

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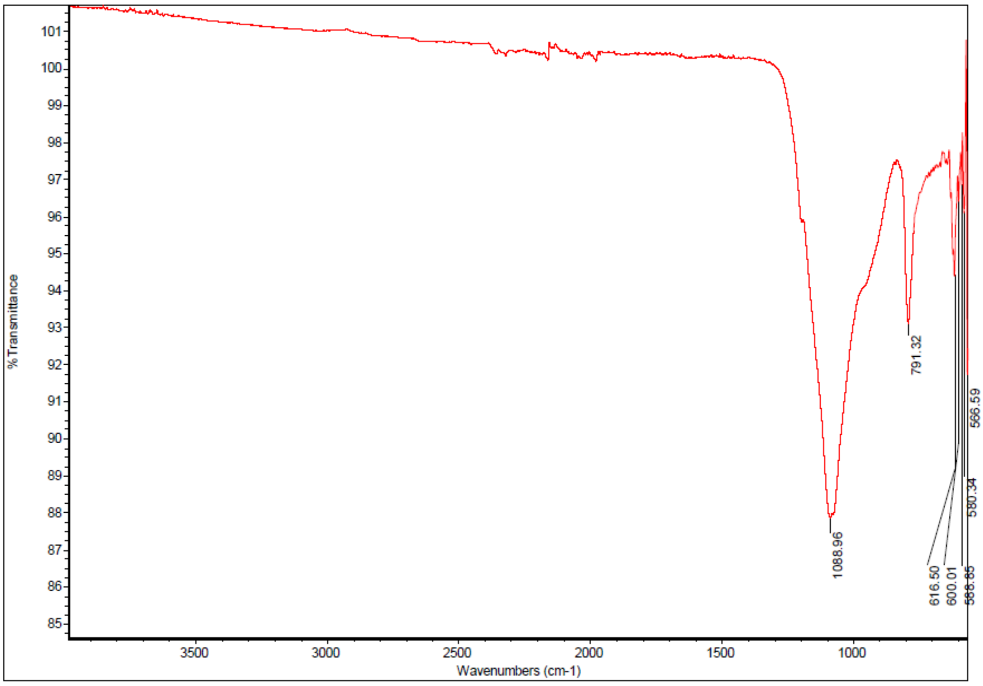
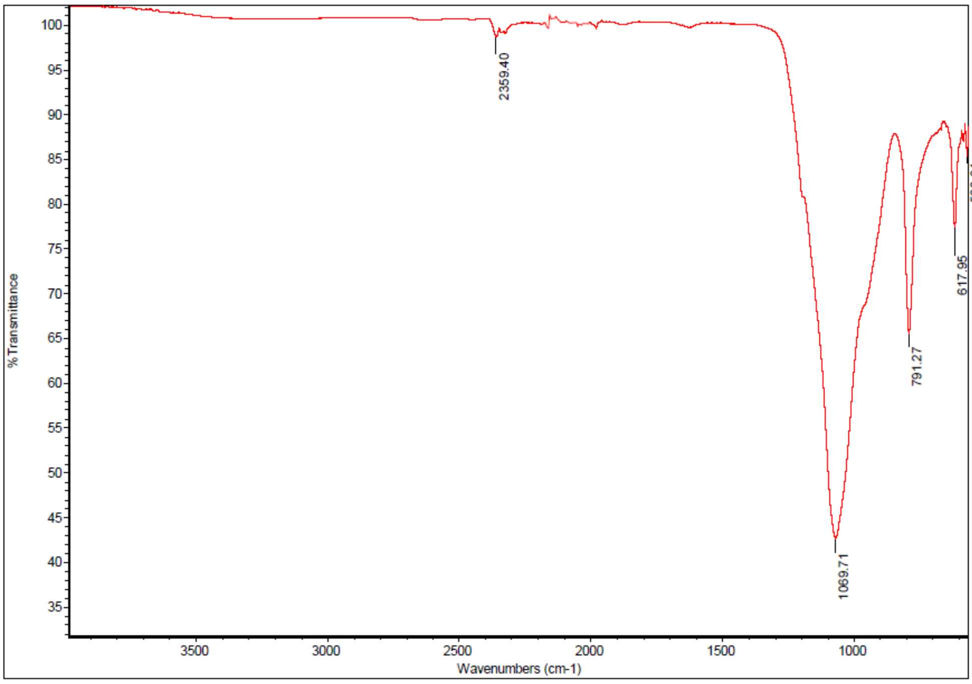
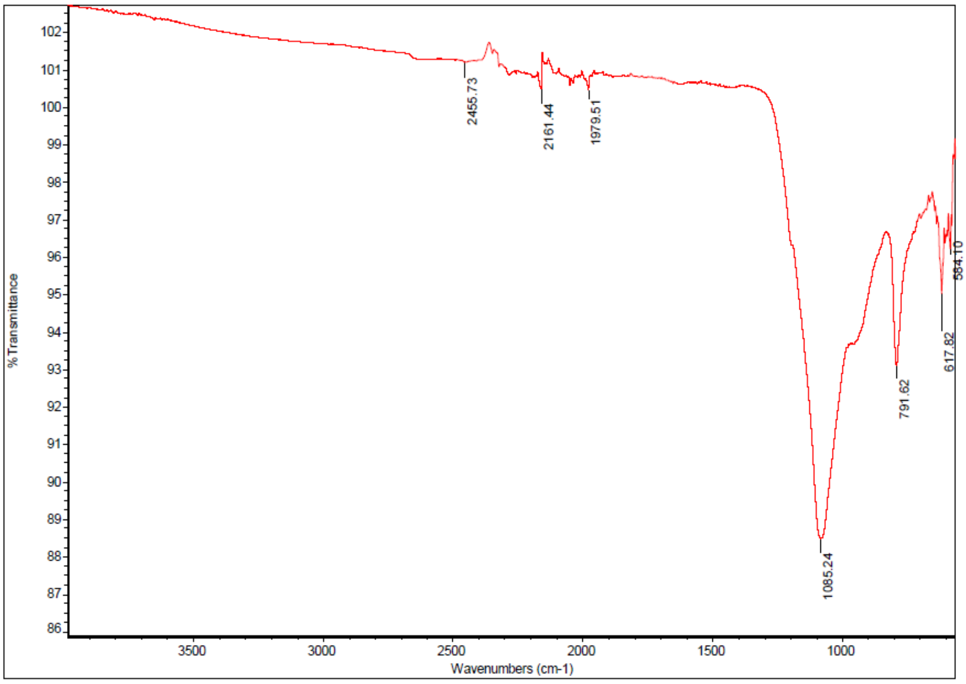
Number of dipping affected the pore size reduction of the fabricated ceramic membranes. Increasing in number of dipping will decrease the size of the membrane pore as the silica sol was deposited on the membrane surface and penetrate into the membrane pore walls. Fabrication of the ceramic membrane through sol dip coating, resulting in uniform pore size distribution, while the number of dipping affecting the characteristics of the fabricated membrane and also the reduction of the membrane pore size.



(a)



(b)



(c)

Fig. 2. FTIR image of fabricated silica ceramic membrane via sol dip-coating method at different dipping number; a) first dipping; b) second dipping; c) third dipping

4. Conclusion

In this study, the fabrication of ceramic membrane was done by sol dip-coating method. The differences in characteristics of membranes were investigated as they were fabricated by using different numbers of dipping. The differences in dipping numbers affect the morphology and also the pore size of the membrane. In FTIR analysis, the intensity peak did not affected by increased number of dipping. This research will be continued by varying at different parameter such amount of acid and calcination temperature in fabrication of silica ceramic membrane via sol dip-coating. All the fabricated membranes will be compared to their optimum characteristic to be applied for biogas separation application.

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