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Effect of Calcination Temperature on the Silica Ceramic Membrane Synthesized via Sol-gel Dip-coating Method

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ABSTRACT. Ceramic membranes became one of the most important ceramic products because of their numerous benefits to many applications especially in gas separation processes. The attractive features offered by this technology include high energy efficiency, simplicity design and construction of membrane modules and environmental compatibility. The aim of this project is to get the optimum calcination temperature thus will give the optimize pore size for separation gas without cracking or pinholes the membranes. Therefore, the silica ceramic membrane was fabricated via sol-gel dip-coating method and the effect of calcination temperature on the membrane pore size was investigated. The ceramic support was dipped in the mixture solution of tetraethyl orthosilicate (TEOS), distilled water, ethyl alcohol and nitric acid with the desired molar ratio followed up by the calcination process at 400 °C, 500 °C and 600 °C. For X-ray diffraction (XRD) results, the fabricated silica ceramic membrane shows the existence of silicate hydrate when calcined at 400 °C, 500 °C and 600 °C. The XRD analysis showed the highest peak intensity at 22.5° which proved the presence of silica. From the field emission electron microscopy (FESEM) images, the pore size of the ceramic support was around 0.5 to 0.6 µm. After the silica ceramic membranes were fabricated, the pore size no longer visible under the FESEM proves that the pore size of the membranes was reduced. Fourier transform infrared spectroscopy (FTIR) showed adsorption spectra of the fabricated membranes with different calcination temperature. The broad band in the region around 1060 to 1090 cm^{-1} correspond to the O-Si-O bond of mesoporous silica altogether confirming the existence of silica. Based on the result analysis, the suitable calcination temperature at 500 °C with less crack and more consolidated surface membrane.

KEYWORDS: Silica,Ceramic membrane, Calcination temperature, Sol-gel, Dip-coating
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1. INTRODUCTION

Currently, the interest in application of membranes for various gas separation has increased significantly in term of economic importance as compared to other conventional methods. Due to the increase of environmental regulation, membranes are favourable as it reduces waste disposal expenditure and allow the recovery and recycling of the materials which results in economic advantage [1]. Membranes categories can be divided into organics, inorganics and mixed matrix membranes [2]. Inorganic membranes have received great attention as it overcomes the limitation of organic membranes in term of chemical and thermal resistance [3]. Inorganic membranes can be divided into two categories, which are porous and nonporous [4]. The porous inorganic membrane is favourable as they can tolerate higher temperatures and limit the connection between permeability and selectivity [5]. Ceramic inorganic membranes possess the characteristics of high stability, resistance to chemical and solvents, long term durability and high mechanical strength [1].

There are various ways to prepare ceramic membrane based on material used and also required characteristic (porosity, pore size, thickness) [4]. Dip-coating method are the most preferable process for ceramic membrane fabrication as it possessed many advantages, such as preparation of materials with exceptional purity and homogeneity and well-defined distribution of pore size [6,7]. Dip-coating method is used to fabricate ceramic membrane when reduction of pore size is required [4]. Example of ceramic membrane that can be fabricated using the dip-coating method is silica membrane, which then resulting in greater surface area and microporosity [4]. Basically, the processing variables such as temperature, particle size, applied pressure, particle packing, compositions and calcination atmosphere influence the microstructure that is produced at different calcination temperatures. Hence, the calcination temperature will give different pore size, the higher the calcination temperature, the smaller the pore size. Gaber et al. [8] mentioned that the pore size of the membranes decrease as the calcination temperature increase due to the external pressure during heating [8].

Therefore, in this paper, the silica ceramic membranes are synthesized by sol-gel dip-coating process at different calcination temperature.The synthesized silica ceramic membrane has been characterized by XRD, FESEM and FTIR to identify the physical and chemical properties of the membrane.

2. Metarials and Methods

2.1 Membranes Fabrication. The ceramic support used was 10 inch Doulton OBE Ceramic with 32 mm internal diameter, 48 mm outer diameter while the support length is 200 mm. The water used throughout was distilled water. Tetraethyl orthosilicate (TEOS) was supplied by Sigma Aldrich and nitric acid by R&M Chemical. The ethanol also used was supplied by Systerm.

The silica sol will be applied to the porous ceramic supports of tubular configuration via sol-gel dip-coating method. In this process, TEOS, distilled water and ethyl alcohol (1:4.7:3.8 molar ratio) were mixed at 25 °C. After that, acid nitric added into the silica sol. Then, the ceramic support was dipped into this sol. The membrane was drying 24 hours in atmosphere overnight and continued with the calcination process for three hours. In this works, the calcination temperatures have varied to 400 °C, 500 °C and 600 °C respectively. The process of dipping the support in sol and calcination was repeated for second and third dipping number.

2.2 Membranes Characterization. The morphology of the membranes surface and support were determined using the field emission scanning electron microscope (FESEM) (JSM 6700F, JEOL). X-ray diffraction (XRD), was used to determine the element present of the fabricated membrane. The Fourier transform infrared spectroscopy (FTIR) (Perkin Elmer System spectrum 100) was employed in determining the functional group of the membrane. By using FTIR, it can verify the presence of silica in ceramic membranes and provide the information on the structural bonding.

3. RESULTS AND DISCUSSION

Fig.1 demonstrated the XRD analysis of prepared silica ceramic membrane at different calcination temperatures, which are 400 °C, 500 °C and 600 °C. The XRD peak shows existence of silica hydrated (card number 71-6245) with highest peak 22.5°. Moreover, the peak intensity for ceramicmembrane calcined at 500 °C and 600 °C are lower as compared when ceramic membrane calcined at 400 °C.

This explained that the silica grain growth has achieved optimum at 600 °C, indicated by the decrease in intensity of aluminum phosphate. The detected reflections were solely the expected reactions from ceramic support ($AlPO_4$) and fabricated ceramic membrane (SiO_2), indicating that no other phase was present in the membrane.

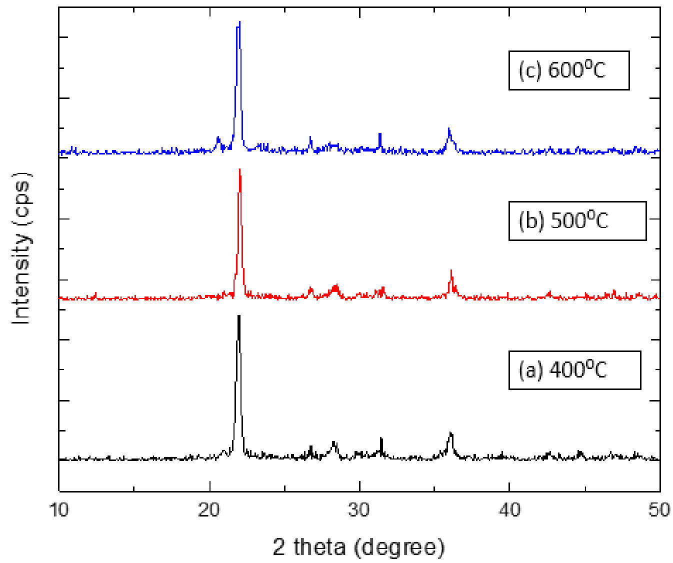

Fig. 1 XRD pattern of fabricated silica ceramic membrane at different calcination temperature, (a) 400 °C, (b) 500 °C and (c) 600° C

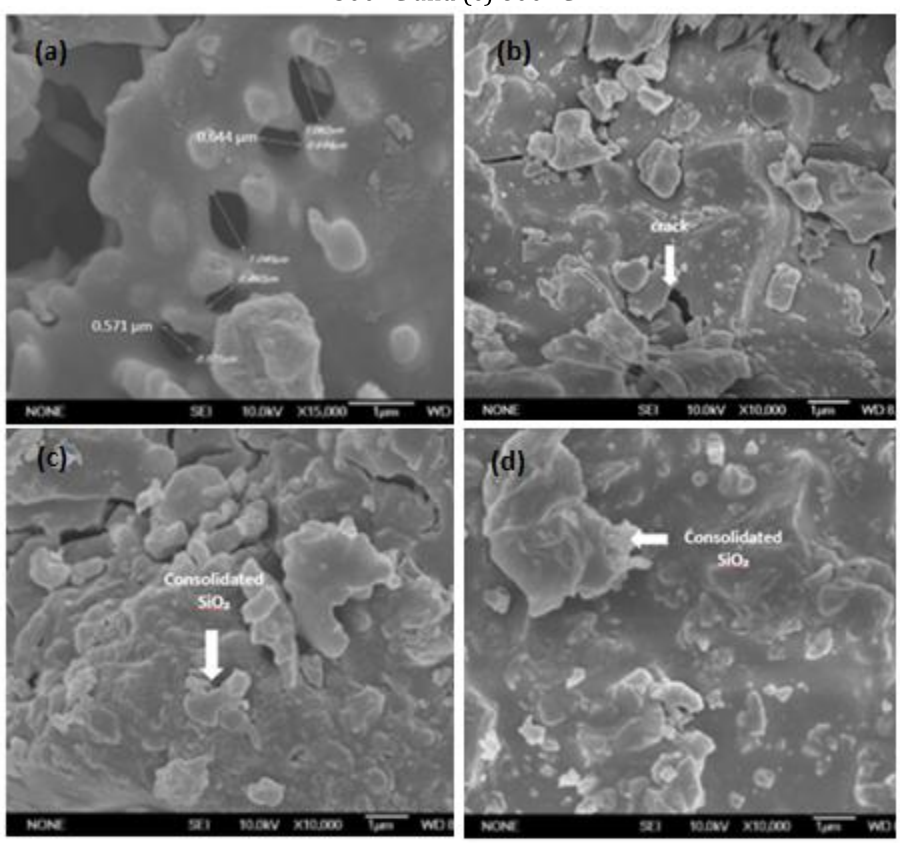

Fig. 2 FESEM images of fabricated silica ceramic membrane at different calcination temperature, (a) ceramic support; (b) 400 °C, (c) 500 °C and (d) 600 °C

Fig. 2 showed the FESEM micrographs of ceramic support and fabricated silica ceramic membranes at different calcination temperature of 400 °C, 500 °C and 600 °C. Besides that, the surface of raw ceramic support show highly porous structure (0.5 to 0.6 µm in diameter) while for the surface of fabricated silica ceramic membranes are more consolidated.

From the FESEM images, it was observed that there has a crack when calcined at 400 °C. Whereas,, when calcined at 500 °C and 600 °C, the silica was consolidated on the surface structure of the membrane. This was a proof that the silica membrane has coated the ceramic support completely, thus covering all the pores on its surface. Next, it was also observed that the silica membrane more consolidated when calcined at 600 °C was noticeably lesser compared to the membrane calcined at 500 °C due to the deterioration of O-Si-O bond start at 565 °C at elevated temperature. This finding was also confirmed by Gaber et al. [7].

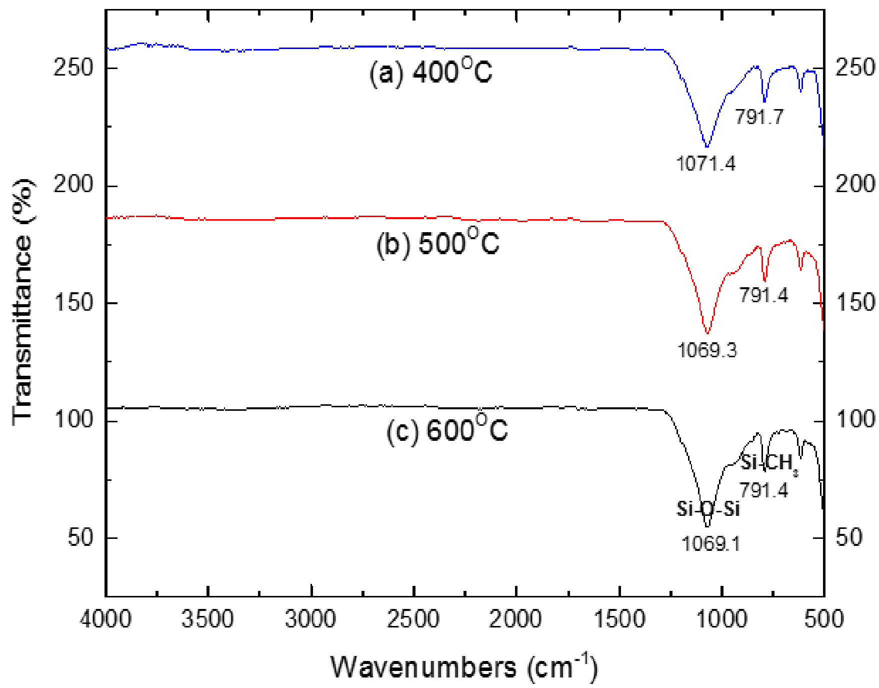

Fig. 3 FTIR adsorption spectra of prepared ceramic membranes at different calcination temperature

Fig. 3(a) shown the FTIR adsorption spectra of ceramic support while in Fig. 3(b) shows the FTIR adsorption spectra of the prepared ceramic membranes at different calcination temperature. Whereas in Fig. 3(b), it shown the highest peak intensity for membrane calcined at 600 °C which means the optimum temperature for silica grain growth. Based on this observation the lowest peak intensity presented for the membrane which calcined at 400 °C due to the deterioration of the O-Si-O bond as mentioned previously. The broad band in the region range between 1060 to 1090 cm^{-1} correspond to the O-Si-O bond of mesoporous silica, altogether verify the existence of silica. IR adsorption at 791.4 cm^{-1} shows the modification of Si-CH_3 group on the silica ceramic membrane surface as mentioned by Bowen et al. [9].

Based on XRD, FESEM and FTIR results, the fabrication of silica ceramic membrane are significantly affect physical and chemical at different calcination temperature at 400 °C, 500 °C and 600 °C. The result also revealed that the the silica ceramic membrane calcined at 500 °C can functionally reduce the present of crack and the surface ceramic membrane are more consolidated.

4. SUMMARY

As a conclusion, the silica ceramic membrane via sol-gel dip-coating method is successfully fabricated. The silica ceramic membranes have been calcined at different calcination temperatures 400 °C, 500 °C and 600 °C. The fabricated ceramic membrane is characterized physically and chemically. The silica ceramic membranes have been characterized by XRD, FESEM and FTIR. The XRD pattern showed the aluminum phosphate element for ceramic support and silicate hydrate element for prepared silica ceramic membrane. Under XRD analysis, the highest peak at 22.5° which refer to the presence of silica. FESEM micrographs showed consolidated SiO_2 on the surface of ceramic support when calcined at different temperatures. It was discovered that the pore size no longer visible when the silica ceramic membrane calcined at 600 °C which proved that the ceramic support was fully coated with silica membrane. Under FTIR analysis, the highest IR adsorption is between 1050 to 1090 cm^{-1}, which is refer the presence of O-Si-O functional group. In terms of calcination temperature, it shows that the optimum temperature was at 500 °C with less crack and more consolidated surface membrane.

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