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Effect of Kaolin Particle Size Towards Preparation of Kaolin Ceramic Membrane

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Abstract: The purpose of this work is to study the effect of kaolin particle size for the preparation of low cost ceramic membrane suspension and ceramic membrane structure. Kaolin particle size is categorized into two categories; i) ≤ 1µm and ii) ≥ 1 µm. The suspension is prepared via stirring technique under 1000 rpm at 60˚C. The particle size of kaolin is characterized using field emission scanning electron microscope (FESEM) and the prepared suspension is characterized in term of its viscosity. Results indicate that the particle size gave significant effect to the viscosity of ceramic membrane suspension. Preliminary data showed that kaolin with particle size ≤ 1µm resulted ceramic membrane with dense structure.

Keywords: Kaolin, ceramic suspension, particle size

1. Introduction

There are two types of membranes in membrane technology: polymer membranes and inorganic membranes. It should be noted that polymer membranes are the most common and well-known membranes. This is due to the low cost of manufacturing polymeric films. However, because inorganic membranes can solve many problems in polymer membranes, there is great interest in inorganic membrane research. Metal film, carbon film, and ceramic film are three forms of inorganic film. Unlike polymer membranes, ceramic membranes usually have unique characteristics, the most

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notable being heat resistance, which allows them to function at higher temperatures, and chemical stability, which allows them to work in corrosive conditions as well as Play a role in a wider pH range. Ceramic membranes are commercially available, but due to the use of expensive industrial oxides (alumina, zirconia, silica and titania), their cost is quite high, and they usually require high sintering temperatures (> 1400° C) [1, 2]. Therefore, these shortcomings are still one of the main limitations of commercial ceramic membranes. It is strongly recommended to study cheaper alternative ceramic materials and/or lower sintering temperatures to construct economically competitive membranes.

In the past ten years, people have been interested in the production of ceramic membranes from low-cost natural raw materials such as clay, especially MF membranes [3]. It is well documented that local clay is often used to construct flat and tubular ceramic membranes. For example, Moroccan, Algerian and Indian clays have proven their use as membrane materials in a wide range of applications, including industrial wastewater treatment, sterilization, and removal of chromate from aqueous solutions. In addition, MF membranes made of clays such as kaolin exhibit excellent performance in water treatment, especially in the purification of industrial wastewater and seawater desalination. Due to its unique physical properties, such as limited plasticity and high membrane refractory properties, kaolin has attracted the most attention in the manufacture of low-cost ceramic membranes [4]. In addition, kaolin is hydrophilic, which is very suitable for making water filtration membranes. Therefore, low-cost kaolin membranes provide new insights for various separation/purification applications, such as oily wastewater treatment, support for gas separation membranes, and catalytic substrates.

The phase inversion method is usually used to make polymer membranes, which results in a unique asymmetric structure. Loeb and Sourirajan demonstrated the first high-throughput phase inversion membrane in 1963, using phase inversion technology in polymer membranes to remove salt [5]. Because of this advantage, phase-inversion ceramic supports have been used for decades. However, there are few studies on the application of this technology to the manufacture of ceramic substrates. This is because phase inversion technology is a difficult but unique manufacturing technology in which various elements affect the final structure. Particle size is a common problem, which affects the use of phase inversion methods to manufacture ceramic substrates. Particle size is considered to be one of the most important components in the manufacture of ceramic membranes because it regulates the generation of porous structure and pore size. According to Fletcher [6], powders of different sizes will have different shapes and densities, which can lead to segregation during solution mixing.

Therefore, the goal of this study is to prepare the low cost ceramic membrane from kaolin clay at two type of particle size ($\leq 1\mu m$ and $\geq 1\mu m$) via phase inversion and sintering technique. In addition, the relationship of particle size and its effect towards the characteristic of the membrane suspension prepared was studied. Prior to the preparation of low cost ceramic membrane using phase inversion process, characterization towards the ceramic suspension with different kaolin particle size were investigated through scanning electron microscopy (SEM), FTIR and viscosity. Thereby, the low cost ceramic membrane was characterized using SEM.

2. Experimental

2.1 Materials

Ceramic membrane suspension were prepared using polyethersulfone (PESf) as polymeric material, N, N, methyl pyrrolidone (NMP) as solvent, kaolin with two group of particle size ($\geq 1\mu m$ and $\leq 1\mu m$) as raw material and distilled water was used as and non-solvent coagulant bath. All chemicals purchased in this work were used without further purification.

2.2 Preparation of Ceramic Membrane Suspension and Ceramic Membrane

According to Figure 1, ceramic suspension is prepared via phase inversion and containing three primary components: (i) raw material, (ii) polymer (binder), and (iii) solvent. Prior to the preparation of ceramic suspension, PES as binder and kaolin clay were dried in an oven at 60°C for at least 24 hours. This drying procedure is carried out to eliminate moisture content that has accumulated during storage. To begin the preparation of ceramic suspension, a dope solution was prepared in the same manner as a polymeric membrane in literatures [7, 8]. Accordingly, NMP and PESf were stirred for 4 hours until the suspension became homogeneously combined. After 4 hours, kaolin powder was poured into the suspension and swirled for at least 24 hours using a magnetic stirrer Yellow MAG HS 7 S2 (IKA) set to 60°C and a hot plate stirrer. The purpose of aluminum foil as can be observed in the Figure 1 is to protect the solution from moisture.

In thus study, ceramic membrane prepared via phase inversion and sintering technique according to the Rosalam Sarbatly [9]. Firstly, PES was gently added to a solution of NMP and aggressively agitated until fully dissolved. The kaolin was then added to the PESf/NMP solution. For a totally homogeneous suspension, the suspension was agitated at 1000 rpm for 48 hours at 60°C. To be noted, two ceramic membrane suspension was prepared at two types of kaolin particle sizes ($\geq 1\mu m$ and $\leq 1\mu m$). Afterwards, the suspension was characterized using the viscosity test.

The kaolin ceramic membrane was then prepared in the shape of a flat sheet ceramic membrane. Using a casting knife, the suspension was poured onto a clean and smooth 15 x 15 cm glass plate at room temperature. Using adhesive tape on either side of the glass plate, the thickness of the membrane was kept to within 1.5 mm. At room temperature, the cast film was immediately submerged in a non-solvent coagulant bath. For 24 hours, the solidified ceramic membrane

was dried at room temperature. By cutting the membrane before sintering at 1100°C, the desired form was produced. After the sintering procedure, the final items were obtained.

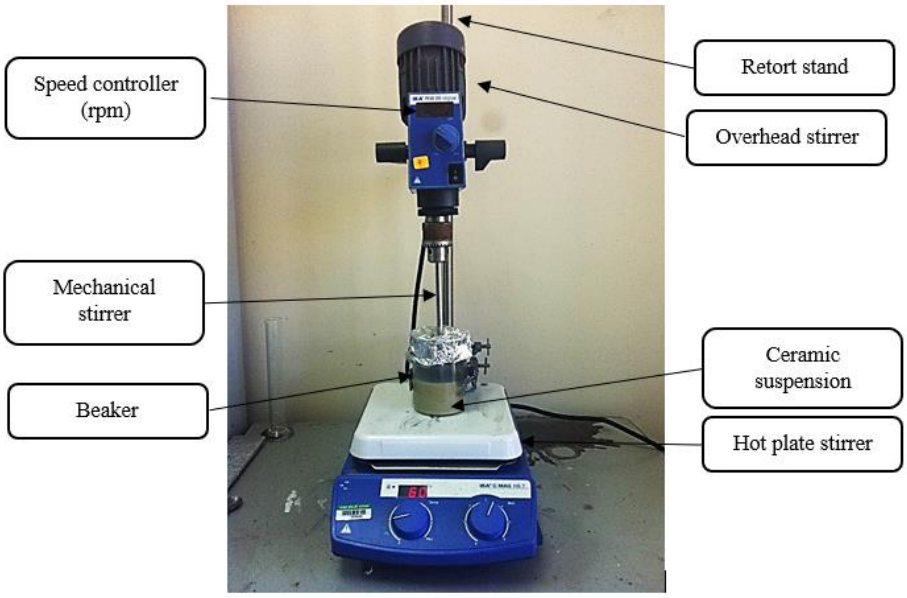

Fig. 1 - Experimental setting for ceramic suspension preparation

2.3 Characterization

Field emission scanning electron microscopy was used to characterize the structure of kaolin powder at different particle size. Fourier transform infrared (FT-IR) spectra using KBr pellets over the range of 4000 to 400 cm^{-1} was used to measure kaolin powder structure and OH-band and stretching, respectively. The characterization of the ceramic suspension with different kaolin particle size were characterized in term of viscosity test using Brookfields Programmable HADV-IV^{+} Rheometer.

3. Results and Discussion

Figure 2 shows the morphology of kaolin, which is divided into two particle sizes. As shown in the figure, the FESEM scan shows that the kaolin is aggregated within the grain size. Besra et al. [10] established the relationship between agglomeration and viscosity. Furthermore, Xin et al. [11] studied the influence of the properties of the powder and the interaction between the binder and the particles during the agglomeration of the melt. Since PES is used as a binder and kaolin is used as a powder, the same principle is used in this experiment. In the case of suspensions, both the volume fraction and the maximum volume fraction can have an impact. The maximum volume fraction (the maximum volume of particles that can be added to the fluid) can be considered as the amount of free space in which the particles must move, and the effect on viscosity was studied using the results of Figure 3.

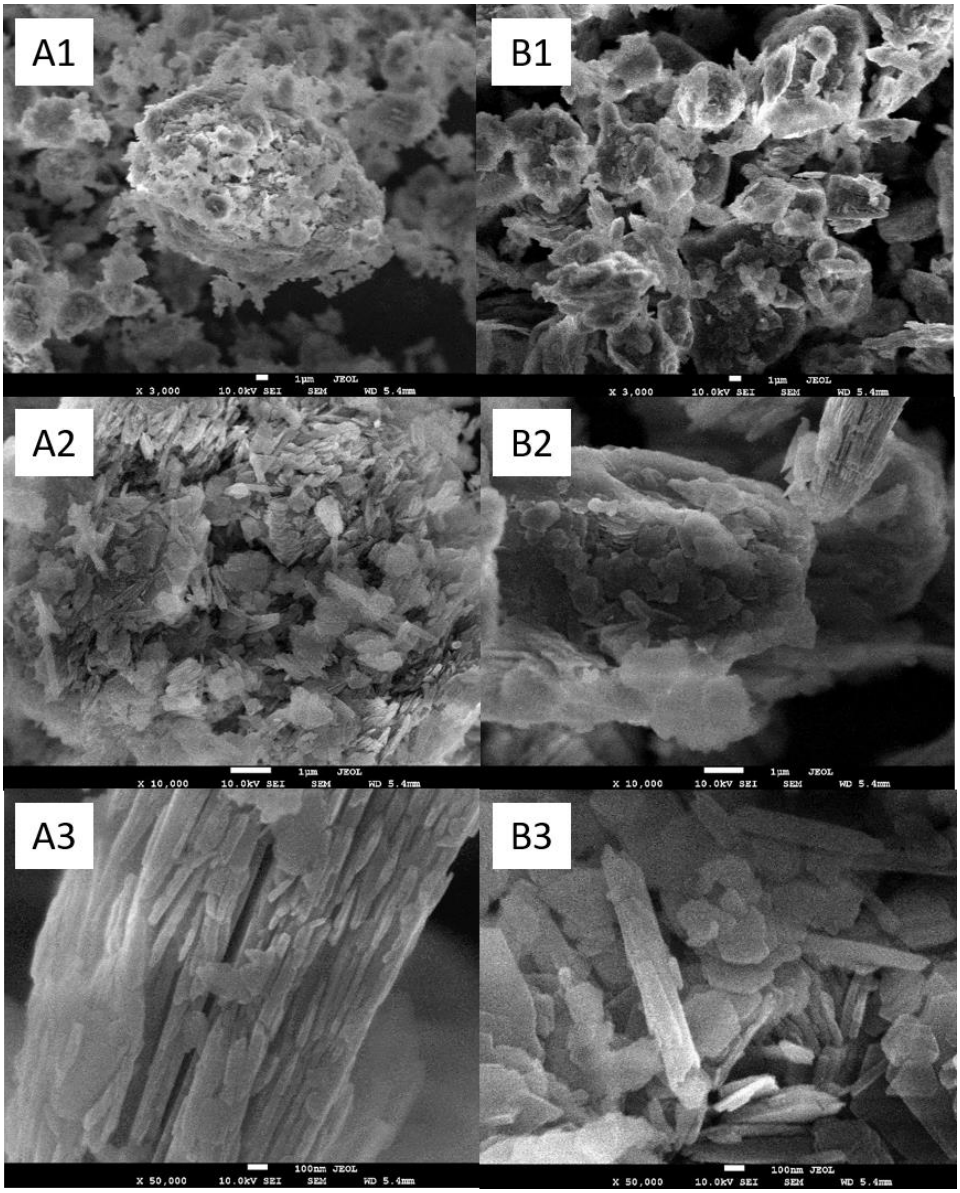

Fig. 2 - FESEM images of kaolin at different magnification of different particle size of kaolin as raw material for ≤ 1µm (A1-A3) and ≥ 1µm (B1-B3), respectively

Fourier transform infrared (FTIR) technology is used to obtain the infrared spectrum of absorption, emission and photoconductivity. In this work, FTIR spectroscopy was used to study the difference in kaolin particle size. It can be seen from the results that there is no significant difference in the FTIR spectra of kaolin particle sizes less than 1 m and greater than 10 µm. The stretching vibration of the -OH group in the kaolinite-gibbsite lattice is represented by the absorption band at 3683-3618 cm^{-1} [12]. The bands at 3450cm^{-1} and 1650cm^{-1} correspond to the stretching vibrations of the water molecules, while the bands at 1114cm^{-1}, 991cm^{-1} and 797cm^{-1} represent the Si-O-Si and Si-O groups. At the network, respectively. Finally, the spectrum shows that there is no chemical interaction between the effects of particle size [13].

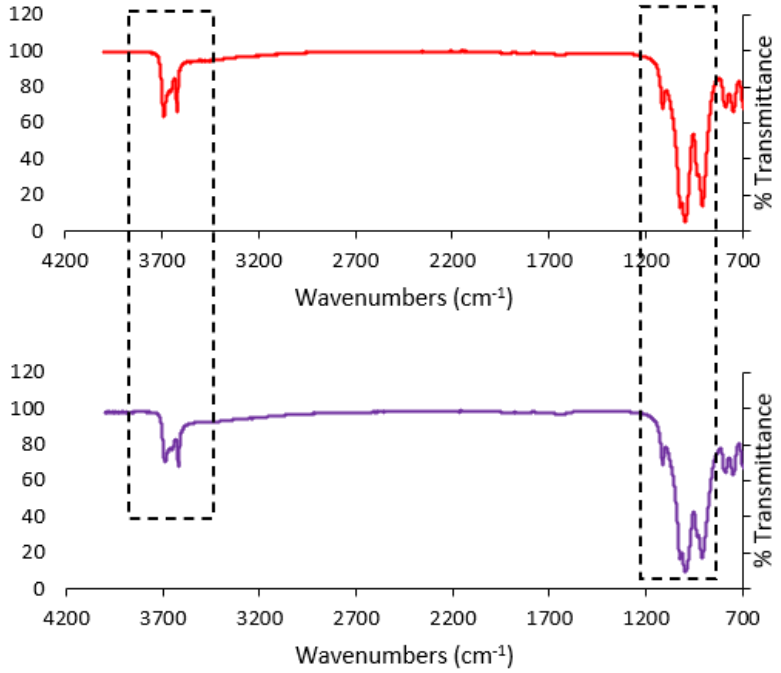

Fig. 3 - FTIR analysis of kaolin at different particle size of kaolin as raw material

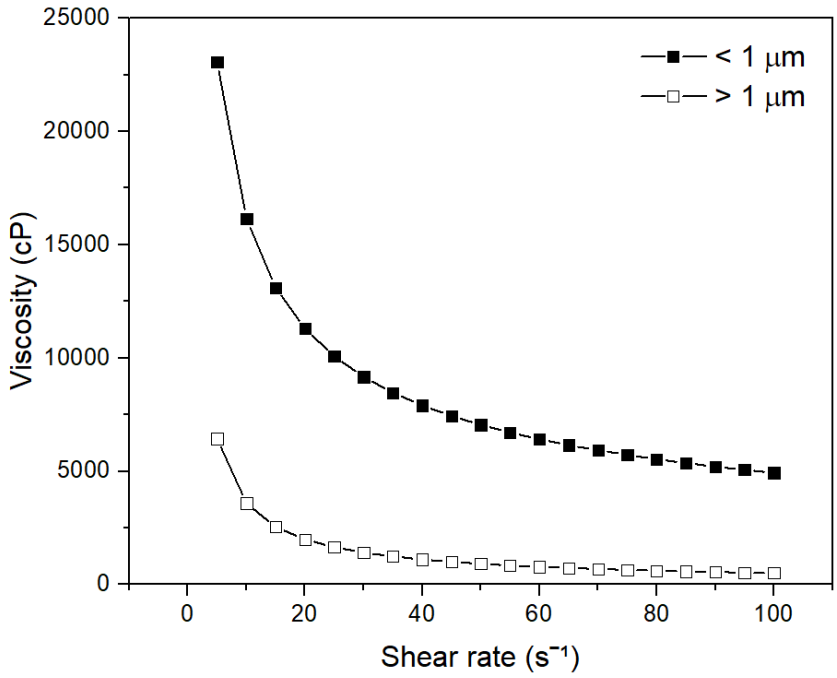

Fig. 4 - Viscosity vs. composition ceramic suspension at different particle size

According to Jamie Fletcher [6], many parameters, including particle size distribution and the percentage of solids present, affect the rheology of the suspension. Keeping the mass of suspended particles constant while reducing the solid particle size will increase the number of particles in the system. Figure 4 shows the viscosity of a ceramic suspension prepared by stirring various kaolin components and particle sizes. It can be seen that for kaolin particles of two sizes, the viscosity of the ceramic suspension increases with the increase of the kaolin component. However, the viscosity of smaller kaolin seems to be much higher than that of kaolin larger than 10 µm. This is because the fluid is shear thinning (viscosity decreases at higher shear rates), and the smaller the particles, the higher the viscosity. In addition, the link indicates that the viscosity increases with increasing volume ratio. As the volume percentage of kaolin particles in the system increases, the particles become tighter, preventing the kaolin particles in the membrane suspension from moving freely. In addition,

kaolin of less than 1 m seems to be more likely to agglomerate than kaolin of more than 10 m. This is because agglomeration often occurs in ultrafine particles due to the high cohesion between the main particles and the narrow spaces between the particles [14]. Therefore, the viscosity of the ceramic suspension is expected to affect the characteristics of the membrane.

The link indicates that as the volume fraction increases, the viscosity also increases. This is explained in the previous part of Equation 1. As the volume percentage of kaolin particles in the system increases, the particles become tighter, preventing the kaolin particles in the membrane suspension from moving freely. This phenomenon is related to the findings in Figure 2. It is found that kaolin agglomerates smaller than 1 m, as shown in the picture. According to Baozerara et al. [15], agglomeration is common in ultrafine particles due to the high cohesion between the main particles. This is due to its high surface-to-volume ratio and its small interparticle distance. Furthermore, at low and medium concentrations of solids the influence of hydrodynamic interactions dominates, whereas it is reported that at low concentrations of solids, the viscosity increases in proportion to the concentration of solids.

According to Frost [16], the viscosity of suspension depends on the volume fraction of particles. The relationship of volume fraction of particle and particle size was then giving a definition towards the interparticle spacing (IPS). IPS is the very important parameter in the estimation of viscosity suspension [17]. IPS can be defined as a distance from the surface of one particle to another. This can be explained in the equation 1:

$$\delta = d\sqrt{\frac{\pi}{6f}} \tag{1}$$

where δ is interparticle space, d is the particle size and *f* is volume fraction. The equation was manipulated for the volume fraction is less than 0.01 (1%). With regard to suspensions, the volume fraction and the maximum volume fraction can also be influential. It is possible to think of the maximum volume fraction (highest volume of particles that can be added to a fluid) as the amount of free space the particles have in which to move around. For instance, Rutgers observed that after a certain solids concentration, the viscosity of the slurry increase significantly with small increment of concentration [9]. In this work, the small concentration were analyzed via the effect from the kaolin particle size.

Figure 5 shows the SEM image of a ceramic membrane formed from kaolin with a particle size of 1 μm. As shown in the figure, the structure of the ceramic membrane is very dense. As described above, kaolin with a smaller particle size has a higher viscosity, thereby forming a dense structure. Maintaining a fixed mass of suspended particles while reducing the particle size of the solid phase will result in an increase in the number of particles in the system. Figure 3 shows the effect of system viscosity changes under different shear rates. The graph illustrates that the fluid is shear thinning (the viscosity decreases with increasing shear rate) and the viscosity increases with decreasing particle size. According to the website, as the volume fraction increases, the viscosity also increases. This is solved in the previous part of Equation 1. As the volume percentage of kaolin particles in the system increases, the particles become more compact, thereby preventing the kaolin particles in the membrane suspension from moving freely. This phenomenon is related to the observations shown in Figure 2. As shown in the picture, kaolin less than 1 m was found to be agglomerated. Yao et al., Believe that agglomeration into ultrafine particles is common due to the strong cohesion between large particles. Due to its high surface-to-volume ratio and small particle distance, it has a high surface-to-volume ratio. Furthermore, at low and medium concentrations of solids, hydrodynamic interactions dominate, but at low concentrations of solids, viscosity has been shown to increase in proportion to the concentration of solids [18].

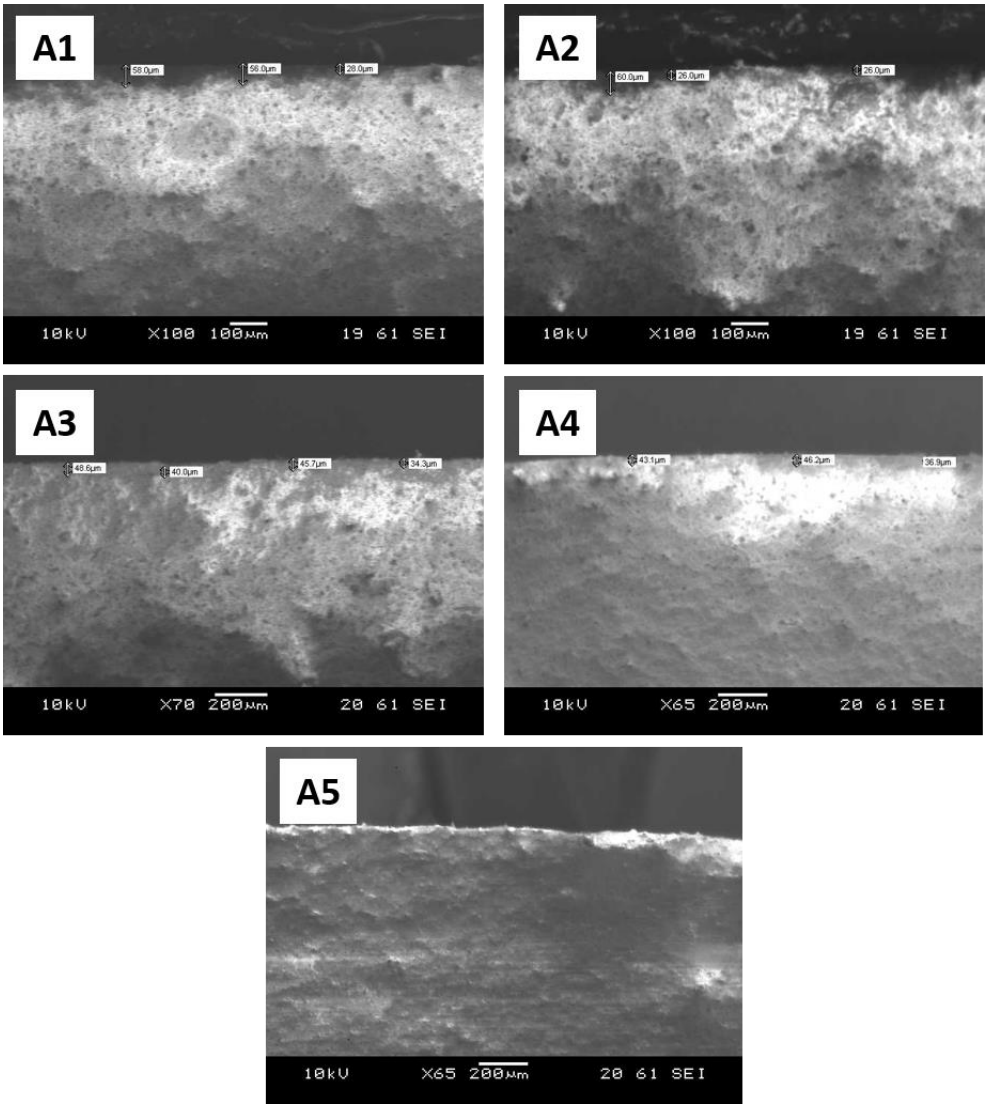

Fig. 5 - Formation of dense layer on the top of ceramic membrane fabricated via phase inversion at different composition; (A1) 40g, (A2) 50g, (A3) 60g, (A4) 70g and, (A5) 80g

4. Conclusion

As far as we are concerned, there are not many studies that have been done the analysis towards the characterization of the ceramic membrane suspension. As in polymeric dope preparation, there is not involve any particle suspension like ceramic membrane. Thus, the theory and the correlation in the system of ceramic membrane suspension was different with that in polymeric membrane dope. In summary, there are many theories involved in this study:

i) The particle size was affecting the characterization towards themselves (formation of agglomeration). This study indicates that the smallest particle sizes tend to agglomerate most.

ii) In metallurgy term, this study describes the relationship between the particle size, volume fraction and interparticle spacing towards the viscosity of the ceramic membrane suspension.

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