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Effects of Al_2O_3 Composition on the Physical Properties of Al_2O_3 Foam Prepared by Foam Replication Method

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***Abstract* — *Ceramic foams can be classified as lightweight materials with low densities, a unique combination of physical and mechanical properties, energy absorption, high porosity, and good thermal conductivity. Alumina Oxide (Al_2O_3) is one of the ceramic materials suitable for fabricating ceramic foam based on the characteristics mentioned above. In this study, the Al_2O_3 foam was produced from the foam replication method. The Polyurethane (PU) foam was used as the template dipped into the Al_2O_3 slurry, followed by drying and sintering to yield a replica of the original polymeric foam. This study's focus was to produce high pores on the Al_2O_3 foam with different compositions of Al_2O_3 powder weight percentage (wt%), which were 40wt%, 50wt%, 60wt%, and 70wt%. Next, the suitable solvent used for the Al_2O_3 slurry needs to be determined either Distilled water or Ethanol. Physical properties of the Al_2O_3 foam, such as density and porosity, were characterized using Archimedes Method with ASTM B962-15 standard. Shrinkage analysis was done to determine the foams' shrinkage before and after the sintering process. The microstructure analysis has been made to observe the types of pores and the structure inside of the Al_2O_3 foam produced. From the result, the shrinkage analysis shows that the higher the Al_2O_3 composition in the slurry, the higher the shrinkage percentage found in the Al_2O_3 foam. Al_2O_3 foam that used ethanol as solvent experienced higher shrinkage compared to distilled water. For the density and porosity of the Al_2O_3 foam, the higher the Al_2O_3 composition in the slurry, the lower the density of Al_2O_3 foam but produced higher porosity on Al_2O_3 foam for both solvents. The microstructure analysis shows that more open pores found in the Al_2O_3 foam produced as the composition of Al_2O_3 increased. Distilled water was the suitable solvent used in the preparation of Al_2O_3 slurry.**

Keywords — *Alumina (Al_2O_3) form, density and porosity, foam replication, microstructure, solvents*

I. INTRODUCTION

Recently, the design of biomimetic materials has rapidly developed the direction in material science, which impacts all engineering branches [1]. The developments of lightweight materials in the engineering world produce a space for the invention of porous material. Porous materials are the materials with pores, void, or cell intentionally integrated into the materials' structure. But not all the pores materials can be called porous material. The decay, holes, or apertures resulting from defects cannot be named porous material. All that defect can affect the material's lower performance [2] while the porous material that had been produced almost have the same performance with the solid material.

Porous materials are the materials with pores intentionally integrated into the structure of the materials. The porous materials have a unique combination not found in the dense metal, dense ceramics, and polymers that make these porous materials interesting to be studied [3]. Porous materials can be classified by the number of low porosity, middle porosity, and high porosity. The lower and middle porosity has close pores that behave as the impurity phase [2]. Besides, the application of these porous materials also rising in the catalyst, electrochemistry, membranes, gas separation, selective adsorption for environmental, energy uses, and lightweight structural materials[4].

There were several methods from the previous study on the fabrication of porous materials such as direct forming[5], sol gel[6], vacuum infiltration[7], space holder[8], thermo-foaming[9], and foam replication[10]. Ceramic foams are usually manufactured by impregnating open-cell polymer foams internally with ceramic slurry and then firing in a kiln, leaving behind only ceramic material[11]. The foam replication can produce the ceramic foam using the common type of ceramic such as Silicon Carbide (SiC), Alumina (Al_2O_3), Zirconia (ZrO_2), Titania (TiO_2), and Silica (SiO_2). Using this method can produce high porosity (70%-90%) of the ceramic[12]. Ceramic foams are porous, brittle materials with closed, fully open, or partially interconnected porosity [13].

The properties of the ceramic foam can be influenced by its parameters. The overall relative density includes the cell morphology, which is the cell with or without the cell walls, the pores morphology, which is isotropic on anisotropic, size or size distribution, the characteristic of the wall or struts between the pores either it were hollow or dense struts, and lastly the ceramic materials itself[5], [14]. The arrangement of the ceramic foam has properties that can be attractive as the catalyst support with 85 to 90% of high porosity[11], [15].

This study focused on the fabrication of Alumina Oxide (Al_2O_3) foam using the foam replication method. Al_2O_3 foam with porosity archived 90% with open and interconnected pores can be obtained using this replication method[16]. The open cell Al_2O_3 foam is mostly used for the filtration, thermal insulation, impact-absorbing structures, high specific strength materials, performs for metal-ceramic composites and biomechanical implants. Due to the good thermal conductivity, the Al_2O_3 can be used in high-efficiency combustion burners [13].

The foam replication method needs to use organic foam as the sacrificial template or mould. The organic foam that is mostly used in the foam replication method is Polyurethane[17]. This process's unique features are the pore structure is nearly the same as the organic foam precursors. The pore size depends on the pores in the organic foam and is related to the coated layer's thickness, drying of the slurry, and the shrinkage during sintering[18]. The pore size for ceramic is a little smaller than the organic foam.

The process includes in the foam replication method is dipping, squeezing, and drying. This process is generally involving the coating of open-cell polymeric foam with the ceramic slurry and then burning out of the polymeric foam by the sintering process. The dipping process is to coat the slurries on the PU foam. The squeezing process is to remove the overabundance slurry by the manual process, which is using hand. Moisture will be removed by the drying process. The purpose of the sintering process is to improve the bonding and mechanical properties of the Al_2O_3 foam[12]. After the sintering process, the resulting ceramic foam is a replica of the original polymer foam used by[19]. The microstructure of the Al_2O_3 is controlled by the processing route and the suitable parameter[20].

Since there is a lack of study about the effect of alumina composition and different types of solvent on the physical properties of Al_2O_3 foam prepared using the foam replication method, this study was conducted.

II. MATERIAL & METHOD

The experimental work was divided into three parts: mixing the raw materials and solvents, drying and sintering process, and characterization for the Al_2O_3 foam.

A. Mixing Process

The weight percentage of the Al_2O_3 powder used in this study were 40 wt%, 50 wt%, 60 wt%, and 70 wt%. The composition of the two binders is fixed at 2.5 wt% each, while the balance composition is solvent, either distilled water or ethanol.

Next, the mixture was mixed using the Roll Ball mill machine. The speed of the roll ball mill was 15 rpm and mixed within 1 hour. PU foam cut in the cuboid shape with 2cm length × 2cm width × 4cm height were dipped into the Al_2O_3 slurry. The foam was then squeezed until all the excessive slurry was removed. The dipped and squeezed process was repeated much time to make sure that the Al2O3 slurry fully adhered to the PU foam struts.

B. Drying and sintering process

After the mixing process, the PU foam coated with the Al_2O_3 slurry was dried using a drying oven. The temperature set for the drying process was 100^oC and conducted within 24 hours. Next, the Al_2O_3 foam was sintered in a box furnace. This process was conducted to allow for chemically bonding between the particles. At the first stage of the sintering process, the samples were heated to 350°C at 2°C/min heating rate. The temperature was kept constant at 350°C for 1 hour to allow for complete binders and PU foam removal before heating up to 1300°C. The second stage of sintering at 1300°C was conducted within 2 hours. Next, the cooling process was fixed at 2°C/min cooling rate to room temperature.

C. Characterization of the Al_2O_3 foam

The Al_2O_3 foam was characterized to determine the shrinkage percentage, density, and porosity and observed the microstructure. For shrinkage analysis, the dimension of samples before and after the sintering process was recorded. Then, the shrinkage percentage was calculated. At the same time, the density and porosity of Al_2O_3 foam were determined by using the Mettler Toledo XS64 Kit Archimedes test. Fig.1 shows the Mettler Toledo XS64 Kit Archimedes test. The porosity of the samples was measured according to the ASTM B962-15 standard.

The microstructure analysis was carried out to observe the differences between the microstructure and the pore size of the Al_2O_3 foam produced due to the different types of solvent and different Al_2O_3 powders composition used. The pore size of the Al_2O_3 foam also can be measured using Rincon Image Analysis Software. Microstructure observation was used SZH10 Stereomicroscope that could observe the open and close pores in the Al_2O_3 foam. Fig. 2 shows the SHZ10 Stereomicroscope that being used in this study.

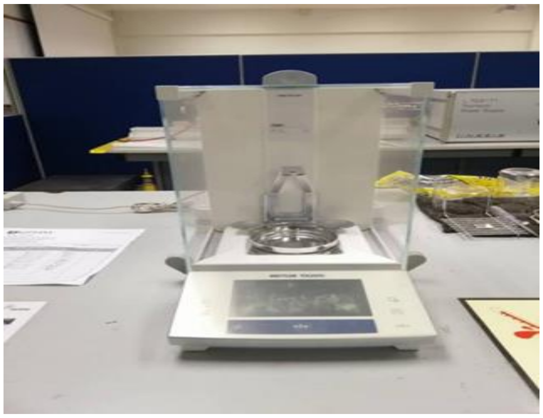

Fig.1 The Mettler Toledo XS64 Kit Archimedes test that used to determine the density and porosity Al_2O_3 foam in this study

Fig.2 The SHZ10 Stereomicroscope that was used to observe the microstructure and the types of pores in Al_2O_3 foam.

III. RESULT and DISCUSSION

This study's results were discussed and analyzed based on the shrinkage, density and porosity, and microstructure of the Al_2O_3 foam due to different Al_2O_3 compositions and different types of solvent used.

A. Shrinkage analysis

The Al2O3 foam sample's average volume is shown in Table 1 for the Al_2O_3 foam produced using distilled water as the solvent, while Table 2 for Al_2O_3 foam is produced using ethanol solvent.

Based on the result obtained, the lowest shrinkage volume was obtained from the Al_2O_3 foam with 40 wt% of Al_2O_3 for both solvents. During the mixing process, the amount of liquid was 55%, higher than the Al_2O_3 and binders composition, which were only 45%. Therefore, the Al_2O_3 and binders powders were easily dissolved in the liquid and produce a slurry with low consistency. This slurry can easily reach the innermost part of the PU foam structure, and the Al_2O_3 particles have adhered to almost all the PU foam struts surface. Hence, this avoids the Al_2O_3 foam from collapsing during the removal of the binder and PU foam during the sintering process. However, as the Al_2O_3 composition increased, the slurry consistency also increases. Although the amount of Al_2O_3 particle is high in the slurry, the possibility of the slurry to reach into the innermost part of the PU foam template become more difficult. Hence the only a limited amount of Al_2O_3 particles will successfully adhere to the PU foam struts surface. This uncoated strut will collapse during the sintering process and result in the sample's high shrinkage percentage.

Table 1 The average volume of Al_2O_3 foam before and after sintering using distilled water

Al_2O_3 composition (wt %)	The average volume of Al_2O_3 before sintering (cm^3)	The average volume of Al_2O_3 after sintering (cm^3)	Average shrinkage volume (%)
40	15.8612	15.1982	0.6630
50	15.7378	14.8856	0.8522
60	15.7725	14.8544	0.9181
70	15.6632	14.5982	1.065

Table 2 The average volume of Al_2O_3 foam before and after sintering using ethanol

Al_2O_3 composition (wt %)	The average volume of Al_2O_3 before sintering (cm^3)	The average volume of Al_2O_3 after sintering (cm^3)	Average shrinkage volume (%)
40	15.4759	15.2732	0.2027
50	15.3446	14.9843	0.3603
60	15.7845	14.7553	0.8732
70	15.7425	14.8693	1.0292

B. Density and porosity analysis

The density and porosity tests were conducted after the sintering process. The sample produced after being sintered was Al_2O_3 foam with high porosity content due to the binders and PU foam removal. Therefore, a density and porosity test was conducted to determine the percentage of the porosity content. The effect of using different Al_2O_3 powders percentages and different types of solvent on the density and porosity also could be determined. Table 3 shows the value of the density and porosity of the Al_2O_3 foam from distilled water solvent, while Table 4 shows the density and porosity of the Al_2O_3 foam made from ethanol solvent.

Table 3 shows the density and porosity test results for the Al_2O_3 foam that used distilled water as a solvent for the composition 40 wt%, 50 wt%, 60 wt%, and 70 wt% in the

Al_2O_3 foam. As shown in the table, Al_2O_3 foam with 70 wt% Al_2O_3 compositions had the lowest density at 3.4853g/cm^3 but had the highest porosity percentage of 76.5432%. Table 4 shows the same pattern of result where the Al_2O_3 foam with 70 wt% of Al_2O_3 composition had the lowest density, which is 3.9456g/cm^3 but had the highest porosity percentage, 72.8335%. This result shows that the higher the Al_2O_3 composition, the higher the porosity of the Al_2O_3 foam produced. The other comparison of the density and porosity shows in Fig.3 and Fig.4.This result is in agreeing with the shrinkage result. The sample with high Al_2O_3 composition experienced the highest shrinkage due to the sample collapsion and indirectly produced more pores inside the sample and reduced the sample's density.

Table 3 The density and porosity result for Al_2O_3 foam using distilled water solvent

Composition of Al_2O_3 (wt%)	Density (g/cm^3)	Porosity (%)
40	4.3931	62.5834
50	3.9821	68.3698
60	3.6448	71.7626
70	3.4853	76.5432

Table 4 The density and porosity result of the Al_2O_3 foam using ethanol solvent

Composition of Al_2O_3 (wt%)	Density (g/cm^3)	Porosity (%)
40	4.5632	60.2640
50	4.3287	64.7305
60	4.2679	68.5632
70	3.9456	72.8335

Fig. 3 shows the density Al_2O_3 foam decrease as the composition of the Al_2O_3 increase. Both solvents show a similar result. However, the difference was that the density of the Al2O3 foam produced using ethanol as a solvent was slightly higher than the Al_2O_3 foam produced using distilled water as solvents. Fig. 4 shows the porosity of the Al_2O_3 foam for both solvents. The porosity of the Al_2O_3 foam produced using distilled water as a solvent is slightly higher than the Al_2O_3 foam produced using ethanol. The ethanol characteristics that are more volatile than distilled water made a difference in the absorption of the foam's slurry during the mixing process. This is based on the observation during the mixing process, where the amount of powder dissolved in the ethanol slurry was less compared to the distilled water.

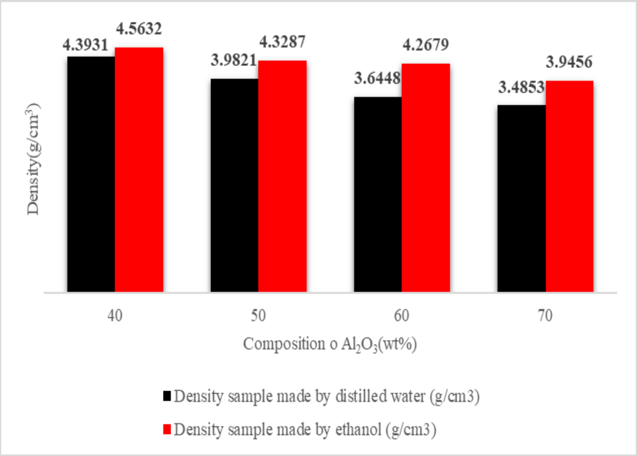

Fig. 3 Density of Al_2O_3 foam from different solvents against the composition of Al_2O_3(wt%)

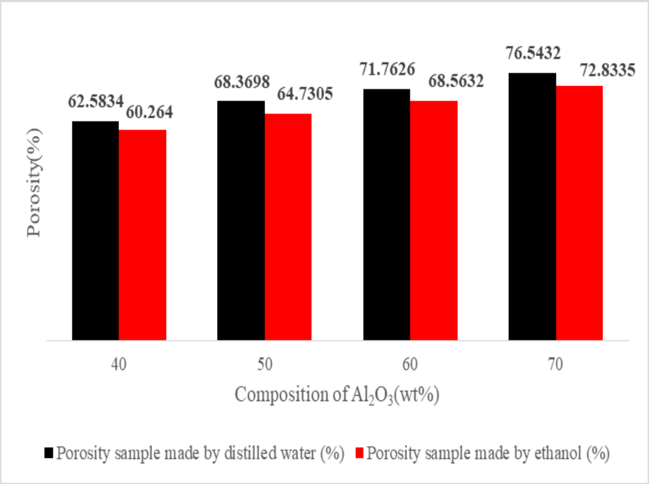

Fig. 4 Porosity of Al_2O_3 foam from different solvents against the composition of Al_2O_3(wt%)

C. Microstructure Analysis

In this analysis, using different Al2O3 compositions, which were 40 wt%, 50 wt%, 60 wt%, and 70 wt%, and different types of solvents that were distilled water and ethanol on the Al_2O_3 foam microstructure were observed.

Fig. 5 shows the microstructure of Al_2O_3 foam at 20x magnification using the SZH10 Stereomicroscope. It can be observed that the distribution of the pores was not uniform. This could be due to the Al_2O_3 slurry was not completely covering all the PU foam during the dipping and squeezing process. Fig. 6 and Fig. 7 show the microstructure of the Al_2O_3 foam produced with 40 wt% of Al_2O_3 composition, while Fig. 8 and Fig. 9 shows the microstructure of Al_2O_3 foam produced with 70 wt% of Al_2O_3 composition with distilled water and ethanol solvent respectively at 20x magnifications.

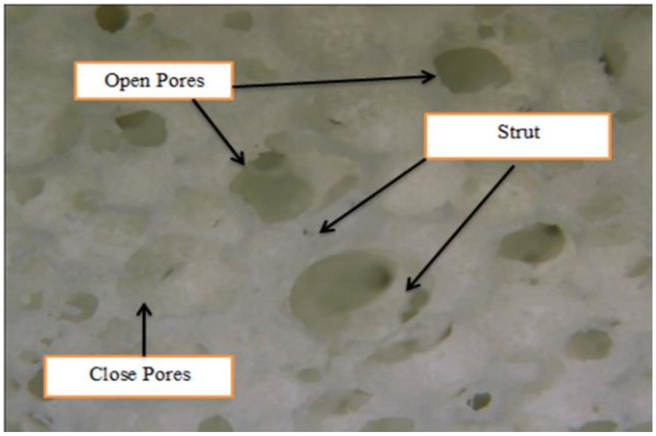

Fig. 5 Al_2O_3 foam microstructure with the type of pores produced

From Fig. 6 and Fig. 7, more closed pores being produced with 40 wt% compositions on the Al_2O_3 foam for both solvents. Next, it shows that the shape of the pores in the Al_2O_3 foam produced using distilled water as a solvent was more rounded than the ethanol. There are also more open pores found in the Al_2O_3 foam produced using distilled water as a solvent compared to ethanol.

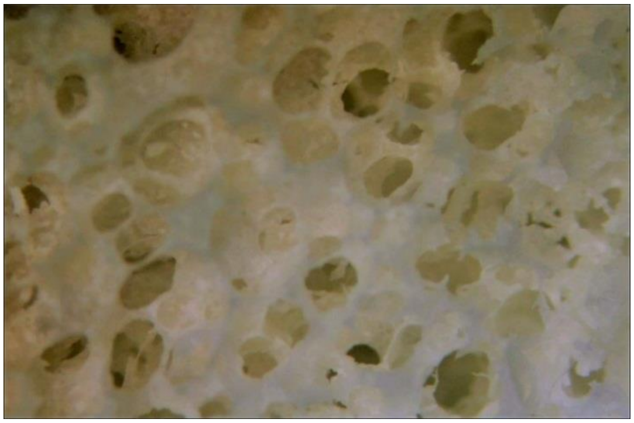

Fig. 6 The microstructure for the Al_2O_3 foam using with 40 wt% of Al_2O_3 composition using distilled water as the solvent

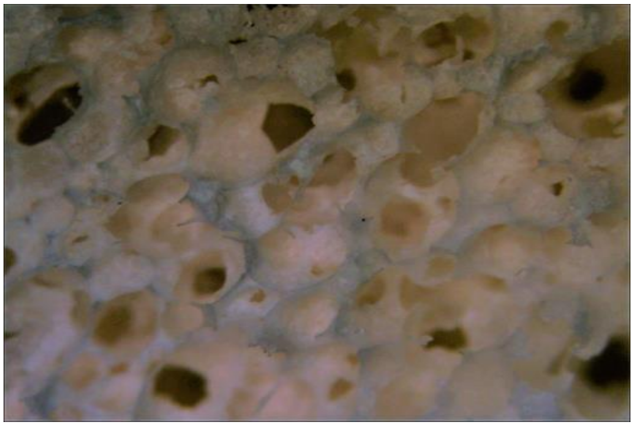

Fig. 7 The microstructure for the Al_2O_3 foam with 40 wt% of Al_2O_3 composition using ethanol as solvent

From Fig. 8 and Fig. 9 observed that more open pore for the Al_2O_3 foam produced at 70 wt% compositions of Al2O3 for both solvents. The open pores were observed on the outer and inner surfaces of the Al_2O_3. The open pores at the foam's inner layer mean that the pore was interconnected while the strut connected all the pores from inside or outside the layer of the Al_2O_3 foam. Next, the Al_2O_3 foam used distilled water as solvent produced a better pore shape compared to the Al_2O_3 foam using ethanol as solvent.

The lowest pore size in this study was from the 40 wt% compositions of Al_2O_3 using ethanol as the solvent, which was 595.8820 µm, while the highest pore size produced in this study occur at the 70 wt% compositions of Al_2O_3 using distilled water as the solvent with 1087.3280 µm. More pores that are open produced as the Al_2O_3 composition increase for both type of solvent. The sizes of the open pores also increased as the composition of Al_2O_3 powder increased.

Fig. 8 The microstructure for the Al_2O_3 foam using with 70 wt% of Al_2O_3 composition using distilled water as the solvent

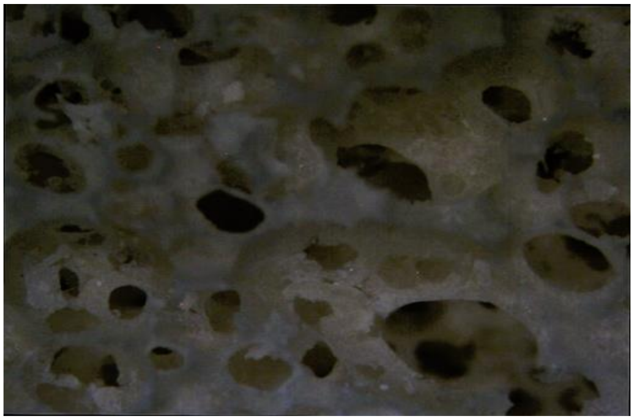

Fig. 9 The microstructure for the Al_2O_3 foam with 70 wt% of Al_2O_3 composition using ethanol as solvent

Fig. 10 shows the average pore size between the two types of solvent against the composition Al_2O_3 graph. From this graph, it shows that the distilled water had larger pores size compared to the ethanol. The significant differences in pores size only occurred for the sample with 40 wt% Al2O3 compositions produced using distilled water as a solvent, 76.6340 µm larger than ethanol. The other composition also shows quite significant differences in pore size between these two types of solvents. Only at the 50 wt% of Al_2O_3 composition shows a slight difference which is lower than 50 µm.

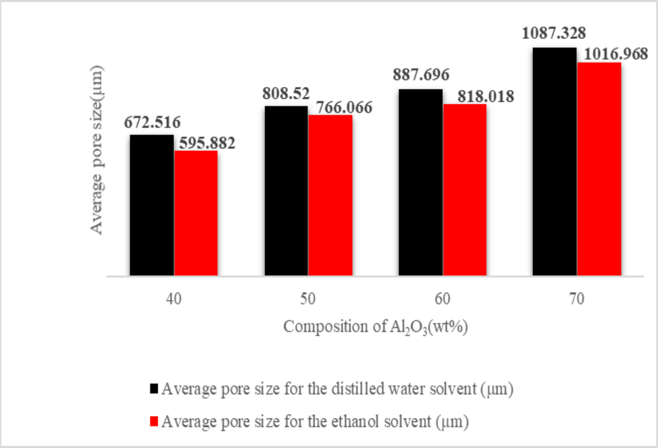

Fig. 10 The graph comparison of Al_2O_3 foam pore size with different types of solvents

IV. CONCLUSIONS

The Al_2O_3 foam had been produced by foam replication method using two different solvents: distilled water and ethanol and different compositions of Al_2O_3. From this study, it can be concluded that for the shrinkage, 40 wt% of Al_2O_3 composition is the best composition. The distilled water as a solvent produces lower shrinkage compared to ethanol. The distilled water was the best solvent to produce Al_2O_3 foam compared to ethanol.

The density and porosity test shows that the higher the Al_2O_3 composition, the higher porosity obtained and the lower density produced. In this study, Al_2O_3 foam with 70 wt% of Al_2O_3 composition produced by both types of solvents has higher porosity but a lower density.

The pore distribution found in the Al_2O_3 foam was not uniform for all compositions and types of solvents. As the Al_2O_3 composition increases, the struts' size also becomes thicker for both types of solvents. Next, the increase of the Al_2O_3 composition in Al_2O_3 foam produced a higher average pores size. More open pores were observed when higher compositions of Al_2O_3 were used. In this study, the 70 wt% of Al_2O_3 composition used distilled water as the solvent produced better microstructure for the Al_2O_3 foam. This composition produces a higher average pore size and produced more open pores for the foam.

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