RESEARCH PROGRESS IN MECHANICAL AND MANUFACTURING ENGINEERING
e-ISSN: 2773-4765

RPMME

Vol. 5 No. 1 (2024) 518-524
https://publisher.uthm.edu.my/periodicals/index.php/rpmme

# The Effect of Space Holder Types and Composition on the Properties of Zirconia Foam

**Muhammad Hadi Asmurad[1], Fazimah Mat Noor[1]*, Azzura Ismail[1]**

[1] *Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, Batu Pahat, 86400, MALAYSIA*

*Corresponding Author: fazimah@uthm.edu.my
DOI: https://doi.org/10.30880/rpmme.2024.05.01.059

**Article Info**

Received: 31 January 2024
Accepted: 19 June 2024
Available online: 15 September 2024

**Keywords**

Sintering, Milling, Porosity, Density and Compaction method.

**Abstract**

Ceramic foams can be classified as porous and brittle materials with pore formation inside. These materials have their own distinct physical properties that have been used for a variety of applications, including thermal insulation, filtration, and as a substrate for catalytic converters, which are frequently used to lower vehicle emissions. This project used the space holder method to create Zirconia foam, which involves adding and removing a space holder material from the mixture. Sodium chloride, sago starch, and tapioca starch were the three types of materials employed as the space holder material. Their weight percent compositions were 10%, 20%, and 30%, respectively. Distilled water was used to dissolve and remove the space holder components. While Polyethylene Glycol (PEG) and carboxymethyl cellulose (CMC) were utilised as binders which act as glue to boost the powder's green strength during the compaction process. All the mixed ingredients were compacted into a solid cylinder sample after being ball-milled. Following that, the samples were sintered in a furnace at 1400°C. Finally, the samples were tested and analyzed by doing the microstructural analysis, density and porosity testing, and shrinkage analysis. Based on the findings, the zirconia foam has been effectively created using the space holder method, and the differences in the composition of the space holder material do affect the qualities of the final product. As the tapioca starch was used as a space holder, the pores were closed. However, after the sodium chloride was used as a space holder, the pores were open and clear. Unfortunately, the sample with the sago starch as a space holder material was totally collapsed after sintering. It can be concluded that sodium chloride is the ideal material to utilise as a space holder since it can create samples with the least amount of shrinkage, the highest density, and the smallest amount of porosity when the optimal composition is used, which is 20% than tapioca starch and other composition.

## 1. Introduction

A space holder is a method for creating porous materials with closed or open porosity [1]. The strength, density, and porosity of silica foam will be affected by the amount and type of space holders used. Zirconia powder was utilized in this project. Space holder materials, binder, and zirconia powder were used to create the foam. The powder metallurgy branch includes the space holder method. Different weight percentages of space holder material were used to acquire the appropriate porosity, density, and strength. The mixing of space holder

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material, binder, and zirconia powder, compaction of the mixed substances, removal process of the space holder material, and sintering process at high temperature below the zirconia foam melting point are the four key aspects of this approach.

## 2. Materials and Methods

Zirconia powder, space holder materials, Carboxymethyl Cellulose (CMC) and Polyethylene Glycol (PEG) are the materials used to produce Zirconia foam. The composition of the space holder materials was varied from 10 wt.% to 30 wt.%. The binder composition was fixed at 5 wt.%, while the balance was Zirconia powders. All these raw materials were mixed using ball-mixing method. The mixture was then compacted into a cylindrical shape using Carver hydraulic press. After that, the compacted samples were sintered in a box furnace at 1500°C for 2 hours. Both heating and cooling rates were set at 3°C/min. Finally, the samples were tested and characterized. The shrinkage percentage was determined by measuring the sample dimension before and after sintering process. While the density and porosity test were conducted using Mettler Toledo XS64 apparatus with referring to ASTM C 271-94 standard. The Zirconia foam microstructure was observed using Hitachi U1510 Scanning Electron Microscope at different magnification.

## 3. Results and Discussion

### 3.1 Shrinkage Analysis

Shrinkage percentage was determined by measuring the samples dimension using a digital caliper with precision of 0.01 mm. Figure 1 shows the zirconia foam samples before and after the sintering process. Some of the samples totally collapsed after sintering. The collapsed samples were identified as the zirconia foam with sago starch as a space holder material.

(a) (b)

**Fig. 1** *Zirconia foam samples (a) before and (b) after Sintering Process*

The shrinkage percentage of the zirconia foam with different type and composition of space holder material is shown in Figure 2. The shrinkages range from 4.25% to 6.56% when employing tapioca starch as a space holder material, and as the composition is increased, the shrinkage percentage also increase. The shrinkages for Zirconia foam with sodium chloride as a space holder are quite minimal, ranging from 0.18% to 1.69%. The percentage of shrinkage decreases as sodium chloride's composition increases. As the tapioca starch composition increased to 30 wt.%, the shrinkage is the greatest. This result is consistent with a previous study [2] that found that tapioca starch can contract when sintered. The average shrinkage increases as the composition utilised increases. The least shrinkage occurred when sodium chloride was used as a space holder. The shrinkage take place after the sintering process because during the sintering process, the space holder in the sample have been burn out and leave the pore [3]. The inter-particle neck growth and mass diffusion that cause the disappearance of micro-pores and densification cause the powder compact to shrink during sintering. Sintered scaffolds shrinking is decreased by compaction's increased green density [4]. Since tapioca starch particles lower green density, shrinkage rises when space holder content is increased. But it is opposite to sodium chloride which got high density because the shrinkage is decrease when the space holder content is increase [3].

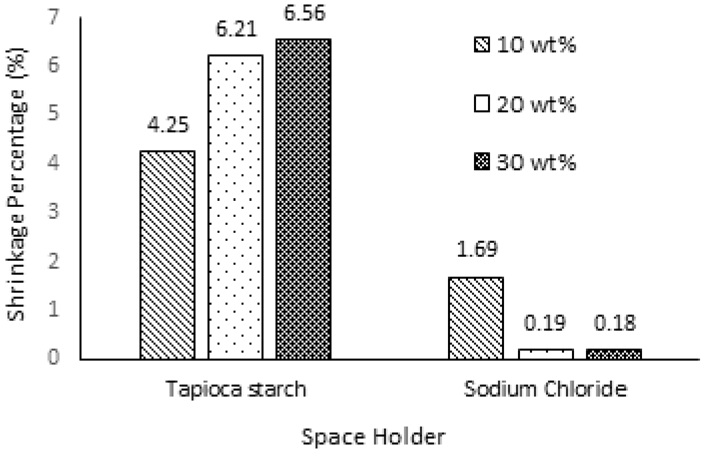
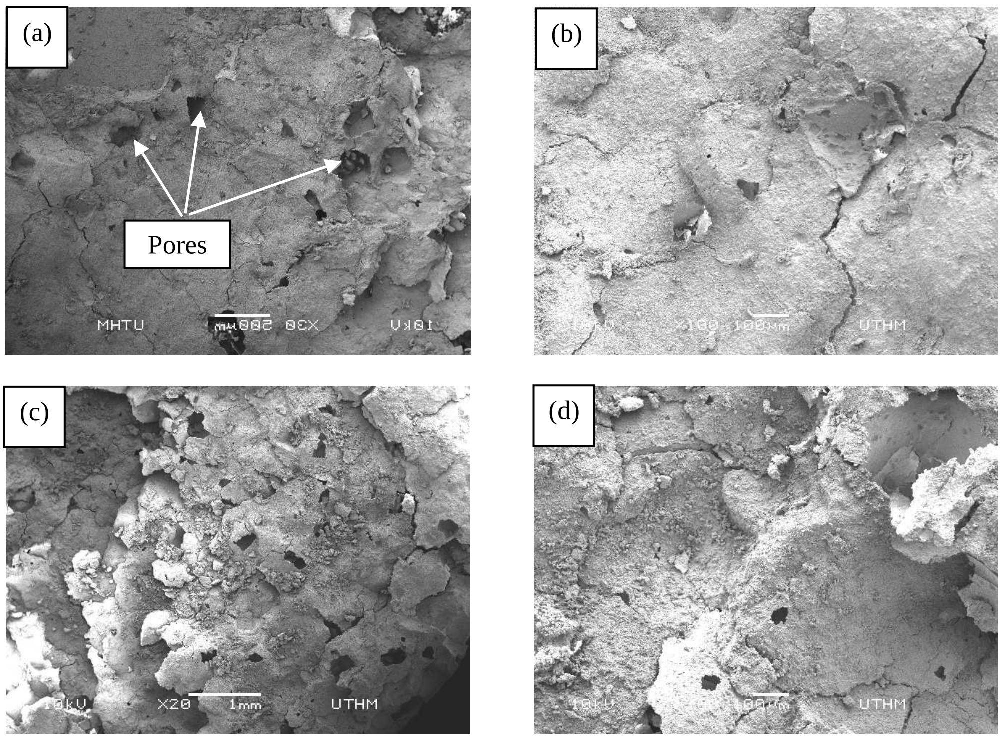



**Fig. 2** *The shrinkage percentage of Zirconia foam with different type and composition of space holder material*

## 3.2 Microstructural Analysis

Scanning electron microscope was used to examine the microstructure of materials. Figure 3.1 show the SEM images for Zirconia foam with 10 wt.%, 20 wt.% and 30 wt.% Sodium Chloride as space holder material at 30x, and 100x magnification, while Figure 4 shows the SEM images for Zirconia foam with 10 wt.%, 20 wt.% and 30 wt.% Tapioca Starch as space holder material at 30x, and 100x magnification. It can be seen clearly that all samples did not have an even pore distribution, shape and size. All samples consisted of closed pores. It is because the amount of space holder that has been eliminated in sintering process cannot been controlled to get the uniform distribution [5]. Therefore, using sodium chloride as a spacer holder is the most obvious pore formation, which is followed by tapioca starch. The composition of space-holder materials has an impact on pore development as well. With a relatively narrow size dispersion, the average pore diameter is 150 µm. This is due to the nature of the foaming process as stated in previous research [6].



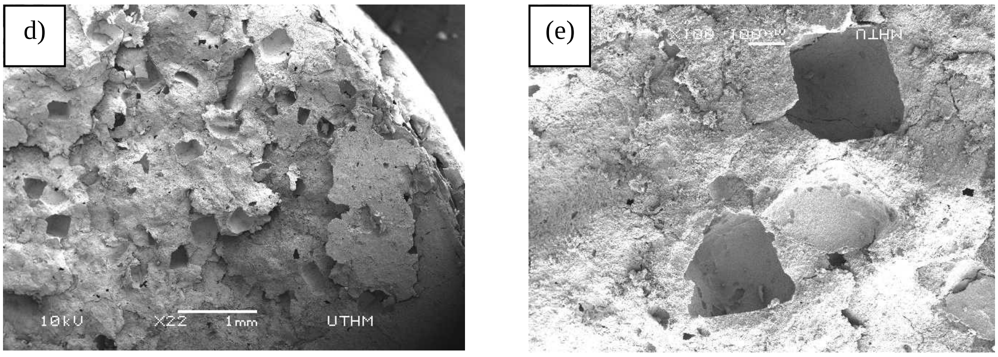



**Fig. 3** *SEM images for Zirconia foam with 10 wt.% of Sodium Chloride (a) and (b) at 30x and 100x magnification respectively, 20 wt.% of Sodium Chloride (c) and (d) at 30x and 100x magnification respectively, and 30 wt.% Sodium Chloride (e) and (f) at 30x and 100x magnification respectively*

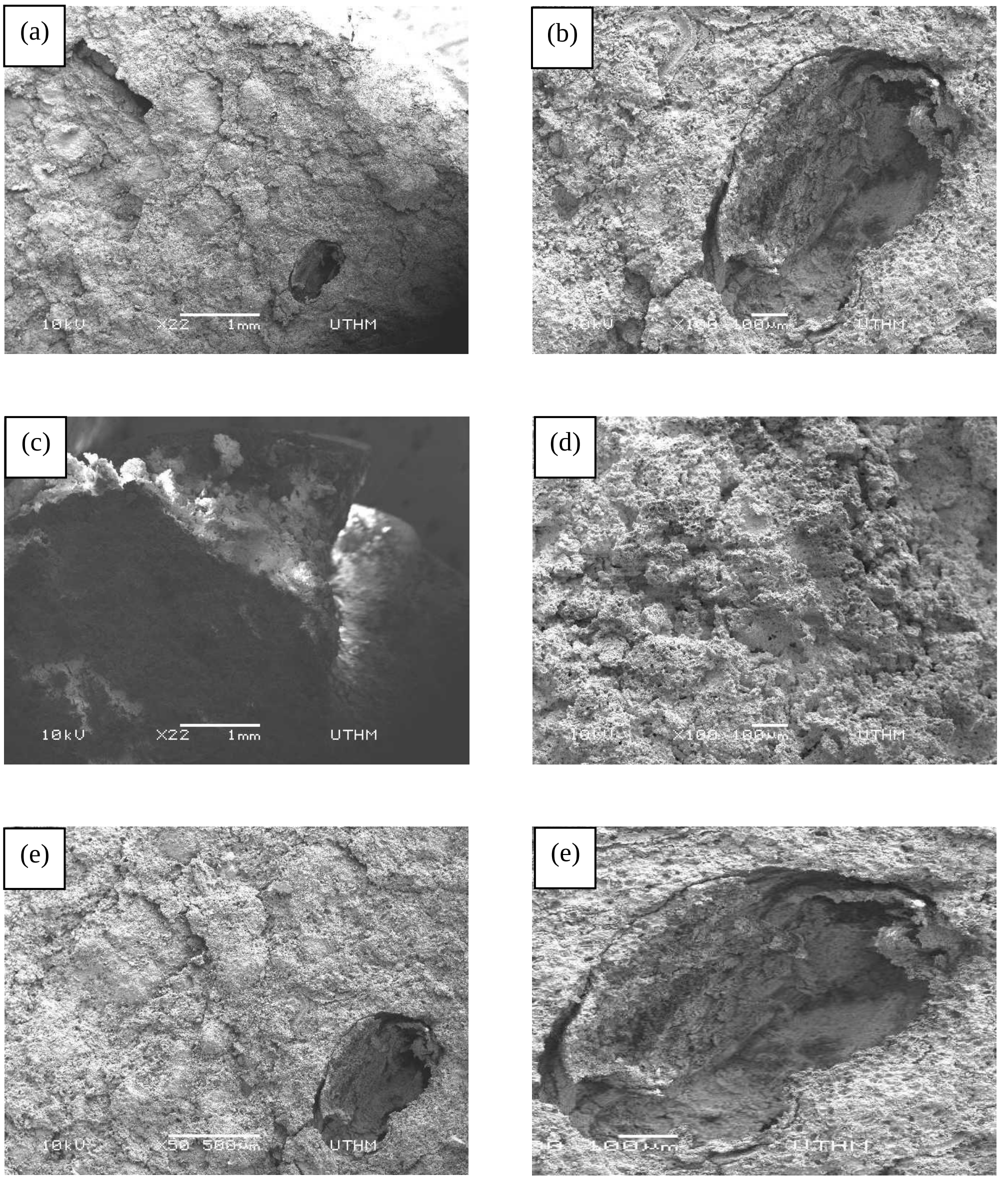



**Fig. 4** *SEM images for Zirconia foam with 10 wt.% of Tapioca Starch (a) and (b) at 30x and 100x magnification respectively, 20 wt.% of Tapioca Starch (c) and (d) at 30x and 100x magnification respectively, and 30 wt.% Tapioca Starch (e) and (f) at 30x and 100x magnification respectively*

### 3.3 Density and Porosity

The density and porosity of the Zirconia foam with Sodium Chloride and Tapioca Starch as the space holder materials are shown in Figure 5 and Figure 6, respectively. Density is more likely to be lower in porous materials than in solid materials. The shrinkage that takes place during the sintering process is strongly related to the decreasing of porosity percentage of the sintered samples' [6]. Utilizing sodium chloride as a space-holding component results in the maximum density, which is 1.8556 g/cm3 at a 10% composition. Based on all results, tapioca starch, which has a 10% content, has the highest average porosity. The premixed powder has agglomeration because of the lack of a sieve procedure following the ball milling operation [7]. When this occurs, the density will not be dispersed evenly, and the size of its pores will vary. The substance sodium chloride has the lowest porosity ever measured, and only a small amount may be attributed to its composition. All samples exhibit a trend of decreasing density as the composition of the space holder is raised. According to a previous study [8], increasing the zirconia percentage can improve foam density, showing the link between higher space holder material composition and lower sample density.

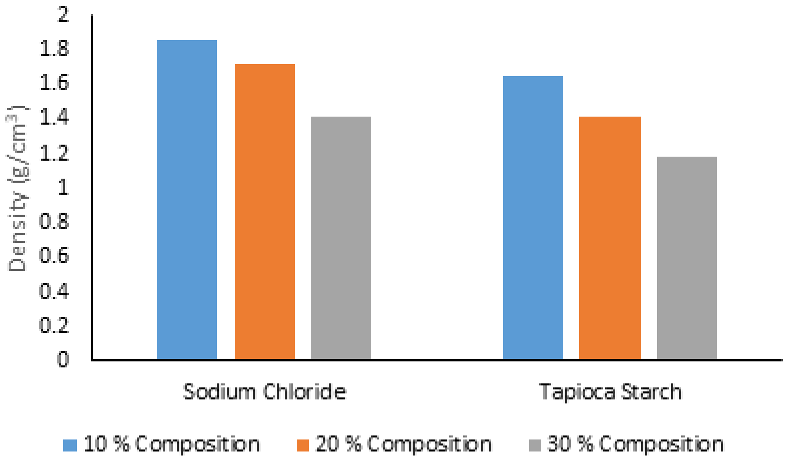



**Fig. 5** *The density of Zirconia Foam with different type and composition of space holder materials*

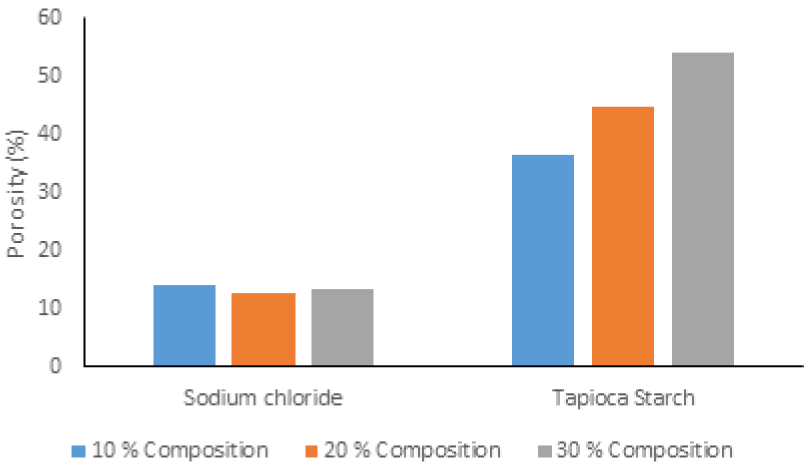



**Fig. 6** *The porosity percentage of Zirconia Foam with different type and composition of space holder materials.*

Based on the overall results, the shrinkage of the Zirconia foam with all the space holder materials is in the range of 0.1% to 5%. The space holder material's composition, which can be changed, has an impact on shrinkage. When tapioca starch with a 30% composition is utilised, the shrinkage is the greatest. The average shrinkage increases as the composition utilised increases. The least shrinking happens when sodium chloride is used as a space holder. The shrinkage happens after the sintering process because when the sintering process occur, the space holder in the specimen have been burn out and leave the pore [9]. The inter-particle neck growth and mass diffusion that cause the disappearance of micro-pores and densification cause the powder compact to shrink during sintering. Sintered scaffolds shrinking is decreased by compaction's increased green density [10]. Since tapioca starch particles lower green density, shrinkage rises when space holder content is increased. But it is opposite to sodium chloride which got high density because the shrinkage is decrease when the space holder content is increase [9].

All the samples did not have an even pore distribution and did not meet the expected outcome which is got intersection pores and closed pores [11] according to the data gathered. It is because the amount of space holder that has been eliminated in sintering process cannot been controlled to get the uniform distribution [8]. Therefore, using sodium chloride as a spacer holder is the most obvious pore formation, which is followed by tapioca starch. The composition of space-holder materials has an impact on pore development as well. With a relatively narrow size dispersion, the average pore diameter is 150 µm. This is due to the nature of the foaming process as stated in previous research [12].

Based on all the test results, tapioca starch, which has a 10% content, has the highest average porosity. The premixed powder has agglomeration because of the lack of a sieve procedure following the ball milling operation [13]. When this occurs, the density will not be dispersed evenly, and the size of its pores will vary. The substance sodium chloride has the lowest porosity ever measured, and only a small amount may be attributed to its composition. All samples exhibit a trend of decreasing density as the composition of the space holder is raised. According to a previous study [14], increasing the zirconia percentage can improve foam density, showing the link between higher space holder material composition and lower sample density.

## 4. Conclusion

Physical testing has established that ceramic foam has a density that ranges from 1.18 $g/cm^3$ to 1.86 $g/cm^3$, and that its porosity ranges from 12.56% to 53.95% on average. It has been found that density decreases with increasing space holder material composition. This happened because the zirconia powder is denser than those space-holder materials. The largest percentage of porosity can be achieved by utilizing tapioca starch, while sodium chloride can be utilized for samples with larger densities. It varies from 0.18% to 6.55%, and sodium chloride and tapioca starch are the best space-holder materials that shrink the least.

Scanning Electron Microscopy (SEM) was used for morphological examination to study the creation of pores during the sintering process. Based on the findings, it can be said that pore creation is not dispersed evenly. The results are consistent across all compositions tested. This is due to the absence of a sieving process after the ball milling process, which is a step to separate the powder particles so that it is smaller and does not clump. As a result, the space holder particles varied in size as they agglomerated, producing larger pores than other particles.

As for the conclusion, it can be said that sodium chloride is the best choice to use as a space holder since it can produce samples with the least shrinkage, maximum density, and smallest percentage of porosity when using the composition that is most appropriate, which is 20% rather than tapioca starch.

## Acknowledgement

The authors would like to thank the Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia for its support.

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