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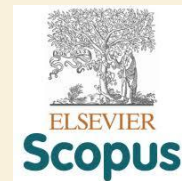
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ISSN: 0974-5823

Subject area: Engineering: Mechanical Engineering

Source type: Journal

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PUBLICATIONS ETHICS AND MALPRACTICE STATEMENTS

## *International Journal of Mechanical Engineering*

ISSN: 0974-5823

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DOI No: DOI:10.56452/00000

 This journal also publishes Open Access articles

### Journal Metrics

Acceptance rate: 30%

Submission to final acceptance decision: 7 - 14 days

Acceptance to publication: 14 - 28 days

Cite Score : 5.2

**Impact Factor : 2.1**

**Subject Area: Mechanical Engineering and Related Area**

Published By: *Kalahari Journals*

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International journal of Mechanical engineering is Scopus indexed Journal. This journal publishes scholarly articles. This journal is Scopus indexed from 2019 and also UGC-CARE List Group II Listed (Journals indexed in globally recognised databases)

**Note: IJME currently inviting scholars to submit a special issue proposal. The purpose of the special issue is to provide collection of articles on a specific topic of Mechanical Engineering Convergence that the journal has not covered substantially and has the potential to be of high interest to the readers.**

#### Aims and Scope

The "*International Journal of Mechanical Engineering*" is an peer reviewed journal. The goal of this journal is to provide a platform for academicians, researchers and practitioners all over the world to promote, share, and discuss various new issues and developments in all areas of Mechanical Engineering. Manuscripts are subject to a rigorous and fair peer-review process. Accepted papers will appear online within 3-4 weeks after their submission. The journal publishes papers including but not limited to the following topics:

Aerodynamics, Fluid Dynamics, Compressible Flows, Computational Mechanics, Biomechanics, Automotive Engineering, Heat and Mass Transfer, Nanomaterial Engineering, Plasticity Mechanics, Fracture, Geomechanics, Multibody Dynamics, Nonlinear Dynamics, Structural Dynamics, Vibrations, Acoustics, Noise Control, Material Engineering, Transport Phenomena, Manufacturing Process, Mechatronics, Transport phenomena (heat, mass transfer and fluid flows), Mathematical modeling in Mechanical engineering, Mechanical engineering education, Plasticity, Fracture and Damage Mechanics, Mechanics of Nanomaterials, Fluid-Structure Interaction, Damage Identification and Non Destructive Evaluation (NDE), Computational and Experimental Mechanics, Geomechanics and Mechanics of Granular Materials, Flows in Porous Media, Impact and Multibody, Dynamics, Nonlinear Dynamics, Structural Dynamics and Control, Dynamic Instability and Buckling, Vibrations, Acoustics and Noise Control, Manufacturing Processes, Advanced Materials and Smart Structures, Micro ElectroMechanical Systems (MEMS), Mechatronics, Transport Phenomena in Micro/Nanoscale, Aerodynamics and Aeroelasticity, Computational Fluid Dynamics (CFD), Turbulence and Multiphase Flows, Biomechanics and Biomaterials, Surface Engineering and Contact Mechanics, Heat and Mass Transfer.

**Focus of the journal:** Mechanical Engineering; Mechanics

**Submission:** Authors are requested to submit their papers [editorkalaharijournal@gmail.com](mailto:editorkalaharijournal@gmail.com)

**Indexing and Abstracting :** The journal is index in *Scopus, Worldcat, Google Scholar, Research Gate...*

**Frequency:** IJME published two issues per year from 2016-2020; three issues in the year 2021 and 2022 onwards IJME aims to publish 12 issues per year.

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*Vol. 6 No. 3 (December, 2021)*

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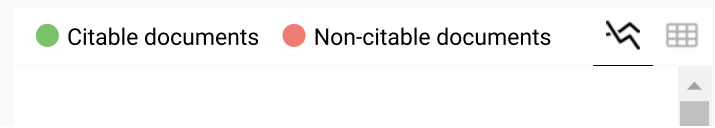
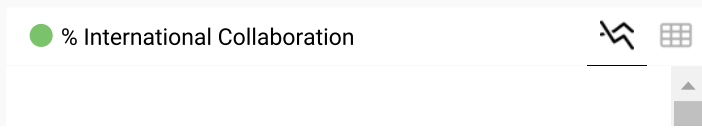
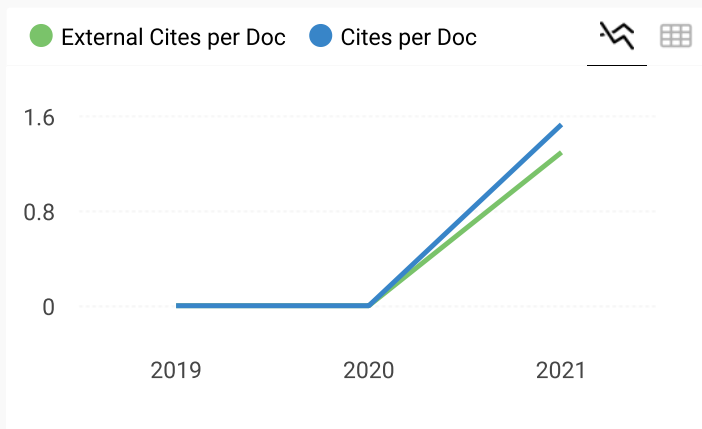
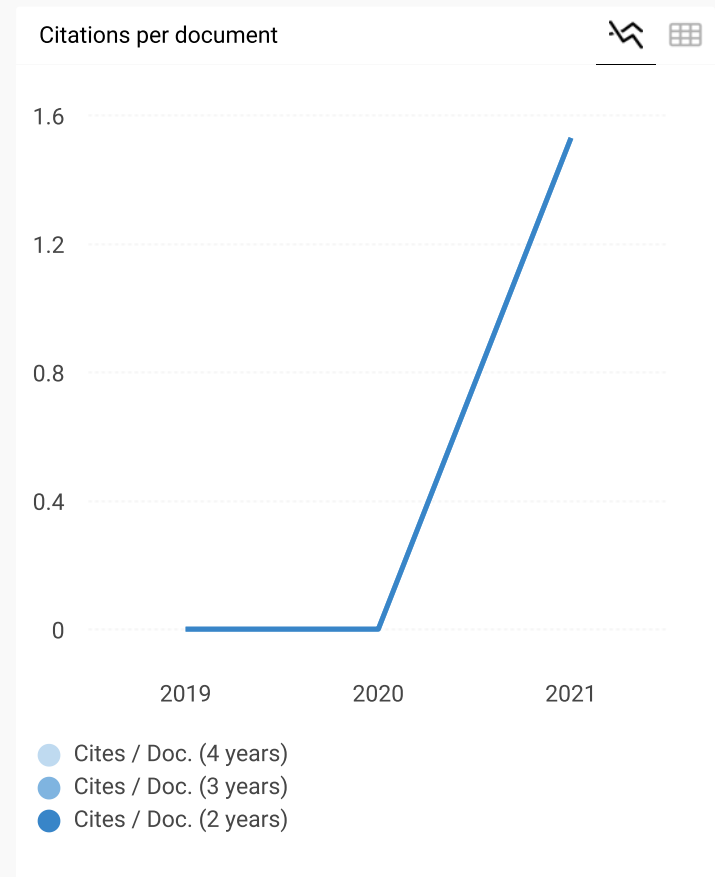
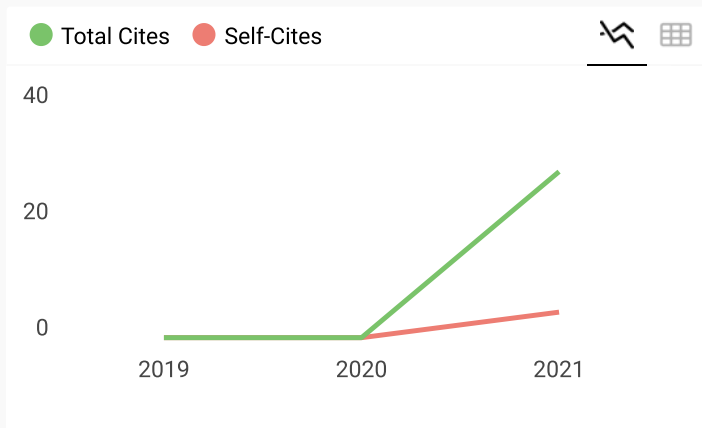
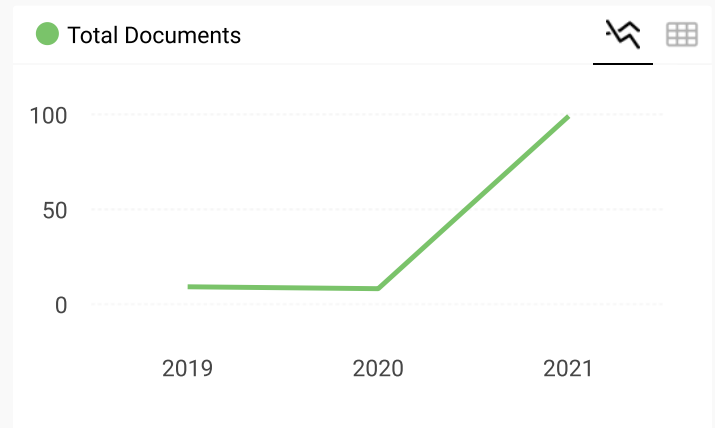
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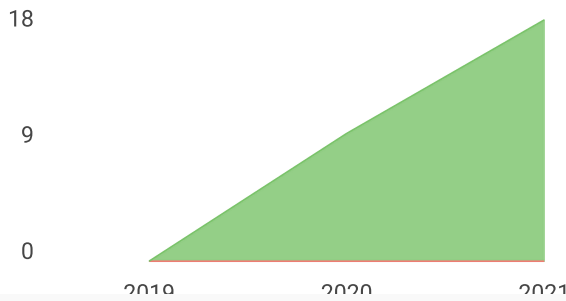
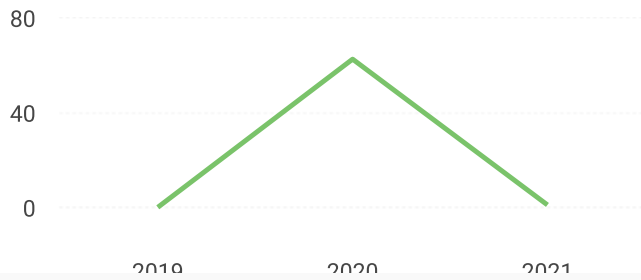
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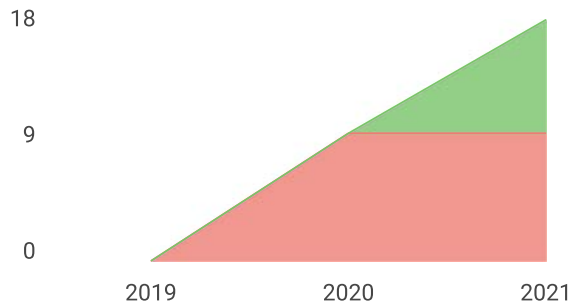
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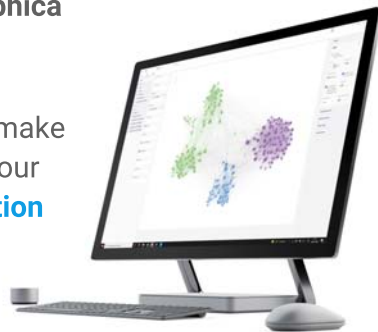
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# The Effect of Calcination Temperature on Ionic Conductivity of Calcia Stabilized Zircon Made Using Sol Gel Method

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**Abstract** - This research aims to study the effect of calcination temperature on ionic conductivity of CSZ pellets made through a sol gel method in three calcination temperatures of 500 °C, 600 °C and 700 °C. In this study, CSZ pellets were made from extraction of Zirconium Silicate (ZrSiO<sub>4</sub>) by caustic fusion and sol gel synthesis method. The characterization of CSZ pellets was performed using X-ray diffraction (XRD), Scanning Electron Microscope equipped with energy dispersive X-ray spectroscopy (SEM-EDS), and LCR meter. XRD results verify that XRD pattern of CSZ pellets have similar pattern to that analysed for reference CSZ pellet which is a cubical structure. SEM images of CSZ pellets exhibited pores formation which influenced ionic conductivity value. The highest ionic conductivity value of CSZ pellets achieved at a calcination temperature of 700 °C with the measuring temperature of 600 °C is 4.83 x 10<sup>-5</sup> S/cm. Therefore, the essential outcome of this research is the recommended optimum calcination temperature of CSZ pellets made through a sol gel method of 700°C.

**Keywords:** Ionic conductivity, SOFC, electrolyte, CSZ, temperature

## 1. INTRODUCTION

Solid Oxide fuel cell (SOFC) is one of potential energy sources which can contribute to supply energy needs in the future. SOFC has demonstrated its high efficiency, high availability and reliability as well as good durability in providing alternative energy. SOFC systems provide an electrical efficiency of around 60% and a Combine Heat and Power (CHP) system efficiency up to 85-90% [1, 2]. Solid oxide fuel cell contains three main components which are an anode, a cathode and a solid electrolyte. The solid electrolyte is the important part of a SOFC, which is sandwiched between the cathode and the anode. It acts as a path for ions to flow from a cathode to an anode [3]. Its electronic conductivity must be kept as low as possible to prevent losses from leakage currents. The solid electrolytes to be used for high temperature fuel cell should have a good number of properties such as high oxide ion conduction with negligible electronic conductivity, phase stability, non-porous, good mechanical strength, thermal shock resistance,

chemical inertness to the reactive gas, compatibility with electrode [4,5]. The solid electrolyte widely used for SOFC is Ytria Stabilized Zirconia (YSZ) which produces sufficient ionic conductivity at operating temperature as high as 1000 °C [6]. However, the current technical challenges associated with operating at high temperatures poses an issue to SOFC durability, and have limited the application of SOFC for developing portable, auxiliary power systems and portable appliance [7,8]. Lowering the operation temperature of SOFC from 1000°C to 600-800°C may allow for a significant cost reduction for SOFC systems by broadening the choice of proper materials [9,10]. In addition, availability of YSZ resources in Indonesia is limited and utilisation of Ytria ( $Y_2O_3$ ) for stabiliser is relatively small hence it is not an economical option. One of the promising oxides for use as dopants for zirconia is Calcia (CaO). Calcia has been increasingly becoming a potential element as Calcia Stabilized Zirconia (CSZ) because of its conductivity and sufficient mechanical properties [11-15]. Abundant source of Calcia in Indonesia and its relatively low price bring potential prospect for research and development of SOFC. Previous research showed CSZ electrolyte has ionic conductivity of  $1.108 \times 10^{-5}$  S/cm at a temperature of 450 °C [16]. The aim of this research is to measure ionic conductivity of CSZ pellets with three different calcination temperatures prepared by sol gel method. In sol gel method, there is a phase changing to be sol (a suspended solid colloid in a solution) and further become gel (a colloid with greater solid fraction than a sol). Microstructure of the CSZ pellets was also analysed. According to [17], conductivity value of the CSZ electrolyte should be higher than 0.1 S/cm at a temperature of 1.000 °C or 0.3 mS/cm at a temperature of 500 °C in order to comply with the requirements of its application [17].

## 2. Experimental Method

### 2.1 CSZ Pellet Preparation

CSZ pellets production using sol gel method has been previously conducted with different parameter of calcination temperatures [18]. Production of CSZ solid electrode was initiated by producing  $Zr(OH)_4$  which was made from extraction of zirconium silicate ( $ZrSiO_4$ ) with caustic fusion. 100 gram of  $ZrSiO_4$  was mixed with 180 grams of NaOH and calcinated for 3 hours at a temperature of 700°C. After the mixture was cooled, the next step was to grind it to gain the powder form. 20 grams of the powder was mixed with 150 ml of aquades and then followed by heating at temperature of 150 °C for 30 minutes while stirring. Once sedimentation occurred, water was drained and 300 ml of aquades was taken to wash the sediment, and followed by heating at 150°C. This process was repeated for 6 times until the solution and sediment could not be separated. The pH was also checked and the value should be within the range of 6-7. The next step was to produce  $Zr(OH)_4$  by the drying process in the furnace followed by the drying under at a temperature of 110°C for 24 hours. Next stage is

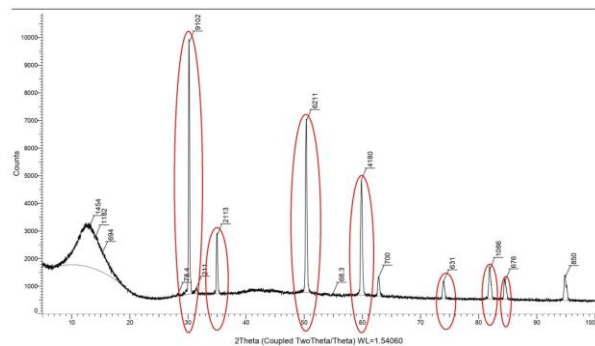
leaching using HCl 5M at a temperature of 90°C for 2 hours and then left it at room temperature for 1 night. Once the acid leaching yield was deposited, filtering was conducted. The filtered solution was then crystallised in a furnace at a temperature of 70°C until it was fully crystallised and produced  $ZrOCl_2 \cdot HCl$ . Afterwards, aquades was added to the  $ZrOCl_2 \cdot HCl$  crystal and hereafter crystallisation process was carried out in a furnace at 70°C, until  $ZOC(ZrOCl_2 \cdot 8H_2O)$  was formed. The yield was then filtered and added an alcohol to remove the remaining acid content. Solution of  $ZrOCl_2$  was mixed and heated with ammonia until its white colour was achieved. ZOC was then dissolved in HCl and ammonia was also added until the colour of solution was white. The next step was washing process using aquades and it was repeated until the solution achieved a pH of 8-9. The filtrate that was obtained from filtration process, was dried completely at a temperature of 150°C in order to transform ZOC to  $Zr(OH)_4$ . Production of CSZ powder using sol gel method was performed by dissolving 4.5 gram  $Zr(OH)_4$  into HCl 5M at a temperature of 90 °C using magnetic stirrer. Further, 4.2 gram of citric acid and 0.5 gram of CaO were diluted in 20 ml HCl 1M and was followed by heating in a furnace under temperature range of 70°C – 90°C until gel was formed. The gel was then processed for calcination under three different temperatures 500 °C, 600 °C and 700 °C for 3 hours respectively. Once the cooling process was completed, CSZ powder was resulted. CSZ pellets were produced from CSZ powder by pressing proses followed by then sintering at a temperature of 1200°C for 3 hours and continued for cooling down.

### 2.2 Characterization of CSZ pellet

The characterization was carried out for 3 CSZ pellets using XRD while their microstructure analysis was done using Scanning Electron Microscope (SEM) operated at 20 kV. SEM-EDS was also performed for chemical analysis. Ionic conductivities measurements were conducted using LCR meter with electrical resistance measurement at a potential of 0.7 volt under the temperature range of 200 °C - 600 °C.

## 3. Results and Discussion

The XRD patterns of CSZ pellets under three different calcination temperatures are shown in Figs. 1, 2 and 3.



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FIGURE 1 XRD PATTERN OF SINTERED CSZ PELLETT AT CALCINATION TEMPERATURE OF 500°C.

Figure 1 showed that the XRD patterns are single phase and exhibited strong diffraction peaks at 30°, 35°, 50°, and 60°.

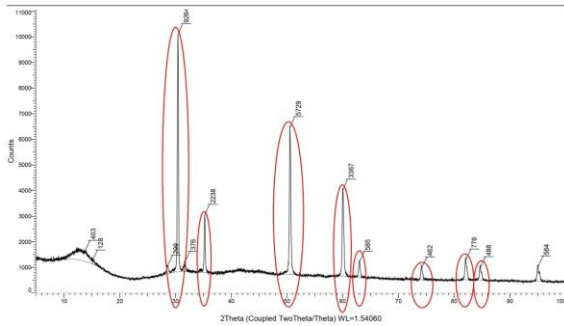


FIGURE 2 XRD PATTERN OF SINTERED CSZ PELLETT AT CALCINATION TEMPERATURE OF 600°C.

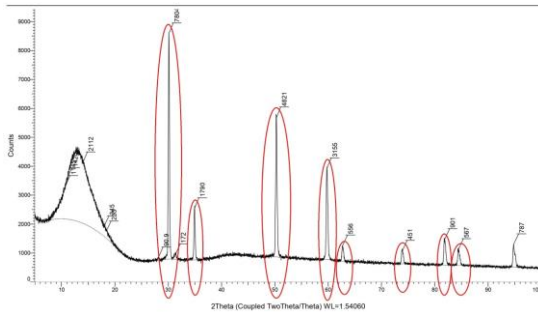


FIGURE 3 XRD PATTERN OF SINTERED CSZ PELLETT AT CALCINATION TEMPERATURE OF 700°C.

It is obviously shown in Figs. 2 and 3 that XRD pattern has the same strong peak as in the Fig. 1. The three XRD patterns of those CSZ pellet samples, further compared to reference pattern of CSZ pellet, as presented in Fig. 4 [18].

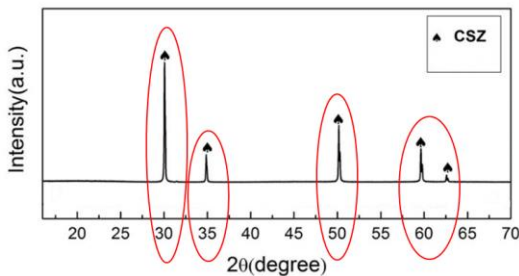


FIGURE 4 XRD REFERENCE PATTERN OF CSZ PELLETT (INDICATED BY RED CIRCLE) [18].

The XRD patterns of all CSZ pellets in Figs. 1, 2 and 3 are single phase. The result shows that the XRD patterns of CSZ pellets at different calcination temperatures have similarity patterns to the reference XRD pattern of CSZ pellet shown in Fig. 4. The diffraction peaks (2θ) are 30°, 35°, 50°, and 60° which correspond to cubic structure. This

structure is known as the most stable structure to allow the flow of oxygen ions.

SEM images of the CSZ pellets formed under the three different calcination temperatures are presented in Figs. 5, 6 and 7.

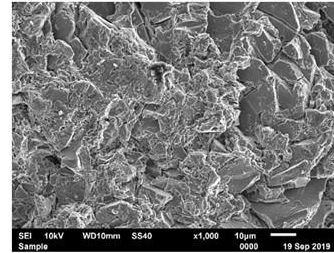


FIG. 5 SEM IMAGES OF CSZ PELLETT AT CALCINATION TEMPERATURE OF 500 °C.

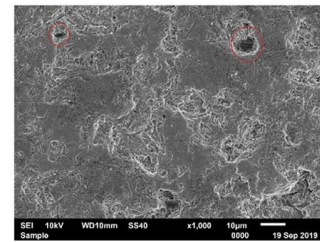


FIG. 6 SEM IMAGES OF CSZ PELLETT AT CALCINATION TEMPERATURE OF 600 °C.

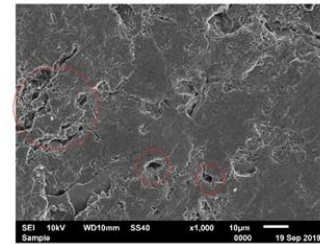


FIG. 7 SEM IMAGES OF CSZ PELLETT AT CALCINATION TEMPERATURE OF 700 °C.

The SEM images in Figs. 5, 6 and 7 showed that the CSZ pellet were not homogenous and still porous, or defect as indicated by red circle in which the higher calcination temperature, the more porosity occurs. The existence of these pores caused the density of the pellet to decrease, and the resulted electrolyte was less optimum. At a calcination temperature of 500°C, the resulted pellet was distantly located and tend to have less binding effect hence many cracks occurred at the pellet boundaries (see Fig. 5). Therefore, it is not recommended to conduct synthesis at a calcination temperature of 500°C. The defects in the pellets could be attributed to the agglomeration of fine CSZ particles as a result of poor compaction and inhomogeneous densification during sintering [19, 20]. The microstructures significantly influenced the resulted conductivity ionic value. In order to measure the ionic conductivity of CSZ electrolyte, LCR meter was used accordingly. This measurement was conducted at temperature of 200 °C to 600 °C and the results are shown in Table 1.

TABLE 1. ELECTROLYTE IONIC CONDUCTIVITY VALUE OF SINTERED CSZ PELLET AT MEASURING TEMPERATURE OF 200-600 °C.

Measuring temperature (°C)	Ionic Conductivity (S/cm) at three calcination temperatures		
	500 °C	600 °C	700 °C
200	$1.41 \times 10^{-7}$	$1.29 \times 10^{-7}$	$1.18 \times 10^{-7}$
300	$1.88 \times 10^{-7}$	$1.13 \times 10^{-7}$	$1.29 \times 10^{-7}$
400	$2.56 \times 10^{-7}$	$3.59 \times 10^{-7}$	$8.23 \times 10^{-7}$
500	$4.16 \times 10^{-6}$	$5.21 \times 10^{-6}$	$6.58 \times 10^{-6}$
600	$3.89 \times 10^{-5}$	$3.68 \times 10^{-5}$	$4.83 \times 10^{-5}$

Table 1 shows the result of ionic conductivity measurement of CSZ pellets at different calcination temperatures with measuring temperature at the range of 200 °C-600 °C. The higher the measuring temperatures, the higher the ionic conductivity values measured under three different calcination temperatures. This is a consequence of temperature increase which increases the amount of oxygen ion that could exceeds activation energy [15].

None of ionic conductivity value on those three CSZ pellets achieved the target. However, ionic conductivity value of CSZ pellet at a measuring temperature of 600 °C for three calcination temperatures are higher compared to the previous research which was  $1.108 \times 10^{-5}$  S/cm at a temperature of 450 °C [16]. The electrolyte material in SOFC should be dense enough so that the fuel and oxidant could not pass through the electrolyte [21]. As mentioned earlier, the microstructures of CSZ pellets showed the presence of pores. The presence of pore in the grain boundaries of pellet interferes ion transport between grains since pore acted as inhibitor of ion motion. Pores distributed in the grain boundaries had higher blocking effect than pores that were concentrated at the junction of the three grains. SEM images in Figs. 5, 6 and 7 show that the pores were distributed in the grain boundaries. The pore formation of CSZ pellets reduced cell performance by decreasing the ionic conductivity.

SEM-EDS measurement results of CSZ pellets under the three calcination temperatures are shown in Fig. 8 and quantitative analysis of SEM-EDS of CSZ pellets is shown in Table 2.

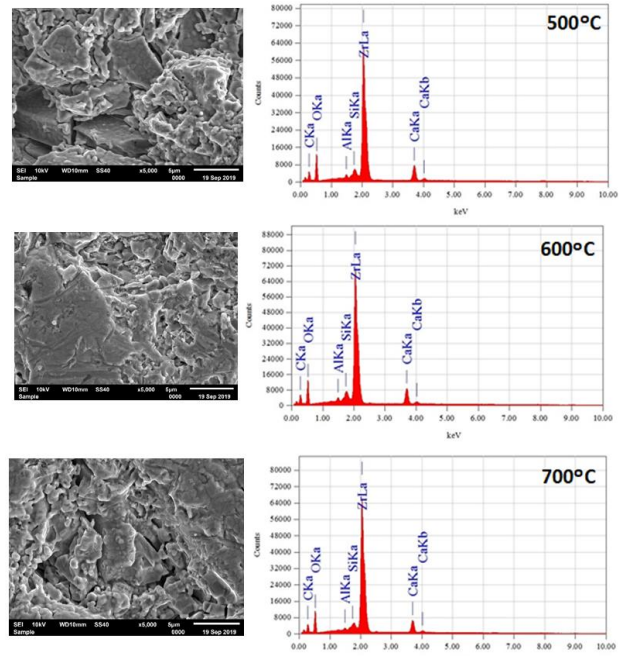


FIG. 8 SEM-EDS RESULT OF CSZ PELLET AT THREE DIFFERENT CALCINATION TEMPERATURES.

Qualitative SEM-EDS analysis of the three CSZ pellets in Fig. 8 showed the presence of silica as an impurity. Silica content inside the CSZ pellets was due to the extraction process of ZrO<sub>2</sub> which was performed using natural materials. The amount of Silica compound in the electrolyte was less than 1% mass as shown in Table 2 which indicated good quality of CSZ pellets.

TABLE 2. QUANTITATIVE ANALYSIS OF SEM-EDS OF CSZ PELLETS UNDER THREE CALCINATION TEMPERATURES (IN % MASS.)

Element	Quantitative analysis of SEM EDS (in % Mass)		
	500(°C)	600(°C)	700(°C)
C	22.82	24.09	23.82
O	31.59	29.38	28.77
Al	0.47	0.50	0.34
Si	0.54	0.66	0.23
Ca	3.33	3.46	2.88
Zr	41.27	41.94	43.97

Nevertheless, although the CSZ pellets have cubical structure and less Silica content of the impurity of less than 1 % mass, ionic conductivity was significantly influenced by impurity. This impurity was seen to decrease the ionic conductivity values. Low concentration of its presence in the solid electrolyte of CSZ was impactful in the reduction of ionic conductivity value and raised resistivity particularly in the grain boundaries [23]. Therefore, low ionic conductivity achieved on CSZ pellets produced in this research was strongly affected due to the pores formation in the grain boundaries and under the presence of silica impurity.

## 4. Conclusion and Recommendation

### 4.1 Conclusions

Based on the measured data, the following conclusions can be drawn:

- a) XRD results showed that the CSZ pellet made through a sol gel under different calcination temperatures of 500 °C, 600 °C, 700 °C flowed by a sintering temperature of 1200 °C respectively formed cubical structure.
- b) No experimental results of ionic conductivity measurement achieved the target due to pore formation in the pellet boundaries and the presence of silica impurities. The highest ionic conductivity value of CSZ pellets was  $4.83 \times 10^{-5}$  S/cm which was achieved at calcination temperature of 700 °C with the measuring temperature of 600 °C.
- c) It is suggested to produce CSZ electrolyte made through sol gel method with the parameter used in this research under the calcination temperatures of 700°C.

### 4.2 Recommendation

By using similar synthesis method, it requires further experiments on varying sintering temperature and adding doping compound to minimise pore formation and remove the impurity hence the targeted conductivity ionic value can be achieved.

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