

Characteristics of Gadolinium Doped Cerium at Different Calcination Temperatures for Intermediate Temperature SOFC

(Pencirian Gadolinium Terdop Serium pada Suhu Kalsinasi Berbeza untuk Suhu Pertengahan SOFC)

DAMISIH, ADE UTAMI HAPSARI, AGUSTANHAKRI, YELVIA DENI, MAHENDRA ANGGARAVIDYA, OKA PRADIPTA ARJASA, JAROT RAHARJO* & MAHENDRA RAO SOMALU

ABSTRACT

Gadolinium doped cerium ($Ce_{0.9}Gd_{0.1}O_{1.95}$ or GDC10) was successfully synthesized using the solid-state method. Commercially available CeO_2 and Gd_2O_3 powders were used as starting materials. They were mixed in a ball mill where alumina balls were added as grinding medium with the ratio to powders as of 1:2. The obtained powders were dried and then calcined at temperatures of 600, 700 and 800 °C, respectively. The objective of this research was to investigate the effects of calcination temperature on the properties of GDC10. The powders were characterized using XRF, TGA, XRD, and PSA instruments. XRF analysis shows the presence of Ce, Gd and O elements in stoichiometric composition without any impurities. XRD analysis showed single phase structure of CeO_2 where the crystallite size and lattice parameter increases and decreases, respectively, as the calcination temperature increases. The smallest particle size of 647.3 nm was obtained at the calcination temperature of 600 °C. The density of all GDC10 samples sintered at 1350 °C was found to be higher than 95%. In addition, the calcination temperature also influenced the ionic conductivity where the highest obtained value was 0.0153 S.cm⁻¹ at 800 °C for the sample calcinaed at 600 °C. The results suggest that the calcination temperature affected the properties of GDC10 for solid oxide fuel cell application.

Keywords: Ball milling; calcination temperature; IT-SOFC; solid-state

ABSTRAK

Gadolinium terdop cerium ($Ce_{0.9}Gd_{0.1}O_{1.95}$ atau GDC10) telah berjaya disintesis dengan menggunakan kaedah keadaan pepejal. Serbuk CeO_2 dan Gd_2O_3 yang tersedia komersial digunakan sebagai bahan pertama tindak balas. Kesemua bahan pemula telah dicampur dalam pengisar bebola dengan bebola alumina digunakan sebagai medium pengisar dengan nisbah kepada serbuk ialah 1:2. Serbuk yang diperolehi dikeringkan dan kemudian masing-masing dikalsin pada suhu 600, 700 dan 800 °C. Objektif penyelidikan ini ialah untuk mengkaji kesan suhu pengkalsinan ke atas sifat-sifat GDC10. Pencirian serbuk dilakukan dengan menggunakan alatan XRF, TGA, XRD dan PSA. Analisis XRF menunjukkan kehadiran unsur-unsur Ce, Gd dan O dalam komposisi stoikiometri tanpa kehadiran sebarang bahan bendasing. Analisis XRD menunjukkan struktur fasa tunggal CeO_2 dengan saiz kristal meningkat apabila suhu pengkalsinan bertambah manakala parameter kekisi sebaliknya. Saiz zarah terkecil 647.3 nm diperolehi pada suhu kalsinasi 600 °C. Ketumpatan semua sampel GDC10 yang disinter pada 1350 °C lebih tinggi daripada 95%. Di samping itu, suhu pengkalsinan juga mempengaruhi kekonduksian ion dengan nilai tertinggi yang diperolehi ialah 0.0153 S.cm⁻¹ pada suhu 800 °C untuk sampel yang dikalsin pada suhu 600 °C. Keputusan kajian menunjukkan bahawa suhu pengkalsinan mempengaruhi sifat-sifat GDC10 untuk aplikasi sel fuel oksida pepejal.

Kata kunci: IT-SOFC; keadaan pepejal; pengilangan bebola; suhu kalsinasi

INTRODUCTION

Solid oxide fuel cell (SOFC) becomes one of alternative of environmentally friendly technology for power generation which is increasing interest as low emission of energy conversion device (Somalu et al. 2017).

Principle operation of SOFC is based on a combination of electrochemical reaction to produce electricity (Fu 2014). It can generate electricity from different types of fuel for some applications such as for stationary and transportation (Mahmud et al. 2017). The advantages of

SOFC consist of it has high efficiency, high flexibility for fuel usage and low emission. The main component of SOFC is electrolyte where zirconia-based ceramic such as YSZ becomes popular materials for this application (Raharjo et al. 2017). YSZ electrolyte produces high current density and notably power output at operating temperature of 800-1000 °C (Sriyanti et al. 2009). However, the high operating temperature has some issues for commercial-scale such as shorter lifetime, reduced thermal stability for long term application and high cost. So that, lowering the temperature of SOFC operation to intermediate-range between 600-800 °C becomes an appropriate alternative to overcome this issue (Raharjo et al. 2008).

By reducing the temperature, the performance of SOFC will be degraded because the conductivity of YSZ electrolyte will drastically decrease. An approach to solve this problem is by using alternative electrolyte materials that have higher ionic conductivity than YSZ at the intermediate temperature as like cerium based ceramic oxide. Generally, pure ceria (CeO_2) shows a cubic fluorite-type crystal structure from room temperature to its melting point where it is reduced to CeO_{2-x} as temperature increased (Anwar et al. 2018).

Some requirements for SOFC electrolyte such as it should possess high ionic conductivity, high density over 95% and high thermal stability (Fuentes et al. 2008). One strategy to improve properties of ceria based electrolyte is by doping where foreign atoms in small amount are added to the crystal lattice of host atoms. It changes low valence of cations in lattice to keep neutrality of oxygen charges and alter thermal properties as well (Kuphaldt 2010).

Ceria compounds that are doped with rare-earth elements are very potential to have higher ionic conductivity than YSZ (Sun et al. 2018). If rare earth oxide with two or three valences is added to cerium, oxygen vacancies will be created and ionic conductivity will be improved (Sriyanti et al. 2009). There are some rare-earth ions such as Sm^{3+} , Gd^{3+} , Y^{3+} , Nd^{3+} and La^{3+} that can be used as dopant for ceria compound (Anwar et al. 2019). However, dopant element which is widely used is gadolinia (Gd). And recently, gadolinia doped ceria with a stoichiometric composition of $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ or GDC10 has attracted much attention as promising alternative materials for the intermediate temperature of SOFC (Bi et al. 2017). It is CeO_2 with 10 mol% of Gd_2O_3 which is reported for having higher ionic conductivity than other composition (Burinskas et al. 2011). One of several techniques for preparing GDC10 is by the solid-state method using a ball mill. It is employed due to the relatively simple and low-cost process to produce finer particle (Izquierdo et al. 2018).

In this works, gadolinium doped cerium was prepared by the solid-state method using a ball mill. The obtained powders were dried and calcined at various temperatures of 600, 700 and 800 °C. The effects of calcination temperature were investigated in term for the application of intermediate solid oxide fuel cell electrolyte.

MATERIALS AND METHODS

Gadolinium doped cerium with composition $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (or GDC10) has been successfully synthesized using the solid-state method. Solid state reaction is the first method used to refine and synthesize doped ceria powders due to its cost effectiveness (Mahmud et al. 2017). Commercially available of CeO_2 and Gd_2O_3 powder (Sigma Aldrich) were mixed in appropriate stoichiometric in a ball mill. Ethanol was added to powder as a mixing agent. It was used because immediately evaporated and no side reaction occurred. The mixture was put into a stainless steel vial where alumina balls were added as grinding medium with the weight ratio of alumina balls to the powder was 1:2.

Ball milling was performed at around 4 h with the rotation speed of 240 rpm. After ball milling, samples were dried for 24 h in an oven. The temperature was set up at 110 °C. Subsequently, the powders were divided into 4 portions. It was subjected to calcination with different temperature i.e. 600, 700, and 800 °C. One portion of powders was left without calcination.

The obtained powders were characterized by X-Ray Fluorescence (XRF) to analyse powders composition. Thermal Gravimetric Analysis (TGA, LabSys EVO TG-DTA, France) was performed to characterize its thermal stability. X-Ray Diffraction (XRD, Rigaku, Japan) with $\text{Cu-K}\alpha$ operating at 40 kV and 30 mA was used to determine phase and crystal structure. XRD spectra were obtained in the range of 10 - 90° with 0.5° of slit size. The Particle Size Analyser (PSA, Malvern) was used to show the particle size of powders and its distribution.

GDC10 powders were uni-axially pressed with at 3 tons into a disc-shaped pellet. Acrysol was added to powders as a binder to strengthen of pellet structure. The pellets were sintered at a temperature of 1350 °C. Then, its density and ionic conductivity were measured using electrochemical interface (Solartron SI-1286).

RESULTS AND DISCUSSION

Table 1 shows the composition of GDC10 powders obtained by X-Ray Fluorescence (XRF). The results of XRF analysis indicates cerium (Ce), oxygen (O) and gadolinium (Gd) elements were found on the GDC10 powders without any other impurities.

TABLE 1. Composition of GDC10 powders by XRF analysis

Calcination	Mass (%)		
	Ce	O	Gd
No-calcined	93.30	0.0098	6.69
600 °C	93.40	0.0097	6.59
700 °C	93.29	0.0098	6.70
800 °C	93.27	0.0098	6.70

Figure 1 shows the Thermal Gravimetric Analysis (TGA) of GDC powders at various calcination temperatures. TGA analysis was conducted in a dry air atmosphere to analyse the thermal decomposition. As can be seen from Figure 1, weight loss is not significantly occurred in the temperature range of 600-800 °C. It indicates that GDC10 was thermally stable in the mentioned range where intermediate solid oxide fuel cell operates in this temperature. TGA curve in this research can be divided into 3 segments namely segment I, segment II, and segment III, as shown by Figure 1. Segment I was related to moisture or absorbed gas on the surface of powders (Chuang et al. 2013). The elimination of organic residual was almost completed in segment II and III at temperature 600-800 °C. Thermal stability phase was also noted in this segment. Therefore, it may represent a suitable calcination temperature for GDC10 powders. This result was supported by other researchers where Cheng et al. (2003) calcined GDC10 powders at 600 and 800 °C and Herle et al. (1996) performed calcination at 700 and 800 °C.

For GDC10 no-calcined sample, TGA weight loss at segment I was about 1.971%, while at segment II and III was 1.748%. Thus, the total weight loss was 3.719%. It was noted weight loss of sample calcined at 600 °C was 1.569% at segment I. The total weight loss for sample calcined at 700 °C was 2.429% with 1.129% at segment I and 1.3% at segment II & III. The sample calcined at 800 °C showed a total weight loss of 1.653% with 0.317% loss at segment I and 1.336% in segment II & III.

Figure 1 shows that the highest weight loss is found at uncalcined GDC10 powders, while GDC10 powders calcined at 800 °C shows the smallest weight loss. Weight loss tends to decrease as calcination temperature increase. Thermal stability, one of them is determined by weight loss where the smaller weight loss gives the higher thermal stability properties (Awang et al. 2018; Ma et al. 2010). It can be said that GDC10 powders calcined at 800 °C give the best thermal stability for IT-SOFC electrolyte application.

X-Ray Diffraction (XRD) patterns for GDC10 calcined at different temperature are shown in Figure 2. All of the samples show the same diffraction peaks pattern with (111) as the major peak. Other peaks are indexed as (200), (220), (311), (222), (400), (331), (420), and (422). It exhibits a single-phase structure of CeO₂ according to ICDD 01-075-0161 where its crystal shows face-centred cubic (fcc) structure. There was no Gd₂O₃ phase found indicating that the solid solution was directly formed (Arabaci et al. 2012).

It can also be seen from the XRD pattern that the major peak intensity tends to increase with increasing calcination temperature (Choolaei et al. 2018; Prasad et al. 2008). It is clearly observed if comparing the GDC10 powder calcined at 800 °C with the uncalcined powder. It demonstrates the improvement of powders crystallinity during the calcination process.

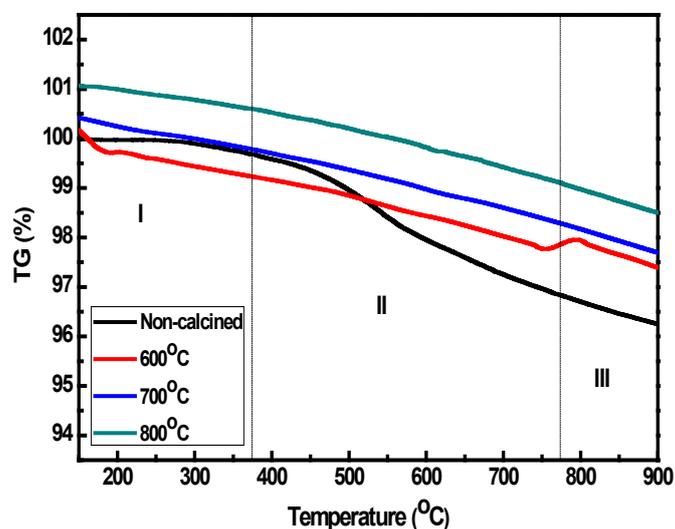


FIGURE 1. TGA spectra plot of GDC10 at different calcination temperature

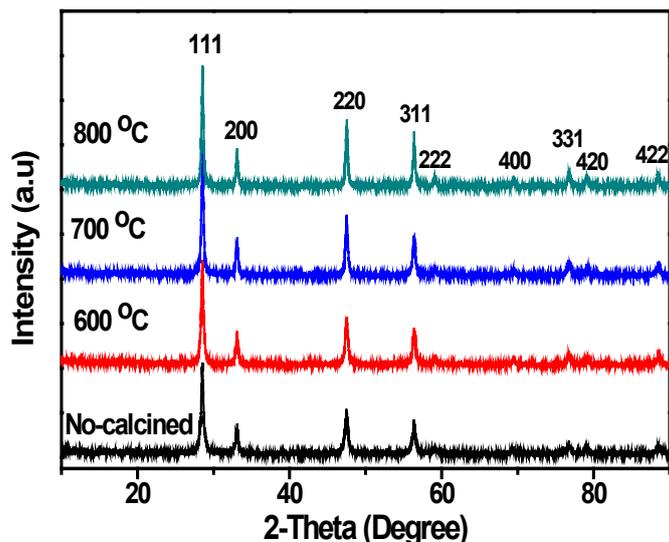


FIGURE 2. XRD pattern of GDC10 at different calcination temperature

The crystallite size and lattice parameter were calculated using Scherrer's formula and shown in Table 2. It can be seen from Table 2 that the crystallite size of the uncalcined GDC10 is 22.12 nm. At calcination temperature of 600 and 700 °C, the crystallite sizes are 22.72 and 22.94 nm, respectively. Crystallite size further increases to 28.08 nm at 800 °C. However, the lattice parameter decreases as calcination temperature increases. Lattice

parameter was observed of 5.4126, 5.4119, 5.4115, and 5.4114 Å for non-calcined, calcined at 600, 700, and 800 °C, respectively. The crystallite size was proportional to the percent of weight loss according to TGA results where the larger crystallite size gave the higher thermal stability. In vice versa, lattice parameter was inversely proportional to the weight loss.

TABLE 2. Crystallite size and lattice parameter of GDC10 at various calcination temperatures

Calcination condition/ temperature	FWHM	Crystallite size (nm)	Lattice parameter (Å)
No-calcined	0.33	22.12	5.4126
600 °C	0.32	22.72	5.4119
700 °C	0.32	22.94	5.4115
800 °C	0.26	28.08	5.4114

Particle size analysis was performed using the Dynamic Laser Scattering (DLS) method with Tween 80 as a surfactant. The particle size and its distribution are shown in Figure 3. The particle size of the uncalcined GDC10 was 622.9 nm with Polydispersity Index (PDI) 0.317. The particle size of 647.3 nm with PDI 0.554

was shown at a calcination temperature of 600 °C. At temperature 700 °C, the particle size was 755.7 nm with PDI 0.328. And the highest particle size was found at a calcination temperature of 800 °C with the hydrodynamic diameter of 811.8 nm.

The results of particle size analyser show that particle size increases as calcination temperature increase. This is in accordance with crystallite size calculation from XRD data which shows that as calcination temperature increases, crystallite size decrease. This is probably due to grain growth where the smaller grains will consolidate with bigger and more stable grains (Lee et al. 2016). It was also reported by Raharjo et al. (2017) that particle size increased if calcination temperature decreased.

Besides particle size, polydispersity index or PDI number is indicated to decrease with increasing of calcination temperature. This number is used to describe the degree of non-uniformity of particle size which ranges from 0 to 1 (Danaei et al. 2018). If PDI number more than 0.7, it indicates that the powder is polydispersed. Here, PDI value is less than 0.7 that means it is appropriate for the Dynamic Light Scattering method.

Figure 4 shows the relative densities of GDC10 pellet with various calcination temperatures. The pellet was sintered at temperature 1350 °C and then measured its relative density using Archimedes method (Arabaci et al. 2012). Sintering was performed to convert powders to become a dense ceramic electrolyte for subjecting to high operating temperature (Winnubst et al. 2009). One of the requirements for IT-SOFC electrolyte is having a high density of over 95% (Yasuda et al. 2012).

As seen from Figure 4, the density of pellet improves after powders were calcined. The particle size after calcination was found to decrease and then it enhanced densification of pellet. The density of pellet is influenced by some factors such as calcination and sintering temperature, particle size and pressure given during fabrication (Caisso et al. 2017). It led the decreasing of volume so that density increased.

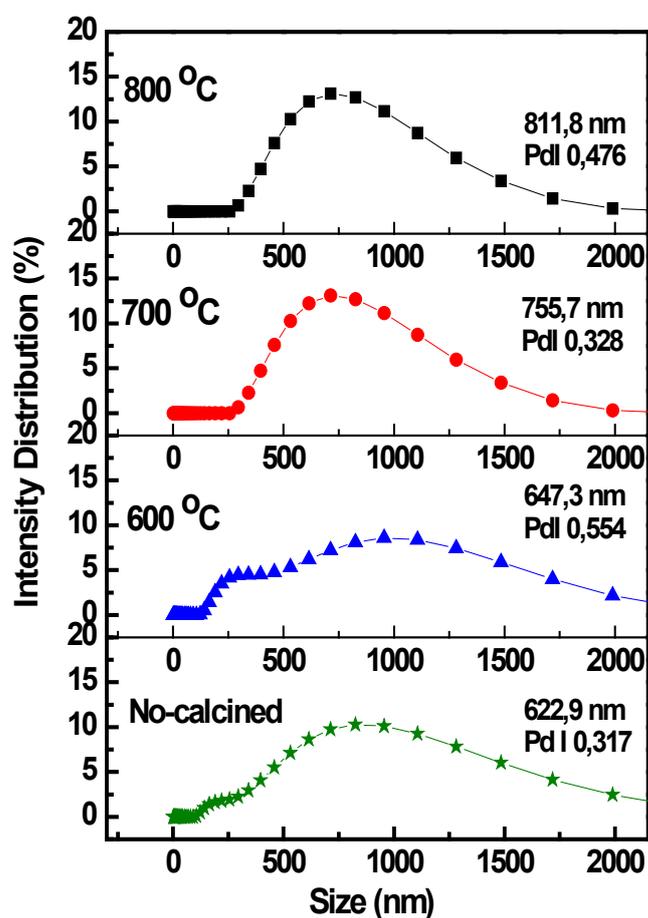


FIGURE 3. Particle size and particle size distribution of GDC10 at different calcination temperature

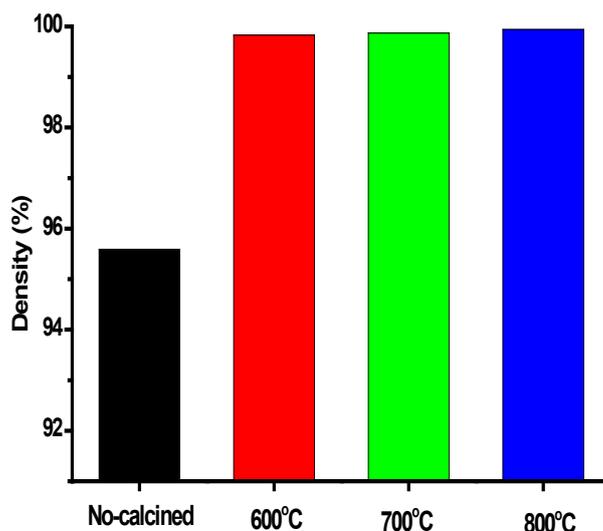


FIGURE 4. The density of GDC10 pellet disk at different calcination temperature

In general, particle size is inversely proportional to its density where the smaller particle will introduce higher density (Taer et al. 2018). However, in this research, the density was getting higher as particle size was getting bigger. It may be due to the heating processes during calcination and sintering where the energy contained in the powder became higher as temperature increase and particles tended to move. Those moving particles would approach each other as the porosity was shrunk. As a result, the distance between particles became closer and density was improved (Baral et al. 2013). GDC10 pellet from powder that calcined at 800 °C obtained more heat energy than other samples so that its energy was higher. It caused the densification process to become faster and then density was higher. On the other hands, the porosity is inversely proportional to the density. The decrease in porosity indicates that the densification process occurred.

The highest relative density of 99.94% was obtained for sample after calcined at 800 °C. However, the density for all calcined samples are not much different from each others. As mentioned before, solid oxide fuel cell electrolyte required minimum density of 95% to minimize hydrogen gas leakage (Dikmen et al. 2010), so that all of GDC10 sample pellet has fulfilled those requirements.

The ionic conductivity of sintered GDC10 pellet that calcined at various temperatures is shown in Table 3. Sintering was performed at 1350 °C according to Dikmen et al. (2010), but it was slightly lower than other researchers (Arabaci et al. 2012; Cheng et al. 2003). As expected, conductivity increased as operating temperature increased. The relative density and grain size were reported to increase with increasing sintering temperature which was followed by decreasing of ionic conductivity (Ali et al. 2017).

TABLE 3. Ionic conductivity of GDC10 pellet at various calcination temperatures

Operating temperature (°C)	Ionic conductivity at calcination temperature (S.cm ⁻¹)		
	600 °C	700 °C	800 °C
400	0.00010	0.00010	0.000076
450	0.00028	0.00048	0.00025
500	0.00082	0.00093	0.00063
550	0.0018	0.0018	0.0013
600	0.0024	0.0031	0.0024
650	0.0047	0.0052	0.0040
700	0.0077	0.0071	0.0058
750	0.0110	0.0095	0.0076
800	0.0153	0.0115	0.0091

The requirement for solid oxide electrolyte was to have a minimum conductivity of 0.01 S.cm^{-1} at operating temperature (Irshad et al. 2016). According to the requirement, GDC10 pellet that was calcined at $600 \text{ }^\circ\text{C}$ when operating at 750 and $800 \text{ }^\circ\text{C}$ has fulfilled that requirement with the conductivity of 0.011 and 0.0153 S.cm^{-1} , respectively. Meanwhile, the powder which was calcined at $700 \text{ }^\circ\text{C}$, could only produce ionic conductivity of 0.0115 S.cm^{-1} at $800 \text{ }^\circ\text{C}$. Moreover, it was observed that all of the ionic conductivity value was below 0.01 S.cm^{-1} at the calcination temperature of $800 \text{ }^\circ\text{C}$ at any operating temperature. Here, the highest ionic conductivity of 0.0153 S.cm^{-1} was obtained at a calcination temperature of $600 \text{ }^\circ\text{C}$ when operating at $800 \text{ }^\circ\text{C}$. The maximum ionic conductivity was lower than the other electrolyte based such as scandia-stabilized-zirconia at the same temperature ($800 \text{ }^\circ\text{C}$) i.e. 0.184 S/cm reported by Jais et al. (2017).

CONCLUSION

In this works, the calcination temperature has been found affecting onto properties of gadolinium doped cerium with composition $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC10) synthesized by solid-state method. XRF analysis shows the stoichiometric composition of elements Gd, Ce and O without any impurities. The high thermal stability at intermediate temperature was shown by TGA. XRD analysis showed single-phase structure of CeO_2 where crystallite size increase as calcination temperature increase and lattice parameter in vice versa. The particle size also increased with increasing calcination temperature. The smallest particle size of 647.3 nm was obtained at a calcination temperature of $600 \text{ }^\circ\text{C}$. In addition, calcination temperature influenced ionic conductivity where the highest value was 0.0153 S.cm^{-1} that was also obtained at a calcination temperature of $600 \text{ }^\circ\text{C}$. This works demonstrate that, by tailoring particle size with increasing calcination temperature, it can give the optimum ionic conductivity.

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- Damisih, Ade Utami Hapsari, Agustanhakri, Yelvia Deni, Oka Pradipta Arjasa, Mahendra Anggaravidya & Jarot Raharjo*
Center for Materials Technology
Agency for the Assessment and Application of Technology
Building 224
Puspipstek Area
South Tangerang 15314
Indonesia
- Mahendra Rao Somalu
Fuel Cell Institute
Universiti Kebangsaan Malaysia
43600 UKM Bangi, Selangor Darul Ehsan
Malaysia

*Corresponding author; email: jarot.raharjo@bppt.go.id

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