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Determination of Sintering Temperature Decrement by Improving Samarium Doped Ceria (SDC) based solid oxide fuel cell (SOFC) Electrolyte Through Rice Husk Silica (RHS)

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Abstract

This research effort addresses the effects of rice husk silica (RHS) on the sintering temperature of samarium-doped ceria (SDC)-based solid oxide fuel cell (SOFC) electrolytes since the high operating temperature is quite high. This research hopes to improve the electrolytes' thermal stability and conductivity by combining RHS. The procedure involves developing SDC-based electrolytes with different weight percentages of RHS and SDC, which are 97:3, 98:2 and 99:1, followed by systematic sintering at detailed temperatures such as 1000°C,1100°C and 1200°C. The outcomes of this research involve improved SOFC performance and efficiency, an increasingly homogeneous microstructure with fewer flaws, greater structural integrity, electrolyte lifespan, increased porosity and unity, and improved ionic conductivity and mechanical reliability. These technological breakthroughs in sintering, SEM, and FTIR are expected to improve SOFC efficiency and applications.

1. Introduction

Solid oxide fuel cells (SOFCs) employ an electrochemical method involving a solid, ceramic electrolyte commonly made of yttria-stabilized zirconia (YSZ) or ceria-based components. These electrolytes allow using a wide range of fuels and function at high temperatures of up to 1000 °C [1]. However, high operating temperatures provide obstacles like longer initial stage times and material difficulties with compatibility. This results in unsatisfactory electrolyte material performance at low sintering temperatures and could lead to the commercialization of SOFC [2].

Furthermore, durability and cost are ongoing issues, and this research is being carried out to improve SOFC lifetime and cost-effectiveness. This study aims to determine the effect of RHS on the characteristics of SDC electrolytes and hopes to improve the electrolytes' thermal stability and conductivity. Due to the presence of silica in RHS, the silica functions as a flux, facilitating components to melt and flow more easily, improving densification at lower temperatures [3]. This research investigates the impacts of different weight percentages of RHS (1%, 2%, and 3%), as well as SDC (97%, 98%, and 99%), on the sintering temperature in order to enhance SOFC performance.

2. Materials Method

This chapter provides a detailed overview of the preparation and characterization processes for SDC, RHS, and three option SDC-RHS compositions.

2.1 Materials Preparation

The material that is used will be listed in Table 1. Table 2 shows the composition of SDC-RHS.

Table 1 *List of Materials Used*

No. of item	Name	Chemical Formulae
1	Samarium-Doped Ceria	$(Sm_xCe_{1-x}O_2)$
2	Rice Husk Silica	SiO_2

Table 2 *Composition of SDC-RHS*

No. of item	Weight ratio (wt%)
1	99:1
2	98:2
3	97:3

2.2 Methods

Rice Husk Silica (RHS) is produced in Muar, Johor, through a multi-step process that extracts and refines pure silica from rice husk. The procedure begins with calcination, in which the rice husk is heated to 700°C for 4 hours in a particular furnace. This stage tries to eliminate contaminants and convert organic matter to silica.

After calcination, the Rice Husk Silica (RHS) is sized using a horizontal ball mill that rotates at 200 RPM for three hours. This procedure refines the component by lowering particle size and improving its characteristics.

A mixing procedure is used to prepare Samarium-Doped Ceria (SDC) from Rice Husk Ash Silica (RHS). All components are first milled to create uniform dispersion, ensuring that the SDC and RHS particles combine well. The combination of materials is then palletized to increase its compactness and enable the creation of uniform pellets.

The combination of samarium-doped ceria and rice husk silica (SDC: RHS) is accomplished using a ball milling method that involves modifying various weight percentages, including 99:1 (SDC: RHS), 98:2 (SDC: RHS), and 97:3 2 (SDC: RHS). Each ratio is precisely calculated and applied in order to study the subtle differences in the final mixture's qualities.

After the samarium-doped ceria and Rice Husk Silica (SDC: RHS) samples (Fig. 1) are fabricated, the compositions transform after being filled with varied weight percentages per the specified ratios. The materials get compressed into compact pallet forms using a hydraulic press machine with a force of 2 tonnes and a holding period of 3 minutes. This thorough technique produces well-defined samples suitable for analysis and future usage in fresh materials and energy technologies.

Fig. 1 *End product of SDC-RHS*

2.3 Analysis of Characterization Process

Fourier Transform Infrared Spectroscopy (FTIR), shown in Fig. 2, is an effective analytical technique for detecting and measuring broad chemical substances using their infrared absorption spectra. The chemical structure of SDC-RHS is identified using FTIR by detecting distinctive absorption bands, which is significant for SOFC electrolyte enhancement.

Fig. 2 *Fourier Transform Infrared Spectroscopy (FTIR)*

The elements distribution of SDC-RHS has been determined using a Scanning Electron Microscope (SEM) (Fig. 3), specifically the SU 1510 model, which was equipped with an Energy Dispersive Spectroscopy (EDS) module. SEM-EDS analysis was performed on cross-sectional samples to thoroughly evaluate the homogeneous microstructure of SDC and RHS.

Fig. 3 *Scanning Electron Microscope with Electron Dispersive Spectroscopy (SEM-EDS)*

3. Results and Discussion

Table 3 shows the compact pallet for all ratios of samples.

Table 3 *SDC-RHS compact pallet forms*

Ratio	Results
99:1	
98:2	
97:3	

3.1 Chemical Bonding of SDC-RHS (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a type of analysis that identifies the chemical structures present in organic and inorganic compounds by measuring their ability to absorb infrared waves over a range of wavelengths. It also measures a sample's infrared spectrum, which is a distinctive indicator of the molecular structure and composition. Each sample undergoes analysis using FTIR to determine its chemical bonding. It was done with a Perkin Elmer Spectrum 1000. The spectra for every single of sample were tuned to a frequency range 600 – 4,000 cm^{-1}. The FTIR spectra were displayed as percent transmittance (%T) versus wavenumber (cm^{-1}).

The SDC-RHS spectra is shown in Fig. 3.1 for ratio 99:1. At 1200°C, the stretching frequencies for C-H asymmetric, CH3 symmetric, and O-H were measured at 2982.36 cm^{-1} (93.91%), 1394.14 cm^{-1} (95.65%), and 1033.54 cm^{-1} (93.41%) respectively. Additionally, the absorption peak at 1056.47 cm^{-1} (92.87%) indicates the presence of CH2O and carbon-oxygen groups. Previous research discovered that the addition of rice husk silica increased its mechanical strength and thermal stability [4]. Hence, there is an indication of weak absence of peaks that happened at 1100°C that shows 97.68 cm^{-1} at 97.68% which shows double bond between two carbon atoms. For 1000°C, a peak show at 1053.11 cm^{-1} (98.12%).

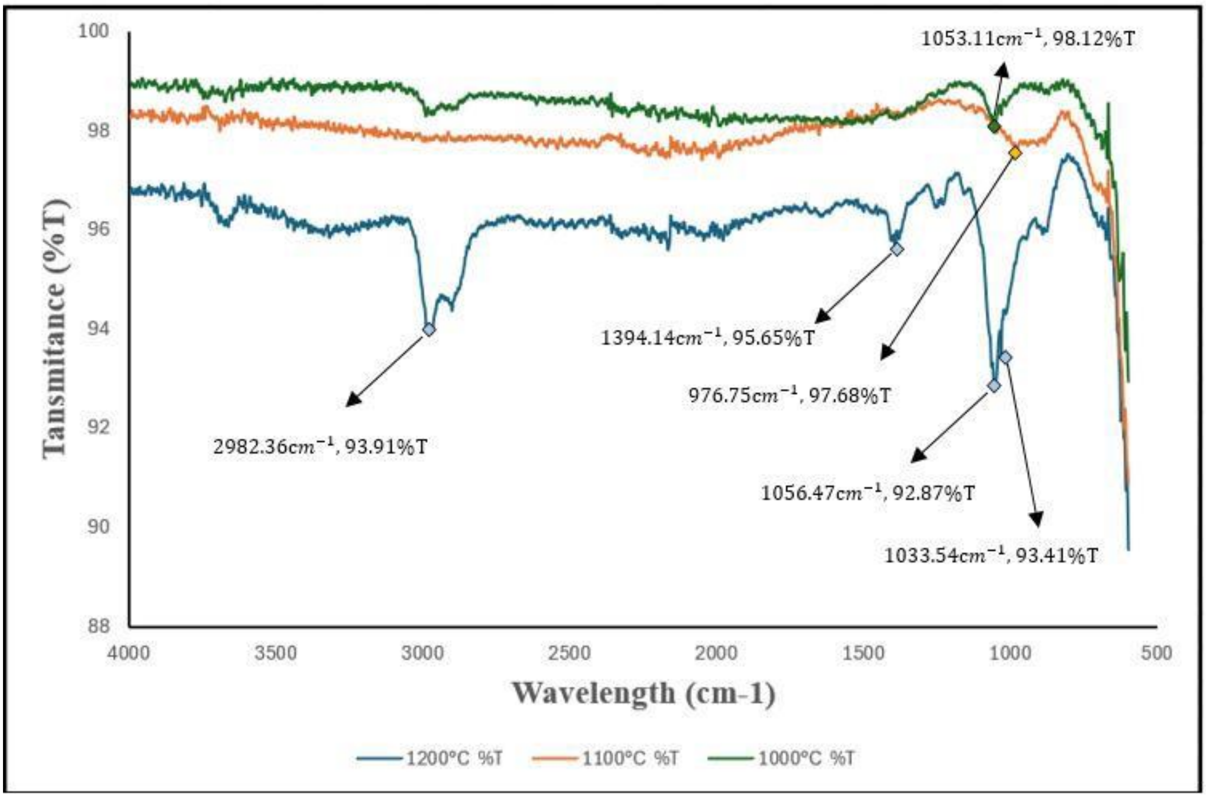


Fig. 4 *FTIR analysis of 99% SDC+ 1%RHS*

The FTIR spectra of the SDC-RHS composite, with a 98:2 ratio as shown in the Figure 3.2 was evaluated to determine the functional groups present in the material. At 1200°C, the C-H asymmetric stretching frequency was detected at 2968.33 cm^{-1}, with a percentage intensity of 96.11%. likewise, at 1100°C, the frequency was 1046.29 cm^{-1} with a percentage intensity of 96.96%. At 1000°C, the spectra revealed absorption peaks at 1058.73 cm^{-1} , with a percentage intensity of 93.80%. These findings are consistent with prior research showing the existence of several functional groups in SDC- RHS composites. Furthermore, a published study found that adding rice husk silica to SDC increased its electrochemical performance in solid oxide fuel cells [5].

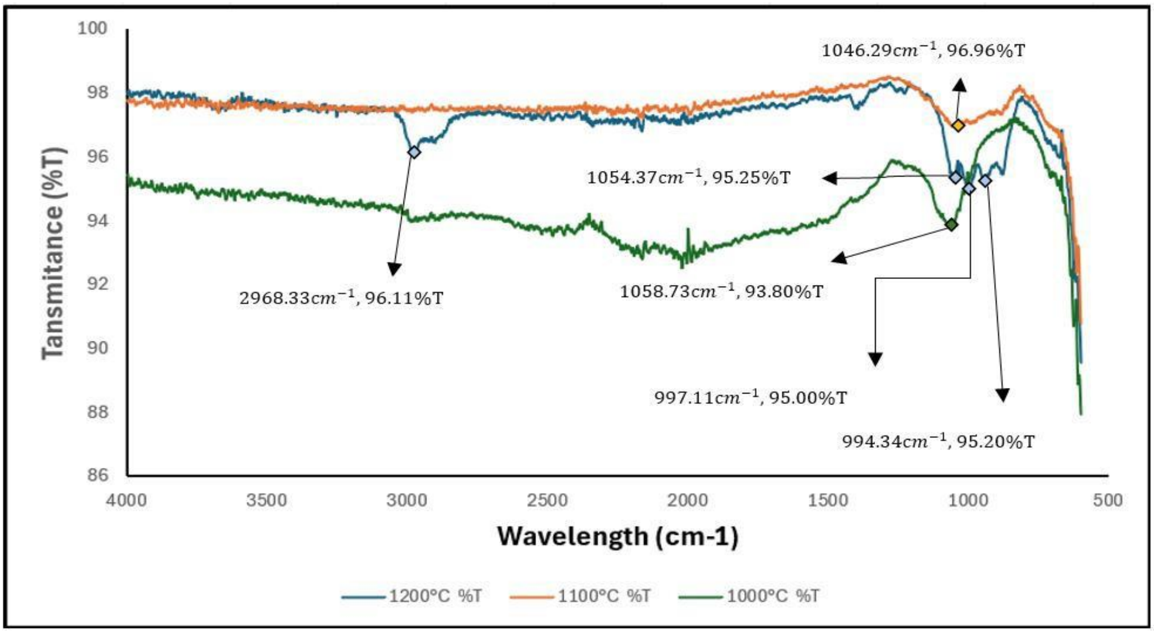


Fig. 5 *FTIR analysis of 98% SDC+ 2%RHS*

The FTIR spectra of the SDC-RHS composite, with a ratio of 97:3, were used to get the results as shown in Figure 3.3 is to determine the functional groups present in the material. At 1200°C, the spectra indicated a lack of peaks, with a peak at 943.27 cm^{-1} at 98.56%, showing the presence of a double bond between two carbons. At 1100°C, the spectra indicated absorption peaks at 1056.89 cm^{-1}, with a percentage intensity of 96.42%. At 1000°C, the spectra indicated absorption peaks at 1070.89 cm^{-1}, with a percentage intensity of 95.80%.

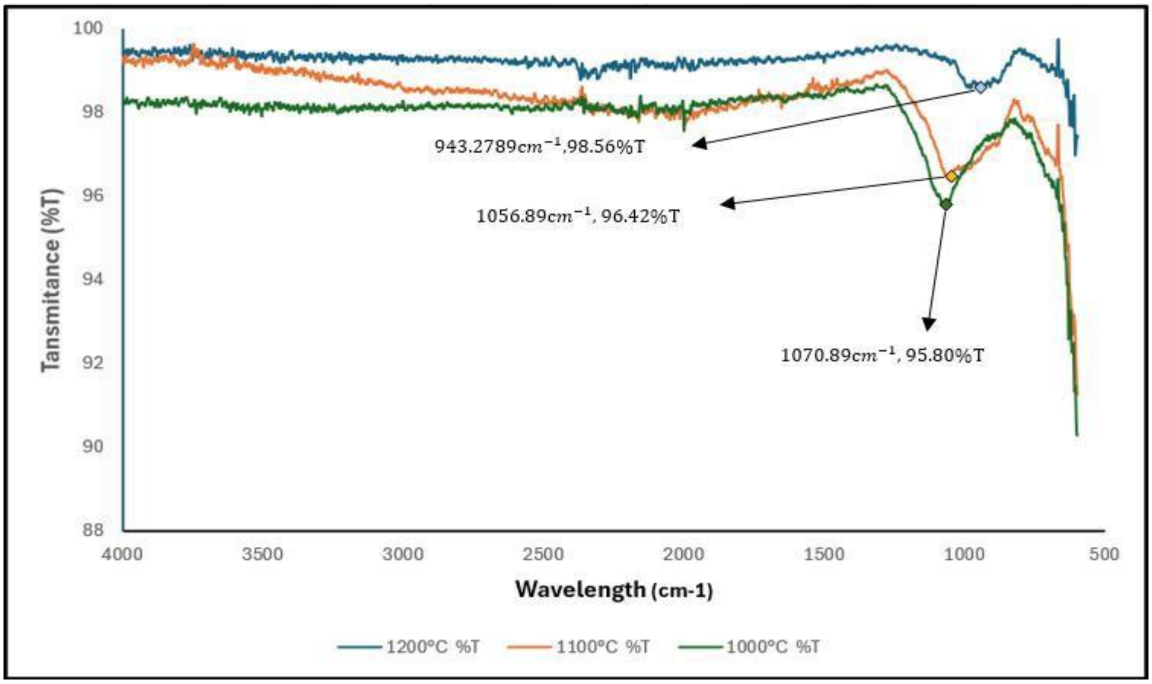


Fig. 6 *FTIR analysis of 97% SDC+ 3%RHS*

3.2 Microstructure Analysis SDC-RHS (SEM)

Tables 4, 5 and 6 display the SEM images of composite electrolyte pallets at magnifications of x1000 and x7000. It is often more vividly created on sloped or side-facing surfaces than perforation, which is black in colour. Tables 4, 5 and 6 show the porosity on surfaces that are more porous. The increased porosity of SDC and RHS composites can be attributed to their larger and more frequent pores.

Table 4 *Results of SEM Illustration SDC-RHS (99:1)*

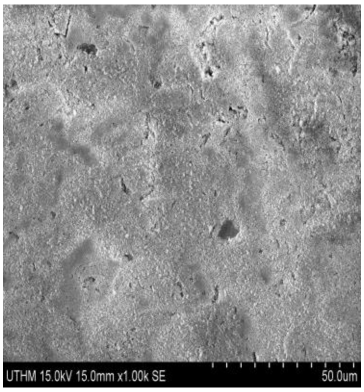
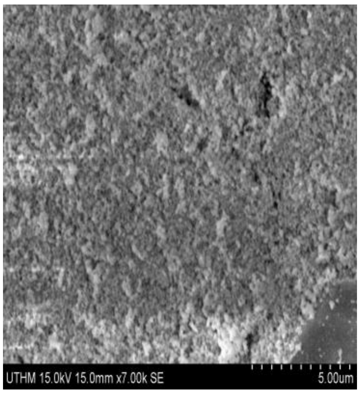
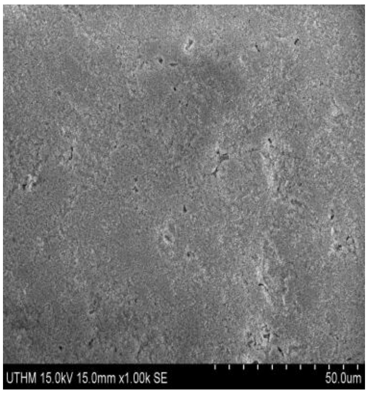
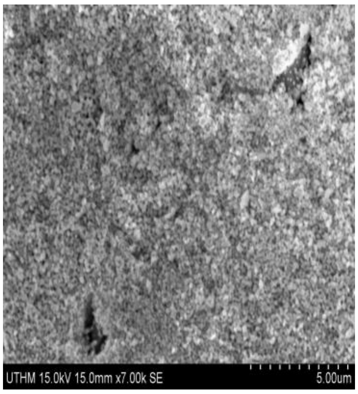
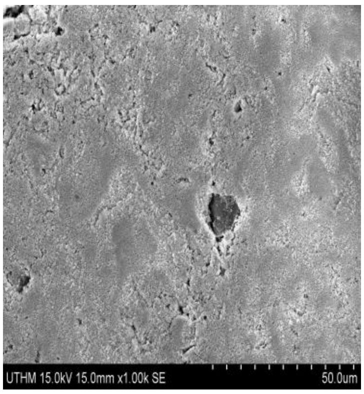
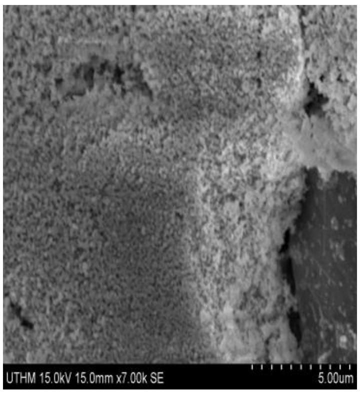
Temperature	X1000 Magnification	x7000 Magnification	Remarks
1000 °C			• Pores are not visible. • Closed pores. • Some cracks were detected. • Porosity increased.
1100 °C			• Pores hardly can be seen. • Closed pores. • A few cracks were detected. • A decrease in porosity.
1200 °C			• Pores can be seen. • Open pores. • A few cracks also detected. • Low chance of porosity.

Table 5 *Results of SEM Illustration SDC-RHS (98:2)*

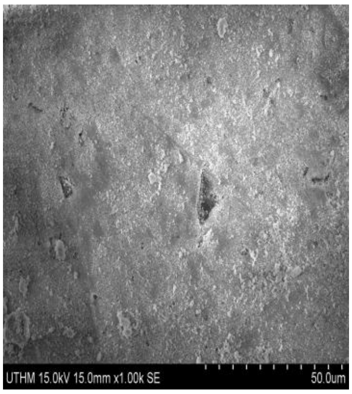
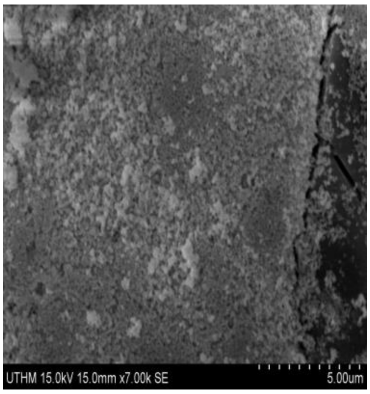
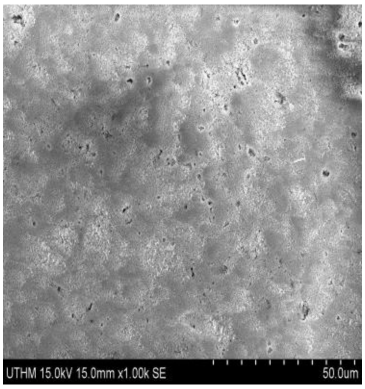
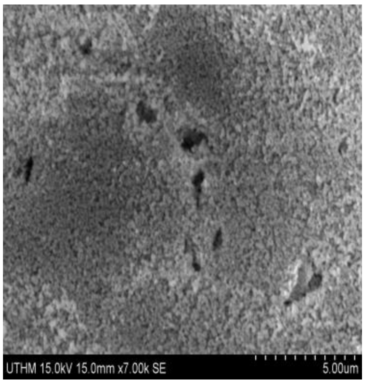
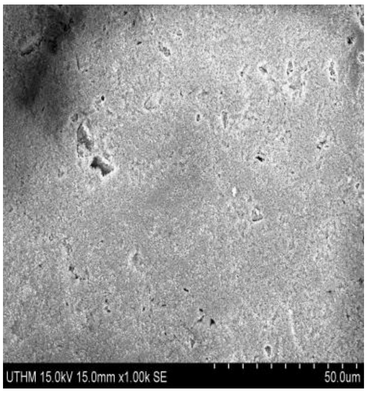
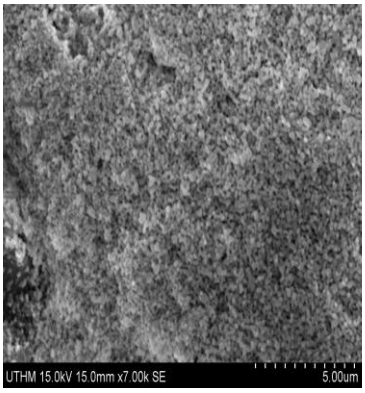
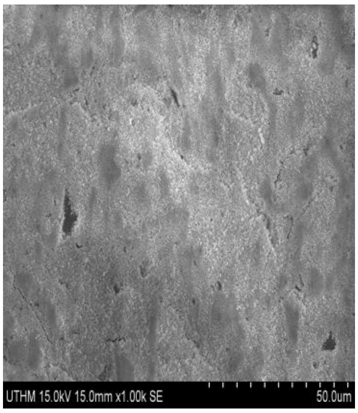
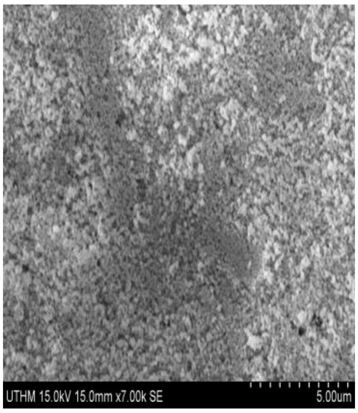
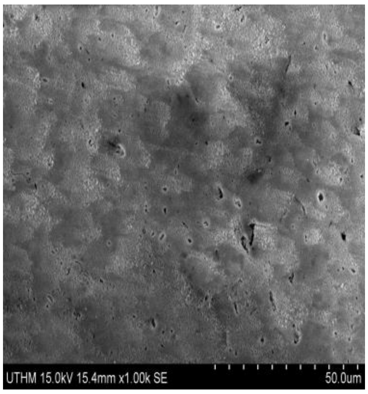
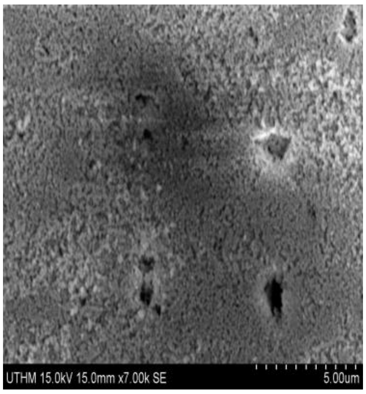
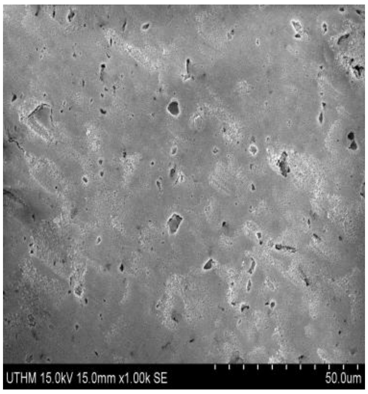
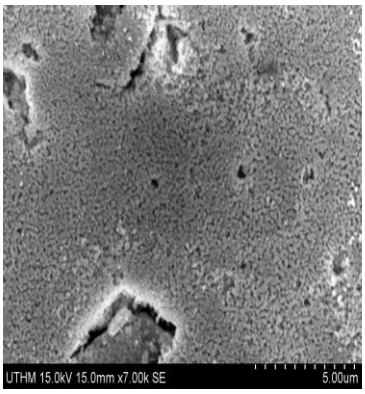
Temperature	X1000 Magnification	x7000 Magnification	Remarks
1000 °C			• Pores are slightly visible. • Open pores. • Porosity decreased.
1100 °C			• Pores can be seen. • Open pores. • A few of cracks was detected. • Low porosity.
1200 °C			• Pores can be seen. • Opened pores. • A few cracks also detected. • Low porosity.

Table 6 *Results of SEM Illustration SDC-RHS (97:3)*

Temperature	X1000 Magnification	x7000 Magnification	Remarks
1000 °C			• Pores are visible. • Open pores. • A crack was detected. • A significant decrease in porosity.
1100 °C			• Pores can be seen. • Open pores. • No cracks were detected. • Low chance of porosity.
1200 °C			• Pores can be seen very clearly. • Open pores. • Some cracks were also detected. • Low chance of porosity.

As stated, two magnifications have been applied for characterization: x1000 and x7000. The RHS distribution on the SDC structure was seen at an accelerating voltage of 15.0kV. The SEM photos show the surface morphology of SDC, RHS, and the composition of SDC-RHS. The distribution, amorphous physical structure, and morphology of SDC-RHS at different ratios and temperatures can be investigated by SEM, as shown in Tables 4, 5 and 6. Porosity is one method for determining the ideal sintering temperature for a Samarium Doped Ceria (SDC)--based Solid Oxide Fuel Cell (SOFC) electrolyte made from Rice Husk Silica. Based on the previous research, the material's porosity should be tuned for the most effective ionic conductivity and mechanical stability [6].

When the sintering temperature was 1000 °C at a ratio of 99:1 (SDC-RHS), the SDC-RHS particles were tightly bound together, even though some cracks were found, the pores were not visible, and the pore wall had numerous micropores. The pores are slightly visible with open pores, possibly decreasing porosity at a ratio of 98:2 (SDC-RHS).

At the ratio of 97:3 (SDC-RHS), the pores are opened pores, and a crack was detected and showed a significant decrease in porosity.

At 1100°C ratio of 99:1 (SDC-RHS), pores can hardly be seen, closed pores and few of cracks was detected with an increment of porosity can be happened too. Abundant pores and a few observed cracks indicate a low porosity ratio of 98:2 (SDC-RHS). At a ratio of 97:3 (SDC-RHS), pores are visible and open, with no detected cracks and a low chance of porosity.

When the sintering temperature was increased to 1200 °C, the microstructure of SDC-RHS exhibits very clear and open pores, and numerous cracks at ratio of 99:1(SDC-RHS). Pores are noticeable at a 98:2 (SDC-RHS) ratio, with some open pores and a few cracks found, indicating a low probability of porosity while for ratio of 97:3(SDC-RHS), the microstructure shows very clear and open pores, some detected cracks, and low chance of porosity due to the increased amount of silica added compared to other ratios.

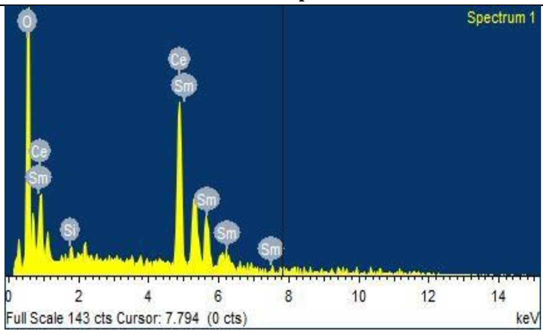
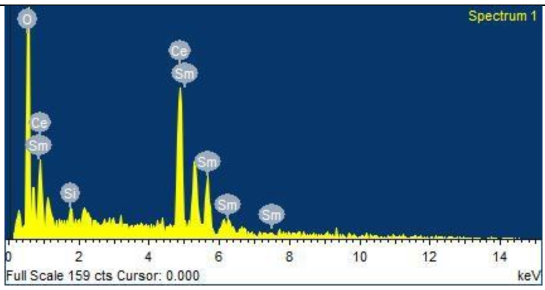
As for that, the suitable ratio and temperature for decrement of sintering temperature for SDC doping with RHS are at 1000 °C for ratio 97:3(SDC-RHS) and (SDC-RHS) as 1000 °C is the lowest temperature between 1100 °C and 1200 °C. Thus, the microstructure shows very clear and open pores, a few detected cracks, and low chance of porosity due to the increased amount of silica added compared to other ratios.

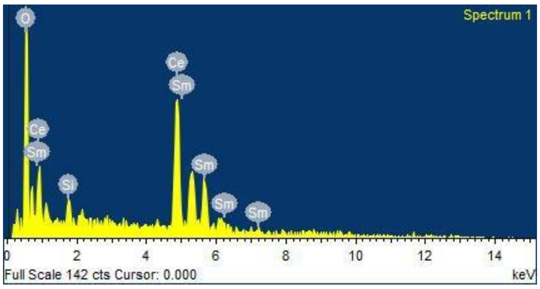
3.3 Element Analysis Composition of SDC-RHS (EDS)

The theoretical behind EDS is based on a comprehension of atomic structure, X-ray emission and the link between atomic number and energy of X-ray lines. This enables the identification and measurement of components in a sample.

Table 7 illustrates the elemental compositions defined from the Elementary Analysis Composition of SDC-RHS. The results obtained show the presence of samarium (Sm), cerium (Ce), silica (Si), and oxygen (O). For reducing the sintering temperature of samarium-doped ceria (SDC), it is necessary to maintain a steady high content of cerium (Ce) and samarium (Sm) while keeping impressive material properties at low temperatures. This approach not only preserves the material's ideal qualities, but it also significantly decreases the energy required for the sintering process. The presence of silica (Si) and oxygen (O) contributes to the material's performance under these changing conditions, guaranteeing that the SDC-RHS composition maintains potent and effective at lower sintering temperatures.

Table 7 *Results Percentage Molecular Composition of SDC-RHS (99:1)*

<table>
<tr><th>Temperature</th><th>Molecular Composition</th><th>Atomic Percentage</th></tr>
<tr><td>1000 °C</td><td>
</td><td>
<table>
<tr><th>Element</th><th>Weight%</th><th>Atomic%</th></tr>
<tr><td>O K</td><td>21.01</td><td>69.44</td></tr>
<tr><td>Si K</td><td>0.71</td><td>1.34</td></tr>
<tr><td>Ce L</td><td>65.59</td><td>24.75</td></tr>
<tr><td>Sm L</td><td>12.69</td><td>4.46</td></tr>
<tr><td>Totals</td><td>100.00</td><td></td></tr>
</table></td></tr>
<tr><td>1100 °C</td><td>
</td><td>
<table>
<tr><th>Element</th><th>Weight%</th><th>Atomic%</th></tr>
<tr><td>O K</td><td>20.92</td><td>68.96</td></tr>
<tr><td>Si K</td><td>1.07</td><td>2.01</td></tr>
<tr><td>Ce L</td><td>65.15</td><td>24.52</td></tr>
<tr><td>Sm L</td><td>12.86</td><td>4.51</td></tr>
<tr><td>Totals</td><td>100.00</td><td></td></tr>
</table></td></tr>
</table>

1200 °C		Element / Weight% / Atomic% O K 19.92 67.03 Si K 1.70 3.25 Ce L 62.84 24.15 Sm L 15.54 5.57 Totals 100.00

Rare earth elements such as samarium (Sm) and cerium (Ce) were recently researched for their ability to improve SOFC performance. Such elements can increase the conductivity and stability of the electrolyte, resulting in improved overall performance. Based on the previous study, rare elements like Samarium (Sm) and Cerium (Ce) have been widely employed in solid oxide fuel cells (SOFCs) due to their distinct features, including as high ionic and electronic conductivity, which are required for effective energy conversion. These elements are especially beneficial in the production of SOFC components, including electrolytes, anode and cathode [7].

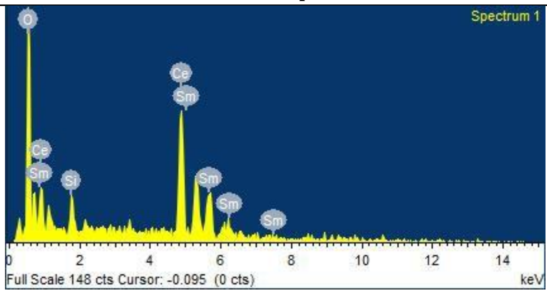
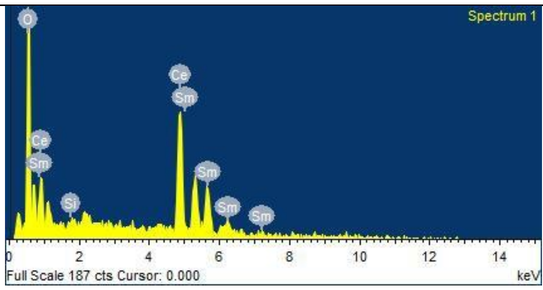
Based on EDS result in Table 7, for the weight percentage of SDC-RHS (99:1), the highest value of Sm -L element exist are at temperature of 1100 °C which is 12.86(weight%) with 4.51(atomic%) while the value of Ce-L is 65.15(weight%) with 24.52(atomic%). However, for temperature of 1000 °C, the element of Ce-L is the highest which is 65.59(weight%) with 24.75(atomic%).

Based on the results in Table 8, the highest value of Sm -L element exists at temperature of 1100 °C which is 14.74(weight%) with 4.93(atomic%) while the value of Ce-L is 65.15(weight%) with 24.52(atomic%) for composition of SDC-RHS (98:2). However, for temperature of 1000 °C, the element of Ce-L is the highest which is 63.72(weight%) with 22.89(atomic%).

According to the results in Table 9, for the composition of SDC-RHS (97:3), the highest value of Sm -L element exists at temperature of 1100 °C which is 14.37(weight%) with 4.79(atomic%) while the value of Ce-L is 62.18(weight%) with 22.22(atomic%). However, for temperature of 1200 °C, the element of Ce-L is the highest which is 65.57(weight%) with 24.14(atomic%).

In terms of enhancing high conductivity and stability in SOFC, cerium (Ce) is an important element needed in electrolyte materials. Hence, the suitable temperature for decreasing sintering temperature of SOFC are at 1000 °C and 1200 °C.

Table 8 *Results Percentage Molecular Composition of SDC-RHS (98:2)*

Temperature	Molecular Composition	Percentage Atomic
1000 °C		Element / Weight% / Atomic% O K 22.05 69.38 Si K 2.03 3.65 Ce L 63.72 22.89 Sm L 12.20 4.08 Totals 100.00
1100 °C		Element / Weight% / Atomic% O K 23.01 72.34 Si K 0.27 0.48 Ce L 61.97 22.24 Sm L 14.74 4.93 Totals 100.00

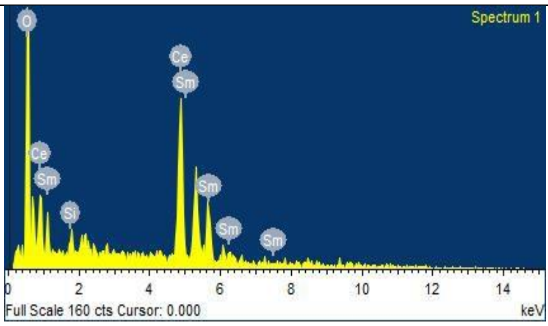
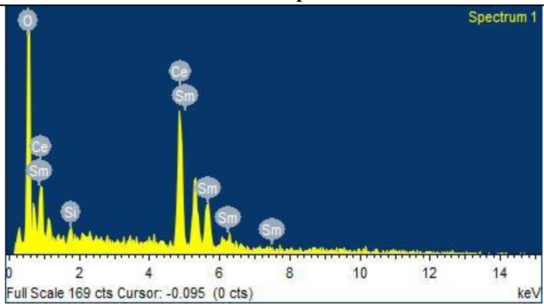
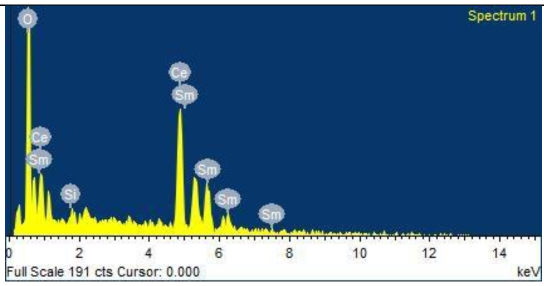
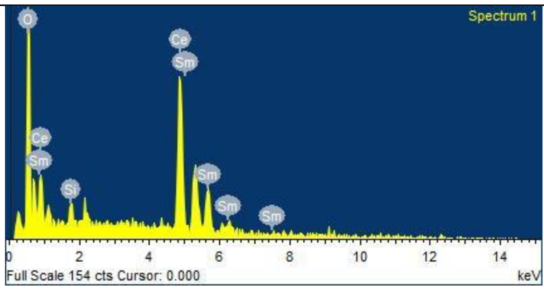
1200 °C		Element / Weight% / Atomic% O K 21.12 69.13 Si K 1.16 2.16 Ce L 64.70 24.18 Sm L 13.02 4.53 Totals 100.00

Table 9 Results Percentage Molecular Composition of SDC-RHS (97:3)

Temperature	Molecular Composition	Percentage Atomic
1000 °C		Element / Weight% / Atomic% O K 22.25 71.23 Si K 0.47 0.85 Ce L 64.12 23.44 Sm L 13.16 4.48 Totals 100.00
1100 °C		Element / Weight% / Atomic% O K 23.15 72.45 Si K 0.30 0.54 Ce L 62.18 22.22 Sm L 14.37 4.79 Totals 100.00
1200 °C		Element / Weight% / Atomic% O K 21.52 69.39 Si K 1.36 2.50 Ce L 65.57 24.14 Sm L 11.55 3.96 Totals 100.00

5. Conclusion

This research focuses more on the effect of rice husk silica (RHS) on samarium-doped ceria (SDC)- based solid oxide fuel cell (SOFC) electrolytes by implementing RHS, which had an important impact on the electrolytes' sintering temperature and performance. The implementation of RHS was intended to improve thermal stability and conductivity, resulting in better SOFC. Furthermore, the addition of RHS increases the ionic conductivity of the SDC electrolyte by increasing densification and grain development, which is critical for effective SOFC performance. RHS also helped to create a more homogeneous microstructure with fewer flaws, which improved the electrolyte's structural integrity and longevity. Hence, RHS can improve the thermal and electrical properties of the electrolytes. The data collection of sintering, SEM, and FTIR could provide useful information about the chemical bonding and microstructure of SDC-RHS composites and advanced SOFC technology and develop more efficient and competent solid oxide fuel cells for environmentally friendly energy applications.

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Conflict of Interest

The authors declare that there is no conflict of interest regarding the paper's publication.

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