



RESEARCH ARTICLE

IMPACT OF CALCINATION TEMPERATURE ON STRONTIUM CARBONATE FORMATION AND MICROSTRUCTURAL-THERMAL PROPERTIES OF SAMARIUM STRONTIUM COBALT OXIDE-SAMARIUM-DOPED CERIA CARBONATE COMPOSITE CATHODES FOR LOW-TEMPERATURE SOLID OXIDE FUEL CELLS**Siti Ameerah Nadiah Jamal¹, Sufizar Ahmad^{1,*}, Umira Asyikin Yusop², Siti Fairus Mohammad¹, Hamimah Abd Rahman¹, Mohd Azham Azmi¹**¹*Faculty of Mechanical and Manufacturing, Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Batu Pahat, Johor, Malaysia.*²*Solid Oxide Fuel Cell Group, Fuel Cell Institute, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia.*

Abstract. Solid oxide fuel cells (SOFCs) operating at low temperatures require cathodes with tailored microstructural and thermal properties to address performance challenges. The formation of SrCO₃ during the calcination process significantly influences the properties of the SSC-SDCC composite cathode for LT-SOFCs. This study systematically investigates the effect of calcination temperature (600 – 750 °C) on SrCO₃ formation and its correlation with the microstructural and thermal properties of SSC-SDCC (60:40 wt.%) composite cathodes. The cathode powders were mixed through the high-energy ball milling (HEBM) method and calcined at four different temperatures (600, 650, 700, and 750 °C). The pellet cathodes were fabricated using the uniaxial pressing method and sintered at 600 °C. Comprehensive characterizations using XRD revealed that calcination temperatures ≥ 700 °C effectively minimized SrCO₃ formation while maintaining phase purity. FESEM analysis revealed that all samples exhibited particle agglomeration, consistent with calcination powder theory, where particle bonding intensifies with increasing temperature. Porosity analysis reveals optimal cathode functionality with measured values of 35.91 – 38.78 %, residing within the established 20 – 40 % target range for effective gas diffusion while maintaining structural stability. The thermal expansion coefficients ($15.1 - 16.00 \times 10^{-6} \text{ K}^{-1}$) align well with ceria electrolytes ($11.1 \times 10^{-6} \text{ K}^{-1}$), exhibiting comparable thermal behavior while retaining beneficial SrCO₃ phases that enable functional compatibility. This systematic variation provides valuable insights for interface engineering in real-world SOFC operating conditions for the development of efficient and durable LT-SOFC systems for clean energy applications.

Keywords: Ceria-based electrolyte, calcination, LT-SOFC, SSC-SDCC cathode, SrCO₃.**Article Info**

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1. INTRODUCTION

Fuel cell technology has emerged as a pivotal solution in the global clean energy transition, offering high efficiency and minimal environmental impact. In Malaysia, growing interest in fuel cells research aligns with national energy transition goals, particularly in distributed power systems and renewable energy integration [1]. Among the various fuel cell systems, solid oxide fuel cell (SOFCs) is particularly stand out due to their fuel flexibility and high energy conversion efficiency [2]. However, conventional SOFCs (high operating temperatures) present challenges in material stability and system longevity [3]. To mitigate these limitations, significant efforts have been shifted toward material development for low-temperature SOFCs (LT-SOFCs, < 600 °C), where cathode performance critically determines overall efficiency. In this context, $\text{Sm}_{0.5}\text{Sr}_{0.5}\text{CoO}_{3-\delta}$ (SSC)-based composite cathodes have gained considerable attention as promising cathode materials due to their exceptional mixed ionic-electronic conductivity (MIEC) and high catalytic activity toward the oxygen reduction reaction (ORR) [4,5].

These advantages make SSC a leading candidate for low temperature SOFCs; however, its performance is strongly influenced by factors such as sintering temperature, operating conditions, microstructure (particle size, porosity), and thermal expansion mismatch with adjacent cell components. A significant challenge lies in the formation of secondary phases like strontium carbonate (SrCO_3 , strontianite) and strontium cobalt carbonate (SrCoCO_3), which arise from carbon dioxide (CO_2) in air during cell fabrication and operation, compromising long-term stability and electrochemical activity [6]. While SrCO_3 formation can degrade conductivity and increase polarization resistance, recent studies suggests that its controlled incorporation may paradoxically enhance ORR kinetics by improving the surface exchange coefficient, particularly at low temperatures [6,7]. Thus, optimizing SSC-based cathodes require a delicate balance that could suppressing detrimental phase segregation while harnessing beneficial interface properties, to achieve both durability and high catalytic performance [7].

The integration of electrolyte material, samarium-doped cerium carbonate (SDCC) with SSC-based cathode presents an encouraging strategy to overcome the challenges of LT-SOFCs. While SSC exhibits high catalytic activity for the ORR, its large thermal expansion coefficient (TEC) and susceptibility to secondary phase formation (example; SrCO_3) at elevated temperatures limit its stability [8,9]. By incorporating SDCC, a composite electrolyte with superior ionic conductivity, mechanical stability, and porosity, the SSC-SDCC cathode enhanced compatibility, enabling efficient operation at low temperature region. The carbonate phase in SDCC not only improves interfacial conductivity but also mitigates detrimental phase segregation during calcination and sintering, preserving the functional binary carbonate structure without major decomposition [10]. Moreover, SDCC's economical processing and tailored thermal expansion properties help counteract SSC's high TEC, minimizing delamination risks [11]. Optimizing the calcination temperature is critical to maintaining the composites particle size, electrochemical properties, and phase purity, ensuring long-term performance. Thus, the SSC-SDCC system address key limitations of conventional SOFC cathodes, offering a viable pathway for high performance, low temperature operation with improved stability and reduced degradation.

This study systematically examines the influence of controlled calcination temperature on the microstructural stability and thermal properties of SSC-SDCC composite cathodes, with the objective of establishing optimal processing parameters for durable, high-performance LT-SOFC applications. Through comprehensive characterization of the calcination induced phase evolution, microstructural development, and thermomechanical properties, this research seeks to elucidate the critical relationship between processing conditions and functional performance, thereby contributing to the advancement of practical SSC-SDCC cathodes systems for next-generation energy conversion technologies.

2. MATERIALS AND METHODS

The samarium strontium cobalt oxide–samarium doped ceria carbonate (SSC–SDCC) composite powders were prepared using a ball milling technique, with distinct milling conditions for the electrolyte and cathode materials. The SDCC electrolyte powder was prepared by mixing 80 wt.% SDC and 20 wt.% binary carbonates ($\text{Li}_2\text{CO}_3\text{:Na}_2\text{CO}_3$ in a molar ratio of 67:33), followed by wet ball milling at low–energy conditions with speed of 150 rpm for 24 hours. The resulting mixture was then calcined at 680 °C for 1 hour to form the SDCC electrolyte powder. For the SSC–SDCC composite cathode, a 60:40 weight ratio of commercial SSC (Kceracell, Korea) and as–prepared SDCC powder was homogenized via high–energy ball milling (HEBM) to ensure optimal particle refinement and dispersion. To investigate the influence of calcination temperature on powder characteristics, the composite cathode was subjected to thermal treatment at four different temperatures ranging from 600 °C to 750 °C. The calcined SSC–SDCC composite cathode powder were then being prepared into pellets via dry uniaxial pressing method and subsequently sintered at 600 °C. The composite powder was characterized for phase composition, and chemical compatibility using X–ray diffraction (XRD, D8 Advanced Bruker diffractometer, Germany), while its morphology was examined via Field emission scanning electron microscopy (FESEM, JEOL, JSM–7600F, Japan). Meanwhile, the composite pellet was analyzed for porosity density using Archimedes principle (Density Kit AY220, Shimadzu, Japan). Additionally, Thermal expansion coefficient (TEC) behavior was evaluated using dilatometer (L75H Linseis, Germany) to assess the materials stability under operational conditions. This systematic approach ensures a comprehensive understating of the structural and thermal properties of the SSC–SDCC composite cathode for potential SOFC applications.

3. RESULTS AND DISCUSSION

This section presents the findings from various analytical technique, including XRD, FESEM, porosity density and TEC. Each subsection provides a detailed analysis of the results, correlating parameters, such as calcination temperature with the observed material properties. The discussion aims to elucidate the optimal conditions that enhance the properties and stability of the SSC–SDCC composite cathode.

3.1 Phase Identification and Chemical Compatibility

The phase composition of the composite cathode powders was determined using XRD analysis performed with Cu $K\alpha$ radiation at $\lambda = 1.5406 \text{ \AA}$. The examination covered crystalline phases in raw commercial powders (SSC, SDC, Li_2CO_3 and Na_2CO_3) as well as the SSC–SDCC composite cathode powders. The diffraction patterns collected over a 2θ range of 10° to 80° at a scan speed of 0.5s per step and a step size of 0.02°. The acquired XRD data were analyzed using Eva diffract plus software to identify phase structures and assess crystallographic properties. Figures 1 and 2 display the results obtain from the XRD analysis.

Figure 1 presents the XRD spectra of commercial SSC, as–prepared SDCC, and SSC–SDCC composite cathode powders. The XRD spectra revealed characteristic peaks corresponding to the orthorhombic perovskite structure of SSC (JCPDS 53–0112, space group Pnma), while SDCC exhibited a face–centered cubic fluorite structure (JCPDS 75–0158, space group $*Fm\text{--}3m$). Notably, the carbonate phase ($\text{Li}_2\text{CO}_3\text{--Na}_2\text{CO}_3$) remained amorphous, as no distinct peaks were observed, indicating its presence as a surface layer on SDC particles without disrupting the host lattice. Furthermore, the composite SSC–SDCC pattern showed no secondary phases, confirming chemical compatibility between SSC and SDCC after the milling process. These results demonstrate the successful integration of both phases while preserving their structural integrity.

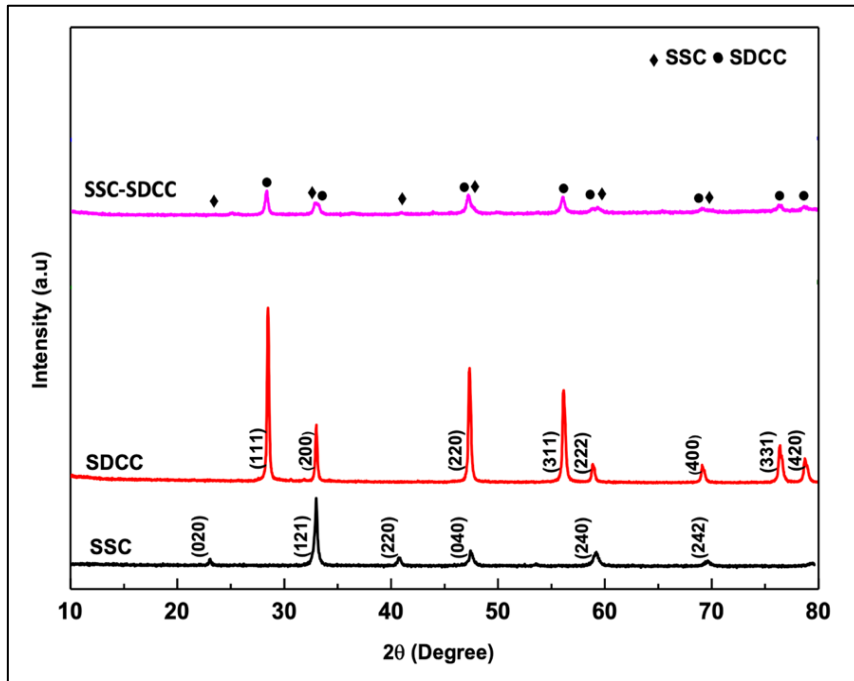


Figure 1: X-ray diffractograms for commercial SSC, as-prepared SDCC and SSC-SDCC composite cathode powder

Figure 2 displays the XRD diffractograms of SSC-SDCC composite cathode powders calcined at temperatures ranging from 600 °C to 750 °C. The analysis revealed the emergence of a secondary phase, strontium carbonate (SrCO_3), identifiable by peaks appearing between $2\theta = 25^\circ$ and 50° . The SrCO_3 peaks intensity exhibits a temperature-dependent trend, increasing from 600 °C to a maximum at 700 °C, followed by a reduction at 750 °C.

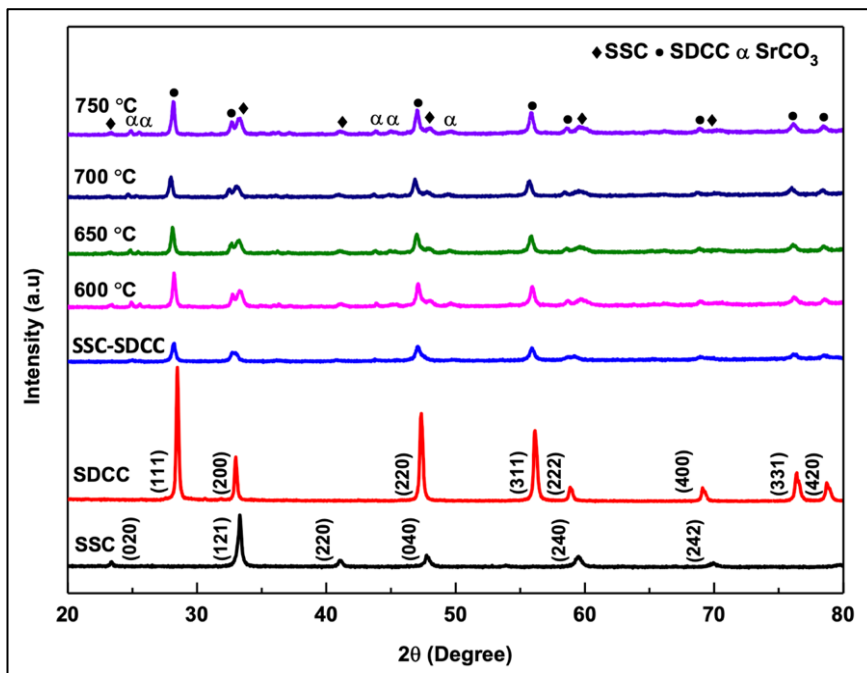


Figure 2: X-ray diffractograms of SSC-SDCC composite cathode powders after calcination process at variable temperature (600 – 750 °C)

Notably, the weakest SrCO_3 peak was observed at 750°C , indicating phase evolution with increasing temperature. The formation of SrCO_3 is attributed to the chemical reaction between strontium (Sr) from the SSC perovskite and carbonate (CO_3^{2-}) derived from $(\text{Li}/\text{Na})_2\text{CO}_3$ electrolyte [12]. This phenomenon aligns with prior studies reporting SrCO_3 formation in SSC-based materials under similar calcination conditions ($600 - 800^\circ\text{C}$ in static air) [13]. Despite the presence of SrCO_3 , the primary crystal structure of SSC (orthorhombic perovskite) and SDC (fluorite cubic) remained intact, as evidenced by the unaltered peak positions and patterns in the XRD spectra. This confirms the robust chemical computability between SSC and SDCC, even under elevated temperatures. Interestingly, studies suggest that trace amounts of SrCO_3 may enhance the cell performance of LT-SOFC by improving ionic conductivity [14]. Thus, while SrCO_3 formation indicates partial phase instability, its limited presence does not compromise the composites structural integrity and may even contribute beneficially to its functional properties [6]. These findings underscore the importance of optimizing calcination conditions to balance phase purity and performance in SSC-SDCC cathodes.

3.2 Morphology

The microstructure and surface morphology of the SSC-SDCC composite cathode powders were examined using FESEM. Images were captured at different stages of the investigation to observe morphological evolution, with a standard magnification of 50,000x to access particle distribution and structural features. This analysis provides critical insights into the powder's physical characteristics and their correlation with processing conditions. Figure 3 presents FESEM micrographs of involve material at the beginning stage of SSC-SDCC composite cathode powder preparation. Based on the figure, the SSC particles (Figure 3a) exhibit non-uniform sub-micron particle sizes and poor dispersion. In contrast, the calcined SDCC powder (Figure 3b) display equiaxed particle morphology with improved uniformity

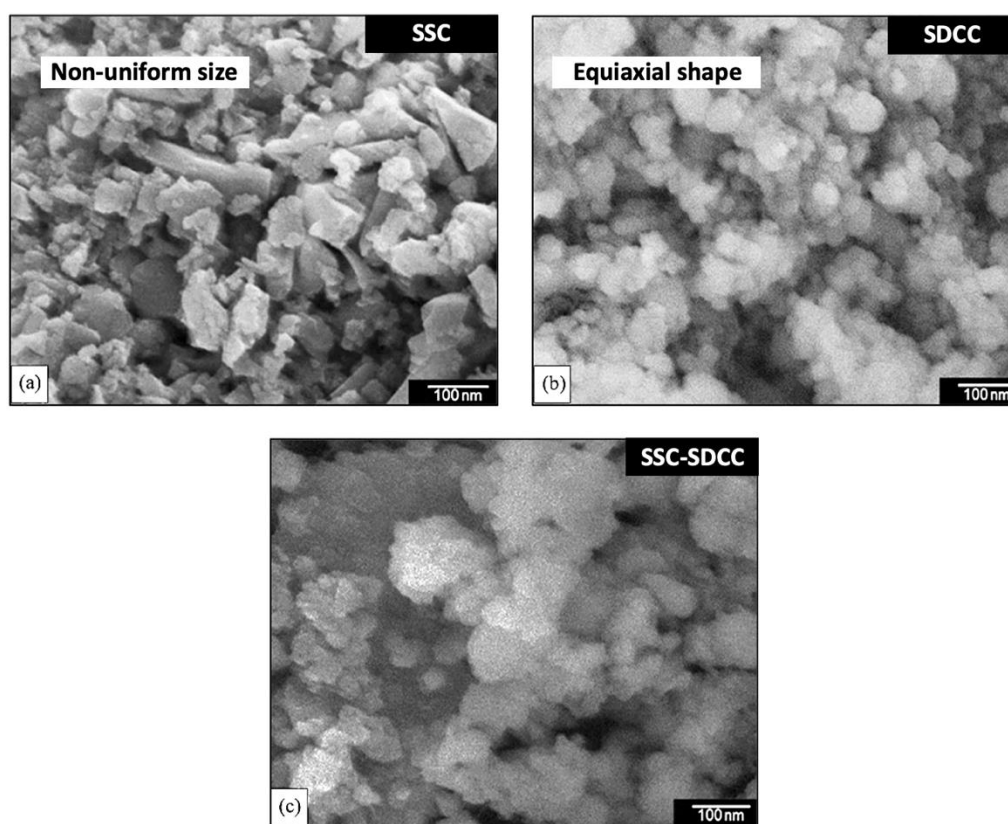


Figure 3: FESEM micrographs of (a) pure SSC raw powder, (b) SDCC composite electrolyte powder and (c) SSC-SDCC composite cathode powder

. The composite SSC–SDCC cathode (Figure 3c) demonstrates homogeneous phase distribution, with no observable agglomeration and uniformly sized particles, indicating effective integration of the two phases through the preparation process. These morphological characteristics correlate well with the enhanced powder processability and suggest favorable microstructural properties for SOFC applications. The well dispersed composite structure is particularly advantageous for facilitating triple–phase boundary (TPB) formation, while the uniform particle size distribution may contribute to improved mechanical stability during cell operation [15].

Figure 4 shows the FESEM micrographs of SSC–SDCC composite cathode powders calcined at temperatures ranging from 600 °C to 750 °C, revealing significant temperature dependent morphological changes. The initial as–prepared powders exhibited progressive agglomeration with increasing calcination temperature, attributable to thermally induced particle bonding and sintering effects [16]. This agglomeration behavior aligns with fundamental calcination theory, where elevated temperature promotes interparticle bonding through enhanced atomic diffusion, ultimately leading to the formation of weakly–bound aggregated [17]. While these agglomerates can be mechanically disrupted prior to the pressing process, their presence demonstrates the temperature sensitive nature of powder consolidation. The observed trend from loose, discrete particles at lower temperatures to compacted agglomerates at higher temperatures, provide critical insight into the processing structure relationship, emphasizing the need for careful temperature control to optimize powder characteristics for subsequent fabrication steps while maintaining desirable microstructural features.

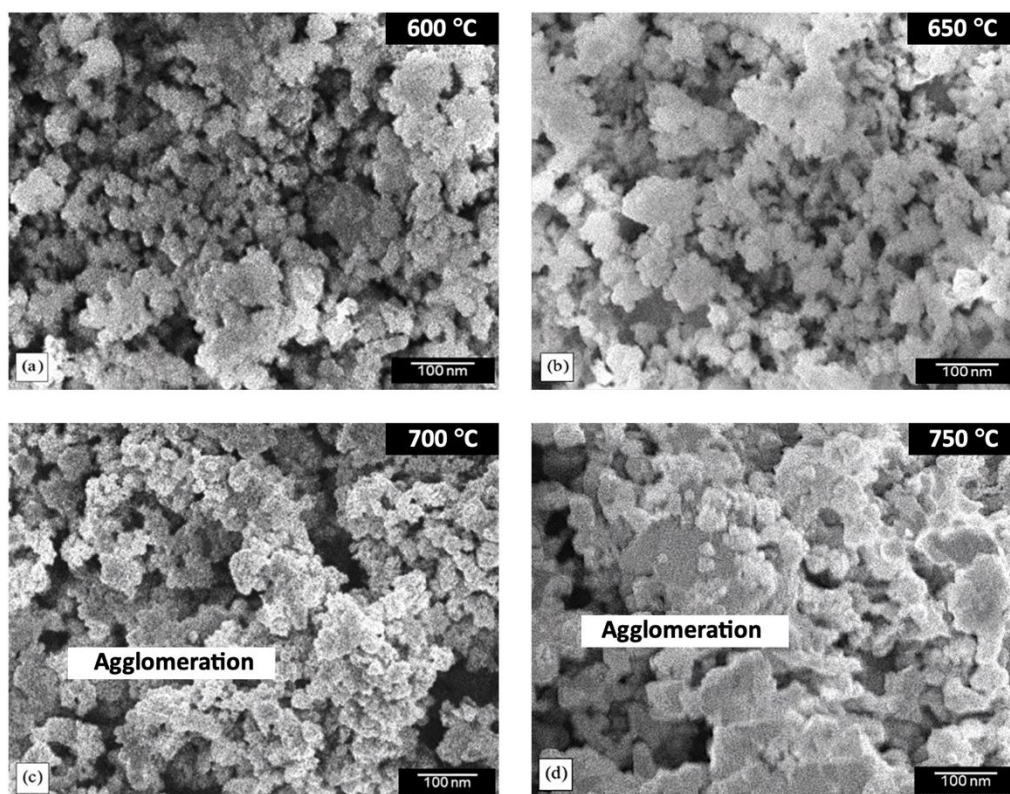


Figure 4: FESEM micrographs of SSC–SDCC composite cathode powder after calcination process at temperature (a) 600 °C, (b) 650 °C, (c) 700 °C and (d) 750 °C

3.3 Porosity and Density

The physical investigation of the SSC–SDCC composite cathode was further examine in the form of pellets sample that focuses on analyzing apparent porosity and density, as well as examining the influence of sintering temperature on these properties. The porosity and density values, as presented

in Table 1, provide essential data for evaluating the structural characteristics of the composite cathode. Additionally, the relationship between these properties and calcination temperature (ranging from 600 °C to 750 °C) is illustrated in Figure 5.

The results demonstrate a clear correlation between calcination temperature and the porosity–density characteristics of the SSC–SDCC composite cathode. Increasing the calcination temperature from 600 °C to 700 °C significantly reduces porosity, as evidenced by the data in Table 1 and Figure 5, indicating improved densification. However, at 750 °C, the porosity increases again to 38.57 %, suggesting a temperature-dependent threshold. This behavior is attributed to microstructural change at elevated temperature, where enhanced grain growth and particle coarsening promote pore coalescence and the formation of larger, interconnected pores. This observation is consistent with the FESEM analysis in Section 3.2, which shows increased particle agglomeration at higher calcination temperature, leading to inefficient particle packing during palletization. A similar trend is observed in density measurements, which reaches a maximum at 700 °C before declining at 750 °C. Additionally, the reduction of secondary phases such as SrCO_3 may alter particle packing and contributed to pore reformation, resulting in increased overall porosity [6,14].

Table 1: The average values of porosity and density of SSC–SDCC composite cathode pellets

SSC–SDCC Composite Cathode pellet	Calcination temperature (°C)			
	600	650	700	750
Porosity (%)	38.78 ± 0.17	38.50 ± 1.10	35.91 ± 0.98	38.57 ± 1.82
Density (gcm^{-3})	5.47 ± 0.35	5.55 ± 0.06	5.62 ± 0.23	5.27 ± 0.28

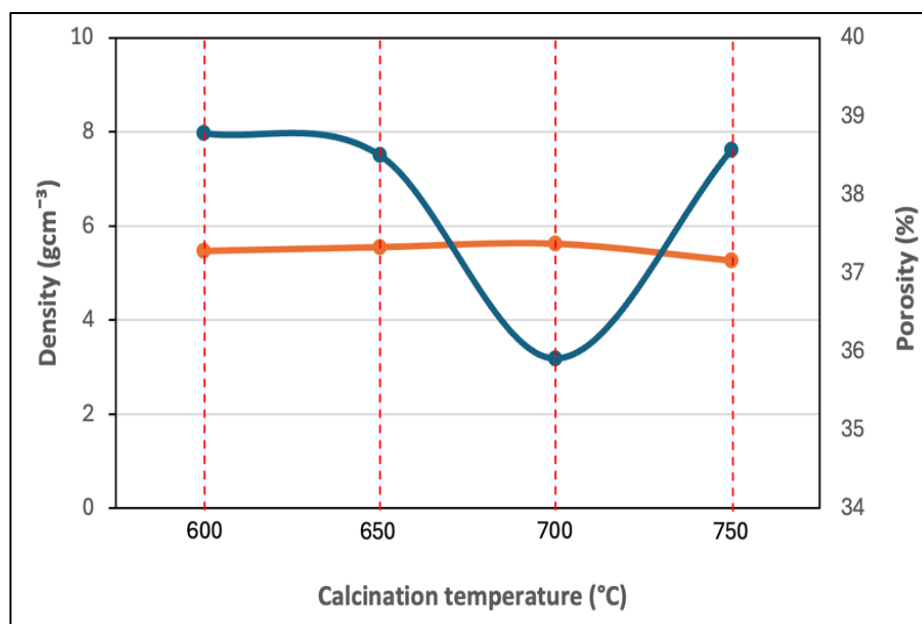


Figure 5: Porosity and density values of calcined SSC–SDCC composite cathode pellet

Further insights from FESEM morphology reveal that rough agglomeration in the powder significantly influence porosity [16]. While pellet pressing reduces some agglomerates, other agglomerates persist contributing to residual porosity. Studies suggest that optimal cathode performance requires porosity values between 20 – 40 %, with some research emphasizing ~30 % for enhanced ionic conductivity [17]. The SSC–SDCC composites exhibit porosities within this range (35.91 – 38.78 %), confirming their suitability as potential cathode materials. These findings align with prior work by Kaur

et al. [17], who noted the limitations of pressing in eliminating hard agglomerated, as well as the broader consensus of porosity requirements for efficient cathode operation. Porosity plays a critical role in SOFC performance, as it directly influences gas diffusion, electrochemical activity and ionic conductivity. Thus, precise microstructure engineering of porosity remains fundamental to advancing high-performance SOFC technologies.

3.4 Thermal Expansion Behavior

Thermodynamic compatibility, particularly thermal expansion matching between cathode and electrolyte, is critical for SOFC durability, as mismatch in TEC can induce interfacial delamination and microcracking during thermal cycling. Such mechanical stresses, arising from differential expansion during heating, degrade cell performance, especially in cobalt-rich cathodes like SSC, which exhibit inherently high TEC values at lower-to-intermediate operating temperatures (400 – 800 °C) [18]. To address this, the TEC behavior of SSC–SDCC composite cathode was systematically evaluated across calcination temperature (600 – 700 °C), with results detailed in Table 2.

Table 2: The thermal expansion behavior of SSC–SDCC composite cathode with variable calcination temperature

Thermal expansion coefficient, TEC (10^{-6} K^{-1}) at operating temperature 30 – 600 °C	Calcination temperature (°C)			
	600	650	700	750
SSC–SDCC Composite Cathode	15.1 ± 0.2	15.3 ± 0.1	15.1 ± 0.2	16.0 ± 0.1
Mismatch (%)	36.0	37.8	36.0	44.1

The SDCC composite electrolyte exhibit an average TEC value of $11.1 \times 10^{-6} \text{ K}^{-1}$, while the pure SSC cathode theoretically demonstrates significantly higher TEC values ranging from 20.0 to $22.0 \times 10^{-6} \text{ K}^{-1}$, revealing a substantial thermal mismatch between these components [19]. However, the SSC–SDCC composite cathode demonstrates a notable improvement in this research, with measured TEC values ranging from 15.1 to $16.0 \times 10^{-6} \text{ K}^{-1}$. This intermediate TEC range indicates that the incorporation of SDCC electrolyte into the SSC cathode matrix effectively mitigates the thermal expansion disparity, thereby reducing the risk of interfacial delamination and microcracking during thermal cycling.

This study reveals that the SSC–SDCC composite cathode calcined at 750 °C exhibits an elevated TEC value ($16.0 \times 10^{-6} \text{ K}^{-1}$), a phenomenon directly correlated with two concurrent microstructural changes: (1) the reduction of SrCO_3 secondary phases as evidenced by XRD analysis, and (2) increased particle agglomeration shown in FESEM micrograph and accompanied by higher porosity (38.57 %). The diminished SrCO_3 content alters the composites intrinsic thermal behavior, while the enhanced agglomeration at higher temperatures exacerbates porosity, collectively amplifying TEC. The interdependent effects demonstrated how secondary phase evolution and particle morphology critically govern thermo-mechanical properties, emphasizing the need for optimized calcination protocols to achieve balance thermal compatibility in SOFC cathode.

The thermal expansion mismatch between the SSC–SDCC composite cathodes and SDCC electrolyte, as quantified in Table 2, reveals significant variability across samples, with percentage differences showing considerable divergence. While thermodynamic compatibility studies suggest that thermal expansion differences should ideally remain below 20 % to ensure stable interfacial integrity, the SSC–SDCC cathodes consistently exhibit higher mismatch values (36.0 – 44.1 %) [20]. This excessive disparity disrupts mechanical compatibility and compromises transient thermal stability during SOFC operation at elevated temperatures, as documented by Zhang et al. [18]. The resultant interfacial thermal stresses accelerate the risk of delamination and microcrack propagation, ultimately

threatening long-term cell performance. Given that the measured thermal expansion mismatch (36.0 – 44.1 %) exceeds the recommended stability threshold (<20 %), targeted mitigation strategies are required. In particular, compositional tuning through optimized SSC-SDCC ratios or incorporation of low-TEC materials, together with advanced processing approaches such as graded cathode structure or optimized low-temperature sintering, is recommended to reduce thermal expansion disparity and enhance interfacial stability during operation. These findings underscore the critical need for further compositional or processing refinements to mitigate thermal expansion gradients and enhance the thermo-mechanical reliability of SSC-SDCC-based SOFC systems.

4. CONCLUSIONS

This study demonstrates that the SSC-SDCC composite cathode exhibits promising structural and microstructural characteristics, as evidenced by phase-pure XRD patterns, well-defined FESEM morphology, and optimized porosity (35.91 – 38.78 %) within the functional range for SOFC applications. The observed reduction of SrCO₃ secondary phases at higher calcination temperatures was found to significantly influence both the porosity and thermal expansion behavior, highlighting the critical role of SrCO₃ in determining material properties. However, despite these favorable characteristics, the TEC mismatch with the SDCC electrolyte (36.0 – 44.1 %) remains a critical challenge, exceeding the recommended threshold (<20 %) and risking interfacial failure under thermal cycling. To advance this material system, future work should prioritize TEC optimization through tailored compositional adjustment (example., graded doping, secondary phase engineering) or advanced sintering protocols to better align with the electrolytes thermo-mechanical properties. Given its excellent phase stability, morphological control, and porosity performance, the SSC-SDCC composite holds significant potential as a cathode material, provided that TEC compatibility is systematically addresses to ensure long-term SOFC durability.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare

Compliance with Ethical Standards

The work is compliant with ethical standards

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