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## Development of Sr, Y, and Zn co-doped Ba<sub>0.3</sub>Ce<sub>0.7</sub>Zr<sub>0.1</sub>O<sub>3-δ</sub> dense electrolyte for intermediate temperature solid oxide fuel cell

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## Development of Sr, Y, and Zn co-doped Ba<sub>0.3</sub>Ce<sub>0.7</sub>Zr<sub>0.1</sub>O<sub>3-δ</sub> dense electrolyte for intermediate temperature solid oxide fuel cell

### Authors

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## ORIGINAL STUDY

# Development of Sr, Y, and Zn Co-doped $\text{Ba}_{0.3}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{O}_{3-\delta}$ Dense Electrolyte for Intermediate Temperature Solid Oxide Fuel Cell

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## Abstract

Polycrystalline perovskite-type  $\text{Ba}_{0.3}\text{Sr}_{0.7}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}$  was synthesized and characterized by modifying two fundamental perovskites of  $\text{BaCeO}_3$  and  $\text{BaZrO}_3$ . Solid state reaction (SSR) method was adopted for synthesizing the material. The traditional techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and electrochemical impedance spectroscopy (EIS) were employed for the characterization. XRD analysis revealed its pure phase with cubic symmetry at 1300 °C sintering temperature. It was found to have high relative density by the Rietveld refinement process. Regarding thermal stability, it remained stable up to 800 °C. This material also posed crack-free surface. However,  $\text{Ba}_{0.3}\text{Sr}_{0.7}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}$  provided relatively low activation energy with conductivity of  $1.056 \times 10^{-4} \text{ Scm}^{-1}$  at 700 °C in air atmosphere. Therefore, the material might be used as a promising electrolyte in intermediate temperature solid oxide fuel cell (IT-SOFC).

**Keywords:** IT-SOFC, Electrolyte, Perovskite, Rietveld refinement, EIS

## 1. Introduction

In the last few decades, solid oxide fuel cell (SOFC) has become one of the most popular electrochemical energy conversion devices in the renewable energy sector. This is because SOFC environmental friendly and produces low emissions [1]. It converts the electrochemical energy into electric current by oxidizing fuel supplied in the anode [2]. Ytria-stabilized zirconia (YSZ) is usually used as electrolyte for the oxide-ion based SOFC (O-SOFC), which operates at high temperatures (800–1000 °C). The high temperatures used in O-SOFC result in cell degradation, a high fabrication cost and so on [3]. But the intermediate

temperature solid oxide fuel cell (IT-SOFC), which normally operates at 600–800 °C [4,5], has some advantages over the O-SOFC. This is because proton-conducting electrolyte is used in IT-SOFC. Although proton-conducting solid oxide fuel cell offers higher efficiency than their oxide ion-conducting counterparts, they typically exhibit less power density and maximum voltage [6]. One of the most effective proton conductors is doped  $\text{BaCeO}_3$ ; however, it lacks stability in the environments containing of  $\text{H}_2\text{O}$  and  $\text{CO}_2$ . This limitation can be addressed through appropriate doping. For instance,  $\text{BaZrO}_3$ —particularly when doped with 10 mole % yttrium—exhibits excellent stability in  $\text{H}_2\text{O}$  and  $\text{CO}_2$  atmospheres, although its proton conductivity is

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lower than that of BaCeO<sub>3</sub>. Thus, the choice of dopant plays a crucial role in determining the material's electrical performance. Owing to their favorable stability and high proton conductivity in humid conditions, both BaCeO<sub>3</sub> and BaZrO<sub>3</sub> have attracted significant interest as base materials for doping [3,7]. Recently, La<sub>0.9-x</sub>Ba<sub>x</sub>Sr<sub>0.1</sub>Ga<sub>0.8</sub>Mg<sub>0.2</sub>O<sub>3-δ</sub> (x = 0–0.08) electrolytes have been synthesized to examine the impact of Ba doping on structural and electrical properties. Doping with 5 mol % Ba improved structural stability, increased free volume by 4.1 %, and enhanced both bulk and grain boundary conductivities [8]. A series of Ba<sub>1-x</sub>Sr<sub>x</sub>Ce<sub>0.7</sub>Zr<sub>0.1</sub>Y<sub>0.15</sub>Zn<sub>0.05</sub>O<sub>3-δ</sub> (x = 0.1, 0.3, 0.5) compositions were studied, combining doped BaCeO<sub>3</sub> and BaZrO<sub>3</sub>. X-ray diffraction showed peak shifts due to reduced unit cell volume with increasing Sr content. BSCZYn (x = 0.1) had the highest ionic conductivity of  $30.3 \times 10^{-4} \text{ Scm}^{-1}$  at 973 K and the lowest activation energy of 0.381 eV [9]. Ba-based proton-conducting BaCe<sub>0.5</sub>Zr<sub>0.3</sub>Y<sub>0.1</sub>A<sub>0.05</sub>Zn<sub>0.05</sub>O<sub>3-δ</sub> (A = Gd, Sm) electrolytes were synthesized for IT-SOFC applications. Electrochemical analysis showed that BCZYGdZn had significantly higher ionic conductivity than BCZYSmZn, with  $12.9 \times 10^{-4} \text{ Scm}^{-1}$  in wet H<sub>2</sub> at 973 K and an activation energy of 0.38 eV. Both materials showed potential as electrolytes for IT-SOFC [10].

A study [11] introduces Ba diffusion sintering as an effective method to improve the densification and microstructure of BaZr<sub>0.8</sub>Yb<sub>0.2</sub>O<sub>3-δ</sub> electrolyte for proton-conducting fuel cells. The incorporation of BaCO<sub>3</sub> at the anode, coupled with optimized sintering processes, results in improved grain growth and a uniform microstructure, effectively doubling the proton conductivity. An investigation [12] addresses the high sintering temperature requirement of the proton-conducting electrolyte BaZr<sub>0.4</sub>Ce<sub>0.4</sub>Y<sub>0.1</sub>Yb<sub>0.1</sub>O<sub>2.9</sub> (BZCYb4411) by exploring three strategies to reduce it to 1400 °C: (1) ZnO addition, (2) Zn doping, and (3) fluorite-phase ZCYb4411 addition. Zn-based methods proved most effective, achieving >90 % relative density and eliminating the need for a separate calcination step. The optimized samples showed high conductivity, with Zn-doped BZCYb reaching  $0.9 \times 10^{-2} \text{ Scm}^{-1}$  and ZnO-added BZCYb reaching  $0.6 \times 10^{-2} \text{ Scm}^{-1}$  at 700 °C in humidified N<sub>2</sub>, demonstrating improved sintering and maintained performance. A new proton-conducting electrolyte BaCe<sub>0.5</sub>Zr<sub>0.3</sub>Y<sub>0.1</sub>Yb<sub>0.05</sub>Zn<sub>0.05</sub>O<sub>3-δ</sub> was introduced [13], designed for IT-SOFCs (400–700 °C). It was synthesized via solid-state reaction at 1400 °C, the material achieved 98 % density and exhibited an orthorhombic structure. It showed high chemical stability and protonic conductivity, with greater conductivity and lower activation energy in wet hydrogen. A single cell using this electrolyte and a BSCF cathode reached a peak power density of 649

mWcm<sup>-2</sup> at 973 K, highlighting its potential for practical SOFC applications. BaCe<sub>0.9-x</sub>In<sub>x</sub>Gd<sub>0.1</sub>O<sub>3-δ</sub> (x = 0.1–0.4) electrolytes were synthesized via solid-state reaction method. Electrochemical tests showed that BaCe<sub>0.6</sub>In<sub>0.3</sub>Gd<sub>0.1</sub>O<sub>3-δ</sub> has stable cubic perovskite structure, over 97 % density, and excellent chemical stability in air. It achieved the highest conductivity ( $7.24 \text{ mScm}^{-1}$  at 873 K in dry H<sub>2</sub>), making it a strong candidate for IT-SOFC electrolytes [14]. Fluorine doping (0–10 mol %) in La<sub>0.9</sub>Sr<sub>0.1</sub>Ga<sub>0.8</sub>Mg<sub>0.2</sub>O<sub>3-δ</sub> significantly enhances ionic conductivity, achieving  $3.8 \text{ mScm}^{-1}$  at 600 °C for 10 % F-doped samples. The result highlighted fluorine doping as a promising strategy for improving IT-SOFC electrolytes [15].

In this investigation, Sr was co-doped with Ba in the A-site, and Y, Zn were co-doped with Ce and Zr in the B-site of ABO<sub>3</sub> structure to understand the impact of dopants in making Ba<sub>0.3</sub>Sr<sub>0.7</sub>Ce<sub>0.7</sub>Zr<sub>0.1</sub>Y<sub>0.15</sub>Zn<sub>0.05</sub>O<sub>3-δ</sub> (BSCZYn70) electrolyte for the possible application in IT-SOFC. Solid state reaction (SSR) technique was used to synthesize the material. Moreover, X-ray diffraction, scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and electrochemical impedance spectroscopy (EIS) techniques were used to characterize the material.

## 2. Methodology

### 2.1. Synthesis

The material Ba<sub>0.3</sub>Sr<sub>0.7</sub>Ce<sub>0.7</sub>Zr<sub>0.1</sub>Y<sub>0.15</sub>Zn<sub>0.05</sub>O<sub>3-δ</sub> (BSCZYn70) was synthesized using the solid-state reaction method. Stoichiometric amounts of SrCO<sub>3</sub>, BaCO<sub>3</sub>, CeO<sub>2</sub>, ZrO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, and ZnO (provided by the company Sigma Aldrich) were precisely measured with an electronic analytical balance. These powders were mixed with acetone and ground in an agate mortar for about an hour, then calcined in air at 1000 °C for 8 h in a muffle furnace. The calcined powders were reground with acetone for another hour, pelletized into 13 mm under 5 tons of pressure, and sintered at 1300 °C for 12 h. In this study, Zn was added to enhance densification and lower the sintering temperature of the material [28,29]. To minimize impurities from unreacted oxides or carbonates, careful mixing was performed. To avoid any impurity or undesired peaks in the XRD patterns, the material usually reground and sintered again at higher temperatures for improving phase purity [16].

### 2.2. Characterizations of the material

Using a Shimadzu-7000 X-ray diffractometer (CuKα<sub>1</sub> radiation, λ = 1.5406 Å), X-ray diffraction patterns were recorded over a 2θ range of 5°–80° with a step

size of  $0.02^\circ$  per minute at ambient temperature to investigate the structural properties of the mixed oxide material. The Rietveld refinement was utilized to examine the diffraction data [17]. The thermal behavior was analyzed utilizing Mettler Toledo (USA) apparatus over a temperature spectrum of  $30\text{--}1200^\circ\text{C}$ , with heating and cooling rates of  $10^\circ\text{C}/\text{min}$ . Microscopic characteristics were analyzed via a fourth-generation NeoScope JCM-7000. EIS was executed with the Admiral Squidstat Plus at open circuit voltage across a frequency spectrum of  $200\text{ kHz}$  to  $0.1\text{ Hz}$ , with a signal amplitude of  $10\text{ mV}$ . BSCZYZn70 pellet was sintered, polished, and uniformly coated with platinum paste utilizing a Quorum Q150RS sputter coater to create a Pt/BSCZYZn70/Pt structure. The pellet measured around  $13\text{ mm}$  in diameter and  $4\text{ mm}$  in thickness, with a  $6\text{ mm}$ -wide platinum-coated region on either side that functioned as the current collector. Impedance measurements were conducted in the air atmosphere across a temperature range of  $500\text{--}800^\circ\text{C}$ , in  $50^\circ\text{C}$  increments, with a heating rate of  $3^\circ\text{C}/\text{min}$ . The sample was let to attain thermal equilibrium at each temperature prior to measurements. The impedance measurements were analyzed with Z-View software, employing the brick-layer model to elucidate the electrical behavior.

### 3. Results & discussion

#### 3.1. Structural analysis

Fig. 1 displays the X-ray diffraction pattern of BSCZYZn70. FullProf and Checkcell software were used

to reveal the indexing of BSCZYZn70 [9]. The indexing pattern was crystallized in cubic symmetry within  $Pm\text{-}3m$  space group. The most intense (highest) diffraction  $2\theta$  peak was observed in the range of  $29\text{--}30^\circ$ , while the least intense (lowest)  $2\theta$  peak appeared in the range of  $76\text{--}80^\circ$ . Fig. 2 displays the Rietveld profile of BSCZYZn70. The obtained parameters from the Rietveld refinement are represented by Table 1. The Rietveld refinement also confirmed the cubic symmetry and  $Pm\text{-}3m$  space group. The phase and space group of BSCZYZn70 can be compared to  $\text{BaCeO}_3$  [18] and  $\text{BaZrO}_3$  [19]. The crystal phase of  $\text{BaCeO}_3$  and  $\text{BaZrO}_3$  depends largely on the B-site composition, specifically the Ce and Zr content. Yttrium-doped  $\text{BaCeO}_3$  typically exhibits an orthorhombic structure, while yttrium-doped  $\text{BaZrO}_3$  tends to be cubic. In  $\text{BaCe}_{0.9-x}\text{Zr}_x\text{Y}_{0.1}\text{O}_{3-\delta}$ , the phase remains orthorhombic for  $x = 0.1$  to  $0.2$  and turns cubic for  $x > 0.3$  [20]. For  $\text{BaCe}_{0.8-x}\text{Zr}_x\text{Y}_{0.2}\text{O}_{3-\delta}$ , the structure is orthorhombic when  $x = 0.1$  to  $0.5$  and becomes cubic at  $x = 0.6\text{--}0.8$  [16]. The small difference in ionic radii between  $\text{Y}^{3+}$  ( $0.90\text{ \AA}$ ) and  $\text{Zn}^{2+}$  ( $0.74\text{ \AA}$ ) allows both to occupy the B-site without disrupting cubic symmetry. However, crystal symmetry can be significantly affected by factors like sintering temperature and synthesis method. Additionally,  $\text{Ba}^{2+}$  ( $1.61\text{ \AA}$ ) has a larger ionic radius than  $\text{Sr}^{2+}$  ( $1.44\text{ \AA}$ ) in the A-site, influencing the lattice structure [9]. As per Vegard's law, the noticed lattice parameter of BSCZYZn70 was  $4.3022\text{ \AA}$ . The theoretical density (from Rietveld analysis) and empirical density (by Archimedes' principle) of BSCZYZn70 were  $5.721\text{ gm}/\text{cm}^3$  and  $5.720\text{ gm}/\text{cm}^3$ , respectively. Hence, the high relative density of  $99.983\%$  indicates the

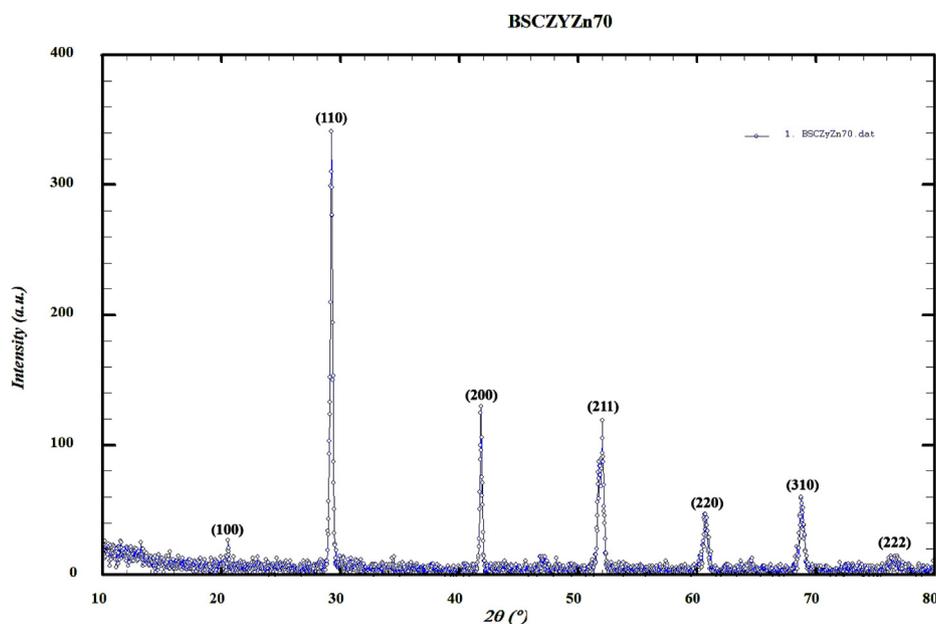
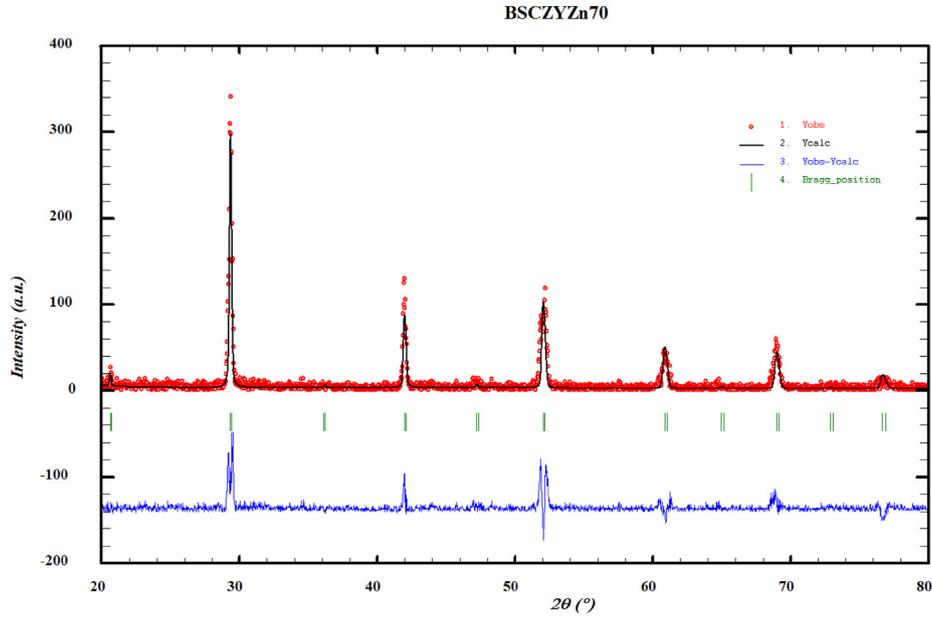
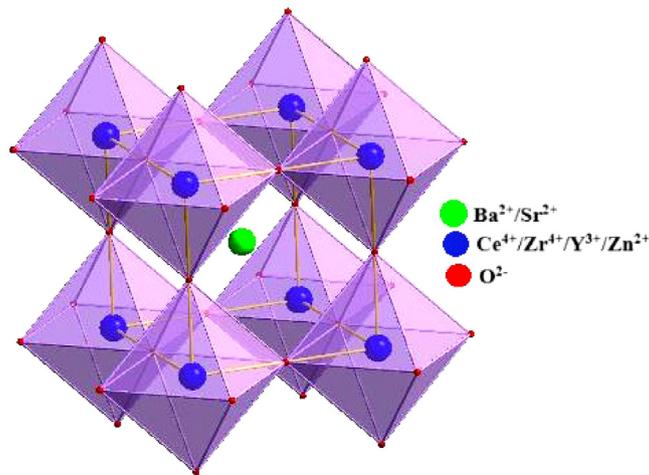


Fig. 1. X-ray diffraction pattern of BSCZYZn70.



(a) Rietveld profile patterns of the XRD data of BSCZYn70



(b) 3D structure

Fig. 2. (a) Rietveld profile patterns of the XRD data of BSCZYn70 (b) 3D structure of BSCZYn70.

material has enhanced reactivity. However, the value of goodness-fit-parameter ( $\chi^2 = 1.68$ ), derived from Rietveld analysis, indicates the strong agreement between observed and calculated diffraction data.

### 3.2. Microstructure analysis

Fig. 3 describes the microscopic images of BSCZYn70. The pellet surface of BSCZYn70 was

utilized to capture the images. The captured images were chosen with (a1) 1000X (a2) 5000X and (a3) 2500X magnifications. Since the electrolyte in SOFC must be gas-tight to prevent the mixing of gaseous reactants, analyzing the microstructure of the electrolyte material is necessary. One key factor affecting ionic conductivity is grain boundary resistance. In proton-conducting SOFC, larger grain sizes reduce the density of grain boundaries, thereby lowering grain boundary

Table 1. Rietveld refinement parameters of BSCZYn70.

Sample	Lattice parameter $a = b = c$ (Å)	Cell volume (Å <sup>3</sup> )	$\chi^2$	$R_p$	$R_{wp}$	$R_e$	$\rho_{theoretical}$ (g/cm <sup>3</sup> )	$\rho_{empirical}$ (g/cm <sup>3</sup> )	$\rho_{relative}$ (%)
BSCZYn70	4.3022	79.628	1.68	30.8	41.9	32.3	5.721	5.720	99.983

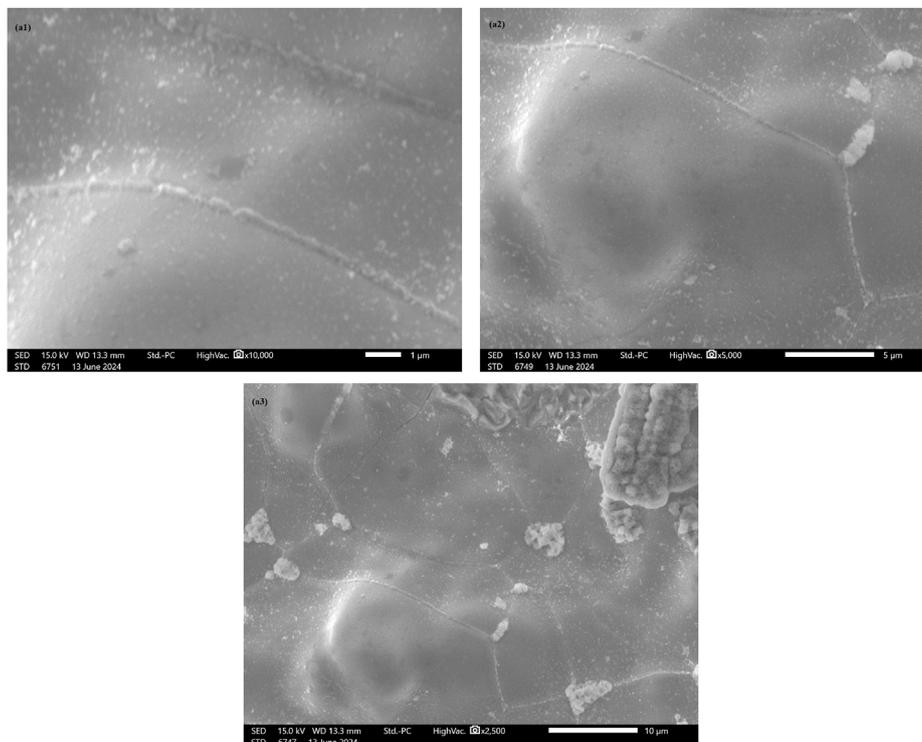


Fig. 3. Microscopic images of BSCZYn70.

resistance and enhancing total ionic conductivity. In this study, the microstructural analysis reveals a highly dense structure with numerous well-defined grain boundaries. These boundaries, having high surface energy, facilitate effective ion transport toward the electrode, which is beneficial for SOFC performance [21–23]. High-resolution imaging confirms beneficial growth of grain, showing a variety of large and small grains with a dense surface morphology. Importantly, no liquid or secondary phases are detected at the grain boundaries, indicating a well-sintered and phase-pure material. The obtained result from this study can be compared to the previous studies [9,13,28,29]. Elemental distribution of BSCZYn70 (represented by Table 2) using EDX further supports the material's integrity. The measured elemental ratios closely match the theoretical values derived from the chemical

formula, with no evidence of unreacted or impurity phases, suggesting high material purity and successful synthesis. Whatsoever, the surface connectivity appears excellent, with no visible cracks or discontinuities. This intact microstructure enables efficient ion transport across the electrolyte, making the material a promising candidate for IT-SOFC.

### 3.3. Thermal analysis

Fig. 4 displays the thermal weight loss of BSCZYn70 in air atmosphere. A thorough understanding of the thermal stability and decomposition behavior of perovskite materials is substantive for their investigation. Thermogravimetric analysis plays an important role in this process, as it allows precise measurement of weight changes in perovskite samples. These measurements provide valuable insights into how the materials degrade when exposed to increasing temperatures. In this case, thermogravimetric measurement was taken under the temperature range of 30–1000 °C. The observed thermal loss was 1.49 % at 1000 °C. But, the initial thermal loss (below 400 °C) was 0.62 %. The initial thermal loss usually happens due to the evaporation of absorbed water from the environment. Beyond that, further weight loss is associated with the valence changes of B-site cations and the formation of oxygen vacancies.

Table 2. Elemental distribution of BSCZYn70.

Sample	BSCZYn70			
	Formula		EDX	
Element	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
Ba	18.45	15	17.80	14.88
Sr	27.45	35	29.07	38.10
Ce	43.90	35	45.91	37.63
Zr	4.08	5	4.12	5.18
Y	5.97	7.5	2.69	3.48
Zn	0.15	2.5	0.42	0.73

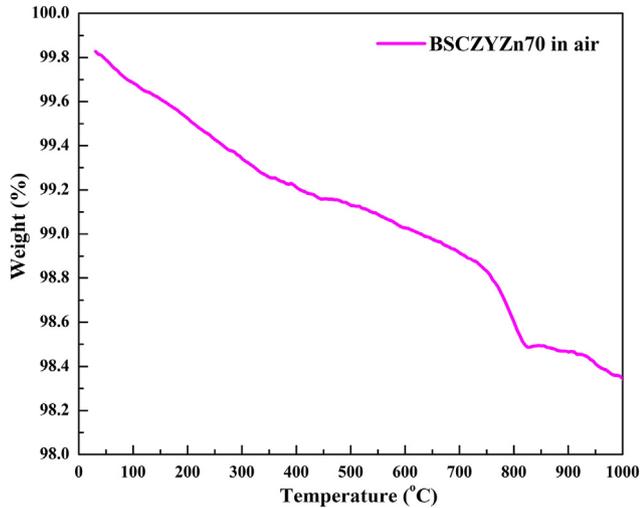


Fig. 4. Thermal behavior of BSCZYn70 in air.

### 3.4. Impedance analysis

Protons are considered to move by following the Grotthuss mechanism in proton-conducting perovskites [24,25]. It admits proton attachment to oxygen, reorientation influenced by nearby oxygen atoms, and subsequent transfer between oxygen sites, especially at high temperatures [6]. The choice of dopant significantly affects binding energy between hydroxyl radicals and dopant ions, with computational models—validated by experimental data—showing stronger, more stable interactions (more negative binding energies) for certain dopants like indium compared to yttrium or ytterbium [26]. However, the electrochemical properties of a material are critical for assessing its potential in fuel cell applications. To evaluate these properties of this study, impedance spectroscopy was executed in air atmosphere across a temperature range of 500–800 °C. This technique effectively measures a material's resistance and conductivity under varying conditions, as lower impedance corresponds to higher ionic conductivity. High proton conductivity, indicated by low impedance, is essential for efficient fuel cell operation. Thus, material demonstrating low impedance in the experiment is considered strong candidates for fuel cell application. Fig. 5 describes the impedance spectra of BSCZYn70 in air. Resistance was obtained through fitting impedance data to a 3RC equivalent circuit model comprising resistors and parallel constant phase elements. For determining the conductivity and activation energy of BSCZYn70, the following formula [9] is used,

$$\sigma = L/RA = \sigma_0 e^{-E_a/RT} \quad (1)$$

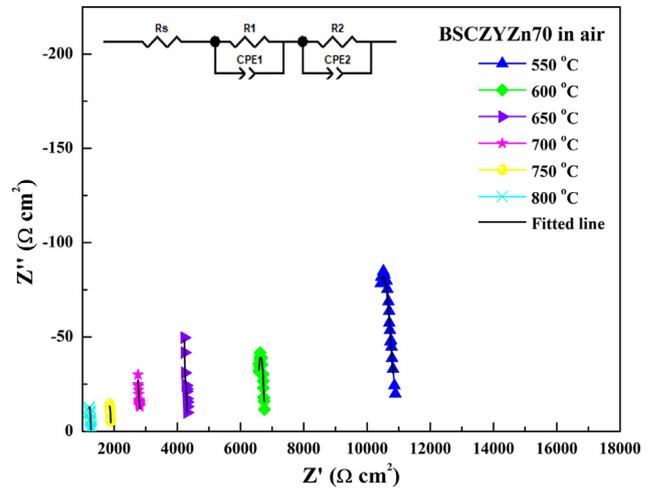


Fig. 5. Nyquist plots of impedance spectra of BSCZYn70 in air and the equivalent circuit of EIS analyses are inserted in the figure.

Where,  $\sigma$ ,  $L$ ,  $R$ ,  $A$ ,  $\sigma_0$ ,  $E_a$ ,  $R$ , and  $T$  represent conductivity, length, resistance, area, pre-exponential factor, activation energy, gas constant, and temperature, respectively.

At 700 °C, the measured conductivity of BSCZYn70 in air, was  $1.056 \times 10^{-4} \text{ Scm}^{-1}$ , as described by Fig. 6. This result can be compared to the previous study [9], measured at dry  $\text{H}_2$  atmosphere. It is notable that conductivity increases with rising temperature, as the thermal energy enhances atomic vibrations and promotes ion transport, thereby improving conductive performance. The measured (500–700 °C) activation energy (as listed in Table 3) of BSCZYn70 in air was 0.60 eV. Activation energy measurements play a crucial role in understanding electrolyte behavior, as they

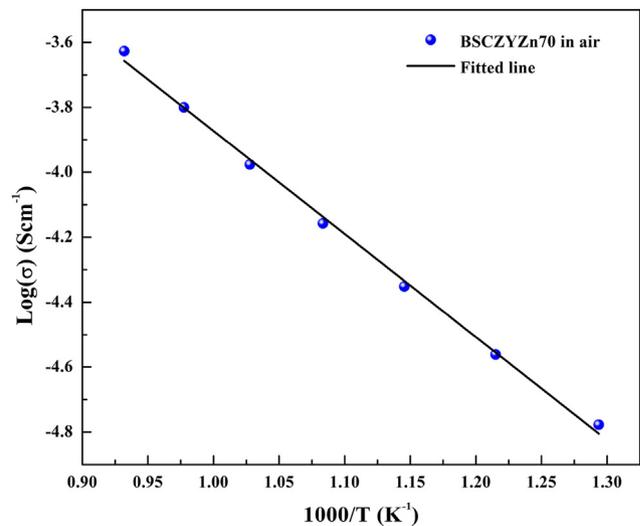


Fig. 6. Arrhenius plots of total conductivity of BSCZYn70 in air.

Table 3. Conductivity and activation energy of BSCZYn70 with other similar sample.

Sample	Conductivity ( $\text{Scm}^{-1}$ )		Activation energy (eV)	Reference
	500 °C	700 °C		
	Air	Air	500 °C–700 °C	
BSCZYn70	$1.669 \times 10^{-5}$	$1.056 \times 10^{-4}$	0.60	This work
BSCZYn30	$9.76 \times 10^{-5}$ (dry H <sub>2</sub> )	$8.99 \times 10^{-4}$ (dry H <sub>2</sub> )	0.733 (dry H <sub>2</sub> )	[9]

reveal key information about energy barriers, ion transport mechanisms, and overall ionic conductivity. A lower activation energy suggests that ions can move more easily through the electrolyte, encountering minimal resistance, which leads to higher ionic conductivity. Conversely, an elevated activation energy indicates greater resistance to ion movement and larger energy barriers, resulting in reduced ionic conductivity. These insights are essential for evaluating and optimizing materials for IT-SOFC [27].

#### 4. Conclusion

The perovskite-type material  $\text{Ba}_{0.3}\text{Sr}_{0.7}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}$  showed cubic symmetry and  $Pm\bar{3}m$  space group by the Rietveld method. The material demonstrated advantageous grain growth, with the amalgamation of large and small grains, and achieved a relative density exceeding 99 %. This high relative density is crucial for electrolyte applications where high density and leak-tightness are essential. In addition, BSCZYn70 demonstrated high thermal stability with a minimal thermal weight loss of only 1.49 %. Its low activation energy of 0.60 eV suggests enhanced ion transport properties, which is highly advantageous for proton-conducting electrolyte. These combined properties indicate that BSCZYn70 is a promising candidate for use as an electrolyte in IT-SOFC. Further investigations, including testing under wet and dry H<sub>2</sub> atmospheres and performance evaluation in operating fuel cell, are planned for the future to fully assess its proton-conducting behavior.

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#### Conflicts of interest

No conflict of interest.

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