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## Highly Dense Ba<sub>0.9</sub>La<sub>0.1</sub>Ce<sub>0.7</sub>Zr<sub>0.1</sub>Y<sub>0.15</sub>Zn<sub>0.05</sub>O<sub>3</sub> Electrolyte Material for Intermediate Temperature Solid Oxide Fuel Cells

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## ORIGINAL STUDY

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## Abstract

A new perovskite-type polycrystalline  $\text{Ba}_{0.9}\text{La}_{0.1}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}$  (BLCZYn) electrolyte material was prepared by the solid-state reaction method in pure phase. The material was studied to understand the impact of co-doping of  $\text{La}^{3+}$  at the A-site and  $\text{Zn}^{2+}$  at the B-site to use in the intermediate temperature solid oxide fuel cells (IT-SOFCs). X-ray diffraction study shows that the material crystallizes in the phase purity with cubic symmetry in Pm-3m space group. Rietveld refinement of the XRD data shows the cell parameter  $a = 4.445$  with 98.35 % relative density. Scanning electron microscopy images confirm the highly dense material with distinct grain boundaries. Thermogravimetric analysis shows about 0.30 % weight loss from RT to 1000 °C. Electrochemical impedance spectroscopy in air shows the ionic conductivity of  $2.83 \times 10^{-4} \text{ Scm}^{-1}$  at 700 °C with the activation energy of 0.62 eV. This material indicates great prospects in using as electrolyte for IT-SOFCs.

**Keywords:** IT-SOFC, Perovskite, Electrochemical performance, XRD, Electrolyte

## 1. Introduction

Solid oxide fuel cells (SOFCs) have gained attention all over the world as one of the cleanest energy conversion devices that converts chemical energy into electricity by adopting electrochemical processes. SOFCs are highly efficient and environmentally friendly with fuel flexibility [1–3]. The typical SOFC operates at high temperatures (800–1000 °C) with some disadvantages such as thermal stress, material degradation, costly manufacturing etc. But intermediate temperature solid oxide fuel cell (IT-SOFC) that operates at 400–700 °C has shown promising result to overcome those disadvantages [4]. Irshad et al. [5] studied  $\text{BaCe}_{0.97}\text{Sm}_{0.03}\text{O}_{3-\delta}$

and  $\text{BaCe}_{0.97}\text{Gd}_{0.03}\text{O}_{3-\delta}$  electrolytes. Both of the electrolytes were found very dense and useful for IT-SOFC. The  $\text{BaCe}_{0.5}\text{Zr}_{0.3}\text{Y}_{0.1}\text{Yb}_{0.05}\text{Zn}_{0.05}\text{O}_{3-\delta}$  electrolyte show high density and proton conductivity which is suitable for IT-SOFC application [6]. The studied electrolyte was found to be very dense with 98 % relative density. They reported ionic conductivity was  $0.0122 \text{ Scm}^{-1}$  at 700 °C. Hossain et al. [7] investigated the  $\text{BaCe}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}$  electrolyte for the use in IT-SOFC. They found 96.76 % relative density with  $0.01 \text{ Scm}^{-1}$  ionic conductivity at 700 °C. The  $\text{BaZr}_{0.8}\text{Y}_{0.2}\text{O}_{3-\delta}$  electrolyte was studied by Li et al. [8]. They found 95.6 % density with  $0.0069 \text{ Scm}^{-1}$  ionic conductivity in wet air at 700 °C. Radenahmad et al. [4] studied

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$\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Ce}_{0.5}\text{Zr}_{0.35}\text{Y}_{0.1}\text{Sm}_{0.05}\text{O}_{3-\delta}$  electrolyte for use in IT-SOFC. Their reported ionic conductivity was  $0.001159 \text{ Scm}^{-1}$  in dry argon at  $700 \text{ }^\circ\text{C}$  with 91 % relative density. Lyagaeva et al. [9] studied the  $\text{BaCe}_{0.5}\text{Zr}_{0.3}\text{Y}_{0.1}\text{Yb}_{0.1}\text{O}_{3-\delta}$  electrolyte for IT-SOFC and found it highly dense with 97 % relative density. The  $\text{LaMg}_{0.67}\text{Nb}_{0.33}\text{O}_3$  electrolyte was investigated by Gao et al. [1] for both of the intermediate and high temperature-based SOFCs and found  $0.0023 \text{ Scm}^{-1}$  ionic conductivity at  $550 \text{ }^\circ\text{C}$ . Leng et al. [10] studied the  $\text{BaCe}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.1}\text{Yb}_{0.1}\text{O}_{3-\delta}$  electrolyte for the IT-SOFCs. The investigated ionic conductivity of this material was  $0.03 \text{ Scm}^{-1}$  at  $700 \text{ }^\circ\text{C}$ . The  $\text{La}^{3+}$  co-doped  $\text{Ce}_{0.8}\text{La}_{0.1}\text{Ca}_{0.1}\text{O}_{2-\delta}$  electrolyte for IT-SOFCs demonstrated an improved ionic conductivity of  $1.06 \times 10^{-1} \text{ S cm}^{-1}$  at  $800 \text{ }^\circ\text{C}$ , with an activation energy of  $1.02 \text{ eV}$  [11].

The absence of  $\text{La}^{3+}$  at the A-site and  $\text{Zn}^{2+}$  at the B-site can result in a lower level of oxygen vacancy concentration, which negatively affects ionic conductivity. This makes it less suitable for applications like intermediate-temperature solid oxide fuel cells (IT-SOFCs). In this paper,  $\text{Ba}_{0.9}\text{La}_{0.1}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}$  (BLCZYZn) electrolyte material has been studied to comprehend its structural phase, surface morphology, thermal behavior, and electrochemical performance. For that, the material was synthesized and characterized by the solid state reaction method, X-ray diffraction, scanning electron microscopy, thermogravimetric analysis, and electrochemical impedance spectroscopy. The main aim of this study is to comprehend the co-doping of lanthanum ( $\text{La}^{3+}$ ) at the A-site and zinc ( $\text{Zn}^{2+}$ ) at the B-site of the  $\text{ABO}_3$  perovskite structure.

## 2. Methodology

The  $\text{Ba}_{0.9}\text{La}_{0.1}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}$  electrolyte was synthesized using the solid-state reaction method. Stoichiometric amounts of  $\text{BaCO}_3$ ,  $\text{La}_2\text{O}_3$ ,  $\text{CeO}_2$ ,  $\text{ZrO}_2$ ,  $\text{Y}_2\text{O}_3$ , and  $\text{ZnO}$  were mixed and ground with an agate mortar and pestle in acetone for approximately 1 h. The resulting mixture was then calcined in air at  $1000 \text{ }^\circ\text{C}$  for 8 h. After the calcination, the oxide powder was grinded and mixed with acetone for another 1 h. Pellets with a diameter of 13 mm were formed using a hydraulic press at 5 tons and sintered at  $1300 \text{ }^\circ\text{C}$  for 12 h. The structural properties of the mixed oxides were evaluated at room temperature using a Shimadzu-7000 diffractometer ( $\text{CuK}\alpha_1$ , wavelength  $\lambda = 1.5406 \text{ \AA}$ ) with a step size of  $0.02^\circ$  per minute across a  $5^\circ$ – $80^\circ$  range. Data refinement was performed using the Rietveld method [12]. The surface morphology and elemental composition were analyzed using a 4th generation NeoScope JCM-7000 along with EDX. Thermal behavior of the materials was assessed with a QMS 403 Aeolos®

Quadro quadrupole mass spectrometer over a temperature range of  $30 \text{ }^\circ\text{C}$ – $1200 \text{ }^\circ\text{C}$ , with heating and cooling rates of  $10 \text{ }^\circ\text{C}$  per minute. Electrochemical impedance spectroscopy (EIS) was conducted at open cell voltage using the Admiral Squidstat Plus, with a signal amplitude of 10 mV across a frequency range of 200 kHz to 0.1 Hz. For impedance measurements, pellets of  $\text{Ba}_{0.9}\text{La}_{0.1}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}$  were sintered, polished, and coated with platinum paste symmetrically ( $\text{Pt}/\text{Ba}_{0.9}\text{La}_{0.1}\text{Ce}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.15}\text{Zn}_{0.05}\text{O}_{3-\delta}/\text{Pt}$ ) using a Quorum Q150RS sputter coater. Platinum paste was applied to the central area on both sides of each pellet, serving as a current collector with a size of approximately 6 mm. The pellets measured about 4 mm in thickness and 13 mm in diameter. Impedance measurements were taken in air from  $500 \text{ }^\circ\text{C}$  to  $800 \text{ }^\circ\text{C}$  at  $50 \text{ }^\circ\text{C}$  intervals, with a heating rate of  $3 \text{ }^\circ\text{C}/\text{min}$ . The impedance data was analyzed by Z-View software in which the brick-layer model was applied to model the electrical response of the samples.

## 3. Results and discussion

### 3.1. XRD analysis

Fig. 1 shows the XRD pattern of BLCZYZn. The Rietveld refinement and 3D structure of BLCZYZn are shown in Fig. 2(a) and (b), respectively. FullProf and Checkcell software were used to index the sintered material. The indexed pattern is crystallized in cubic symmetry with Pm-3m space group. A summary of Rietveld refinement results is presented in Table 1. Rietveld refinement also confirms that the material has cubic symmetry with Pm-3m space group. The lattice parameters of this material presented in Table 1 closely match with typical perovskite materials  $\text{BaZrO}_3$  ( $a = b = c = 4.193 \text{ \AA}$ ) [4,13] and  $\text{BaCeO}_3$  ( $a = b = c = 4.445 \text{ \AA}$ ) [4,14]. Since  $\text{Y}^{3+}$  ( $0.90 \text{ \AA}$ ) and  $\text{Zn}^{2+}$  ( $0.74 \text{ \AA}$ ) have closer ionic radii, both elements can occupy the B-site in the perovskite structure, ensuring the retention of cubic symmetry. The theoretical and empirical densities of the material are  $6.174 \text{ g/cm}^3$  and  $6.072 \text{ g/cm}^3$ , respectively, resulting in 98.35 % relative density. The value of the goodness-fit parameter ( $\chi^2 = 1.43$ ) represents good agreement between the observed and calculated pattern of X-ray diffraction.

### 3.2. Scanning electron microscopy

To prevent the mixing of gaseous reactants, the electrolyte material in an SOFC must be dense and gas-tight. Resistance at grain boundaries hinders the total conductivity of SOFCs. As grain size increases, the number of these barriers is reduced, decreasing resistance and increasing overall ionic conductivity.

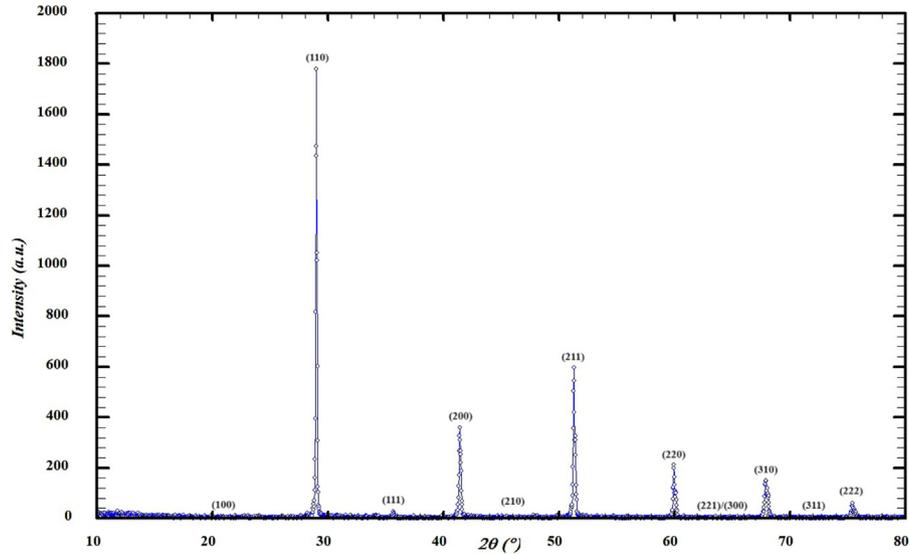


Fig. 1. XRD pattern of BLCZYzn.

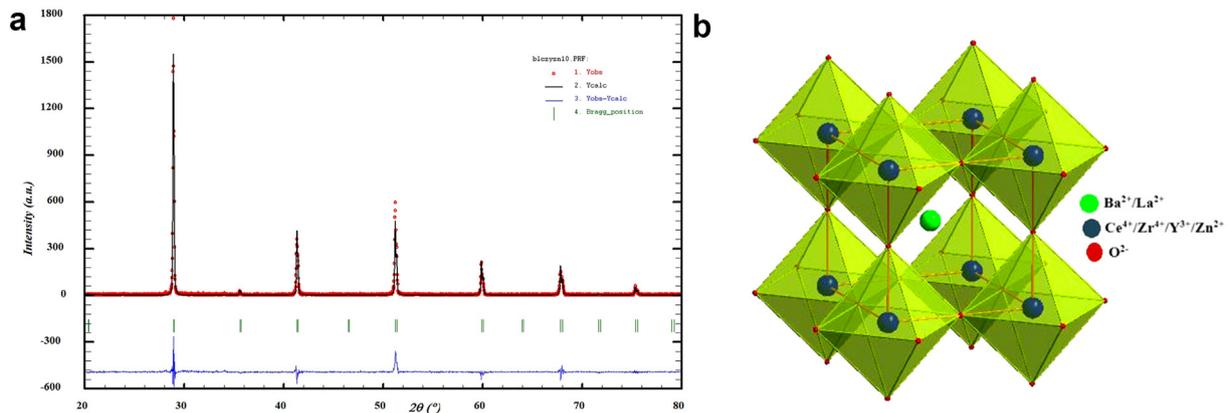


Fig. 2. (a) Rietveld refinement profile of BLCZYzn. (b) 3D Structure of BLCZYzn.

Table 1. Rietveld refinement parameters of BLCZYzn.

Sample	Lattice parameter a = b = c (Å)	Unit cell volume (Å <sup>3</sup> )	$\chi^2$	R <sub>f</sub> -factor	R <sub>p</sub>	R <sub>wp</sub>	R <sub>e</sub>	Theoretical density (g/cm <sup>3</sup> )	Empirical density (g/cm <sup>3</sup> )	Relative density (%)
BLCZYzn	4.3653	83.18	1.43	9.43	35.6	40.8	34.1	6.174	6.072	98.35

However, this adjustment may reduce the material's mechanical strength. The SEM images of BLCZYzn on pellet surface are shown in Fig. 3. The images were chosen with magnification (a) 10,000 X (b) 5000 X and (c) 2500 X. It has larger and smaller grain boundaries. Also, the material is found to be very dense, compact, crack and liquid free. The surface of the material is well connected and can pass the ions efficiently. Similar types of SEM images on electrolyte material were reported by Radenahmad et al. [4] and Hossain et al. [7] for use in IT-SOFCs. Hence, it can be used as an

electrolyte material for IT-SOFCs. The elemental distribution (presented in Table 2) by EDX analysis nearly matches with estimated values based on the chemical formulas.

### 3.3. Thermal analysis

The thermal behavior of BLCZYzn is presented in Fig. 4. Thermal analysis was conducted in air, maintaining a temperature range of 30–1200 °C to comprehend the weight change and thermal

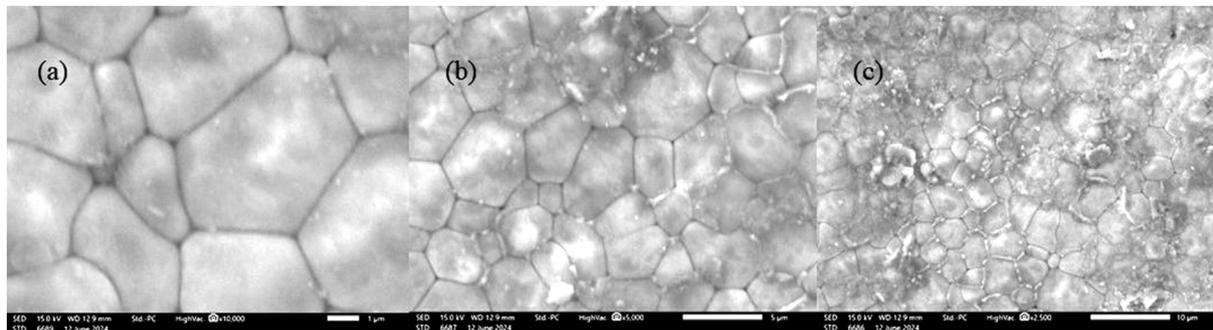


Fig. 3. SEM images of BLCZYZn.

Table 2. Elemental distribution of BLCZYZn.

Sample	BLCZYZn			
	Formula		EDX	
Element symbol	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
Ba	47.30	45	49.92	47.63
La	5.32	5	3.39	3.19
Ce	37.54	35	37.42	35
Zr	3.50	5	3.98	5.72
Y	5.10	7.50	4.07	6.01
Zn	1.25	2.5	1.22	2.45

degradation. The observed weight loss of BLCZYZn was approximately 0.30 % within the temperature range of 30–1000 °C. The initial weight loss of BLCZYZn below 400 °C was 0.03 %, attributed to the evaporation of

absorbed water from the surroundings after final sintering. Additional weight loss begins around 400 °C, caused by the formation of oxygen vacancies and the valence adjustment of B-site cations. Since the weight loss of the material can affect the oxygen occupancy, the oxygen occupancy was calculated at 500–700 °C by using the formula,  $3 - \delta = 3 - \frac{\text{weight of formula} \times \text{weight change}}{\text{atomic mass of } O_2 \times 100}$ , where  $\delta$  denotes the oxygen deficiency. Calculations show that BLCZYZn has an oxygen occupancy of 2.98 and a thermal change of 0.11 %. The observed weight stability is particularly notable due to the lower concentration of La (10 %) and higher concentration of Ba (90 %) at the A-site in the  $ABO_3$  perovskite structure. This enhanced thermal stability makes the composition well-suited for use in IT-SOFCs.

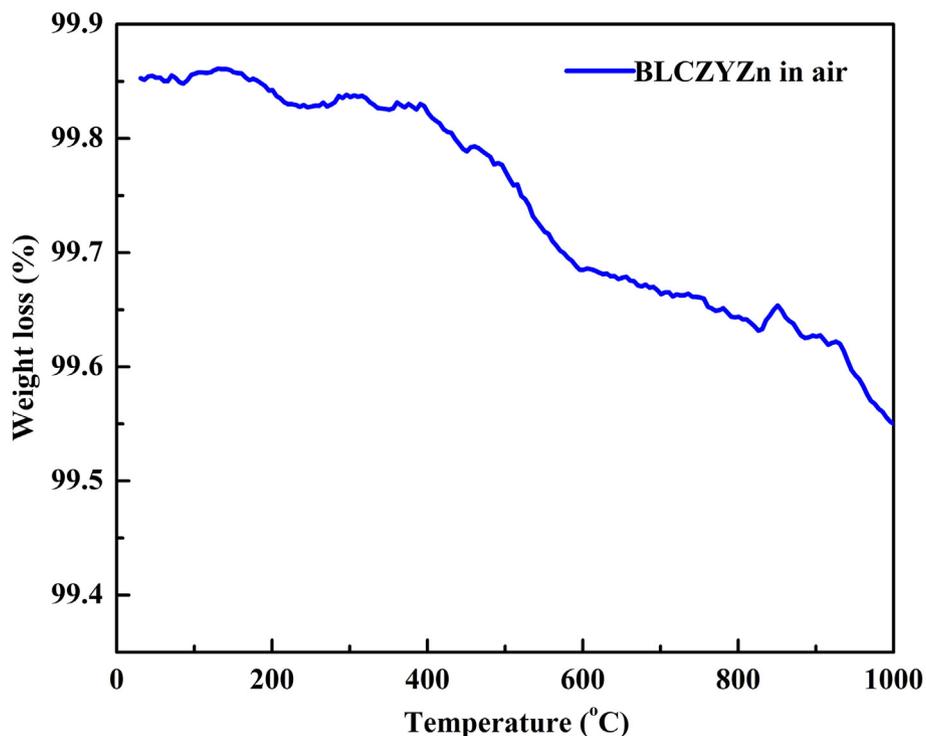


Fig. 4. TGA plot of BLCZYZn.

## 5. Electrochemical impedance spectroscopy

Electrochemical impedance spectroscopy was performed in air from 500 to 800 °C to assess the material's electrochemical performance. As impedance and conductivity are inversely proportional, low impedance suggests high ionic conductivity, which is key for proton transport in fuel cells. Therefore, material showing low impedance in this test is promising for fuel cell applications. For IT-SOFCs, the impedance spectra of BLCZYZn at 700 °C is presented in Fig. 5. Resistance was determined by fitting the impedance data to 3RC (resistance in combination with parallel to CPE) equivalent circuit model, and conductivity can be estimated using the following equation [4].

$$\sigma = 1/\rho = L/RA \quad (1)$$

where,  $\sigma$ ,  $\rho$ , and  $R$  are denoted by conductivity, resistivity, and resistance, respectively.  $L$  and  $A$ , are denoted by the length and cross-sectional area, respectively.

Since conductivity is inversely proportional to resistivity, it can be maintained at higher temperatures according to the Arrhenius equation. The Arrhenius equation can be written [4] as,

$$\sigma = \sigma_0 e^{-E_a/RT} \quad (2)$$

where,  $\sigma$ ,  $\sigma_0$  and  $E_a$  are denoted by the conductivity, pre-exponential factor, and activation energy, respectively.  $R$  and  $T$ , are denoted by gas constant and temperature, respectively.

Fig. 6 represents the Arrhenius plot of total conductivity and activation energy of the BLCZYZn. The ionic conductivity of BLCZYZn is  $2.83 \times 10^{-4} \text{ Scm}^{-1}$  in air at 700 °C and the activation energy of BLCZYZn is 0.62 eV, measured at 500–700 °C, as listed in Table 3. The conductivity exhibits a significant increase as the temperature rises within the range of 500 °C–800 °C. This phenomenon can be attributed to the effects of heat-driven atomic motion and ion transport. At elevated temperatures, the thermal energy supplied to the system enhances the mobility of atoms and ions. This increased mobility facilitates more efficient charge transport, thereby boosting the overall conductivity performance. The activation energy of this material is comparable to BaZr<sub>0.8</sub>Y<sub>0.2</sub>O<sub>3- $\delta$</sub>  (0.61 eV) [15]. In proton-conducting perovskites, protons are expected to follow the Grotthuss mechanism [4,16,17], undergoing multiple transitions, especially at higher temperatures. The process involves: i) a proton attaching to an oxygen atom; ii) reorientation of the proton-oxygen bond due to surrounding oxygen atoms; and iii) proton transfer between neighboring oxygen atoms [18]. The binding energy between hydroxyl (OH\*) and dopants varies by dopant type, with values of –0.26 eV for yttrium, –0.35 eV for ytterbium, and –0.58 eV for indium,

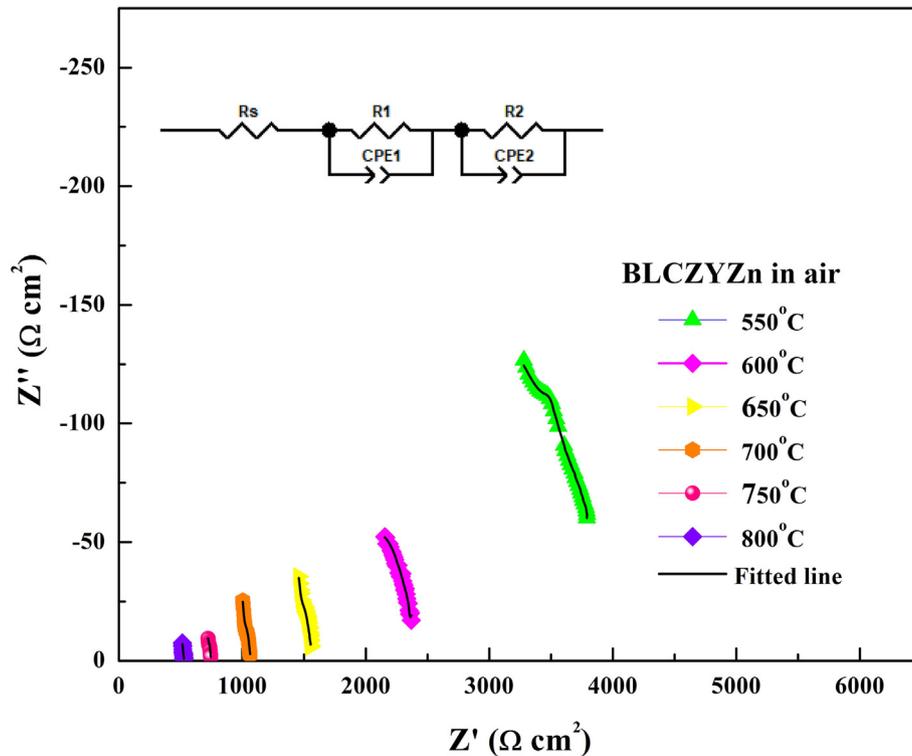


Fig. 5. Nyquist plot of BLCZYZn in air with inserted equivalent circuit.

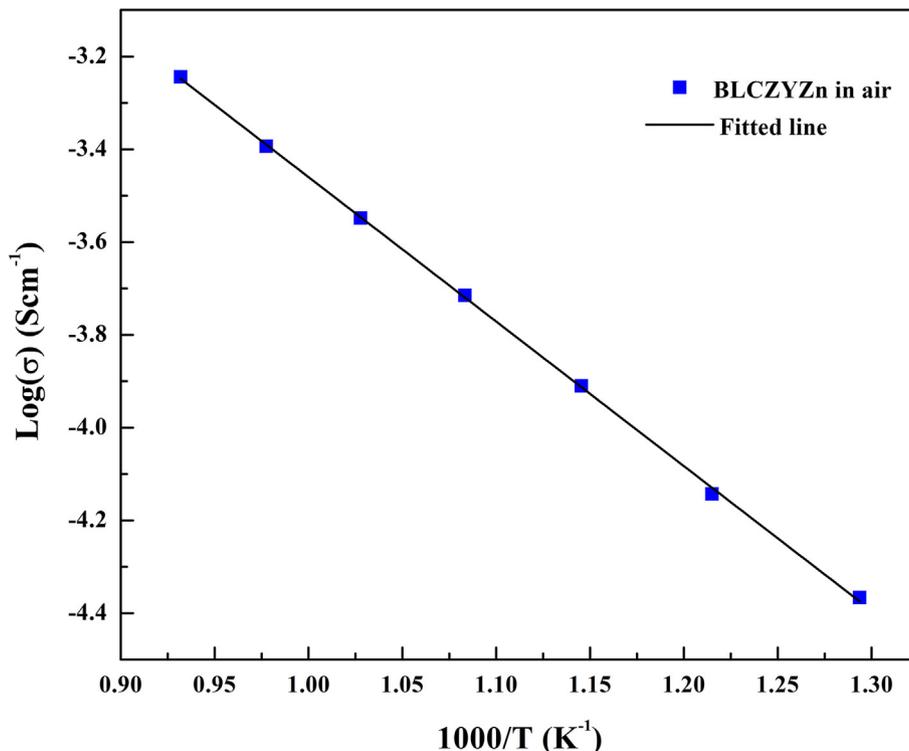


Fig. 6. Arrhenius plot of total conductivity of BLCZYZn.

Table 3. Conductivity and activation energy of BLCZYZn in air and some other related materials.

Sample	$\sigma$ (Scm <sup>-1</sup> )		$E_a$ (eV)	Reference
			500 °C–700 °C	
	500 °C	700 °C	Air	
BLCZYZn	$4.30 \times 10^{-5}$	$2.83 \times 10^{-4}$	0.62	This work
BaCe <sub>0.7</sub> Zr <sub>0.1</sub> Y <sub>0.15</sub> Zn <sub>0.05</sub> O <sub>3-<math>\delta</math></sub>		$7.55 \times 10^{-3}$ (dry H <sub>2</sub> )		[7]
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-<math>\delta</math></sub>		$1.22 \times 10^{-2}$ (wet H <sub>2</sub> )		[6]

aligning well with experimental data. More negative binding energies indicate stronger and more stable hydroxyl-dopant interactions [19].

## 6. Conclusion

The material BLCZYZn crystallizes with cubic symmetry in the Pm-3m space group, as confirmed by Rietveld refinement. SEM analysis reveals that the material is dense, compact, and exhibits a well-connected grain structure with both small and large grain boundaries. Additionally, the material shows minimal weight loss, approximately 0.30 %. Its activation energy of 0.62 eV suggests good ionic conductivity. However, further testing of BLCZYZn in H<sub>2</sub> atmospheres is planned to evaluate its potential as a proton-conducting electrolyte for use in IT-SOFCs.

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## Conflict of interest

The authors have no relevant financial or non-financial interests to disclose.

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