

Biomass Waste Incorporation in $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\alpha}\text{-Ba}(\text{Ce}_{0.6}\text{Zr}_{0.4})_{0.9}\text{Y}_{0.1}\text{O}_{3-\delta}$ Composite Cathode: Effects on Microstructural and Physical Properties

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Abstract: This study explores the incorporation of rice straw as a pore-forming agent in fabricating the $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\alpha}\text{-Ba}(\text{Ce}_{0.6}\text{Zr}_{0.4})_{0.9}\text{Y}_{0.1}\text{O}_{3-\delta}$ (LSCF-BCZY) composite cathode, focusing on its microstructural and physical properties. Conventional cathode materials often face challenges in balancing porosity and structural stability, with synthetic pore formers posing environmental and consistency concerns. To address these issues, rice straw was introduced into the cathode matrix at varying weight percentages, and the composites were sintered at 1000 °C. The addition of rice straw was evaluated using X-ray diffraction, scanning electron microscopy, and densitometry. The results revealed that increasing rice straw content significantly enhanced cathode porosity, rising from 5.53 to 27.74%, with a concomitant reduction in density from 1.33 to 0.93 g/cm³, while maintaining the crystalline stability of the LSCF-BCZY composite. Enhanced porosity suggests improved reactant diffusion to active sites, potentially benefiting the cell's performance in future energy applications. This work highlights the potential of agricultural waste as a sustainable and effective alternative to synthetic pore formers in cathode fabrication.

Keywords: biomass waste; pore former; composite cathode; microstructural properties; protonic ceramic fuel cells

■ INTRODUCTION

Protonic ceramic fuel cells (PCFCs) offer a promising alternative for efficiently and cleanly converting hydrogen and low hydrocarbons into electricity. They present several advantages over traditional fuel cells based on oxygen-ion-conducting electrolytes, such as lower activation energy and higher energy efficiency at operating temperatures between 500 and 800 °C. This intermediate temperature range reduces operating costs, mitigates material degradation, and broadens the selection of suitable materials. Additionally, in PCFCs, water is generated during the electrochemical

reaction forms at the cathode, eliminating the need for fuel recycling [1].

PCFCs consist of three essential components, i.e., cathode, anode, and electrolyte. Among these, the cathode plays a critical role in determining overall cell performance. The cathode serves as the site for the electrochemical reduction of oxygen and must possess several key properties, including high electronic conductivity, thermal expansion compatibility with the electrolyte and interconnect materials, adequate porosity to facilitate oxygen diffusion, stability under oxidizing conditions, high catalytic activity for the oxygen reduction reaction, and low fabrication cost [2].

Various cathode materials have been studied for use in PCFCs, with mixed ionic and electronic conductors (MIECs) featuring perovskite structures being among the most common. MIECs are favored for their high catalytic activity in the oxygen reduction reaction (ORR) and excellent thermal stability [3]. Their high catalytic activity creates optimal conditions for the decomposition and diffusion of oxygen gas within the cathode, expanding the triple-phase boundary (TPB) from the cathode surface into the bulk, which enhances the ORR rate and improves the electrochemical performance of PCFCs. Among MIECs, the lanthanum strontium cobalt ferrite oxide series, $\text{La}_{1-x}\text{Sr}_x\text{Co}_{1-y}\text{Fe}_y\text{O}_{3-\delta}$ (LSCF), has gained significant interest due to its superior catalytic activity, high oxygen self-diffusion coefficients, excellent ionic, and electronic conductivity [4-5].

In addition to single-phase cathodes, dual-phase or composite cathodes, which combine a proton conductor with non-proton-conducting MIECs, have also been widely used in PCFCs due to their enhanced performance. Composite cathodes offer several advantages, including an expanded TPB, improved thermal matching with the electrolyte, increased chemical stability in the presence of CO_2 and water, as well as enhanced power output [3,6]. Composite cathodes, such as $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ (LSCF)– $\text{Ba}(\text{Zr}_{0.1}\text{Ce}_{0.7}\text{Y}_{0.2})\text{O}_{3-\delta}$ (BZCY) and $\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_{3-\delta}$ (BSCF) – $\text{Sm}_{0.2}\text{Ce}_{0.8}\text{CoO}_{1.9}$ (SC), have garnered considerable interest due to their enhanced performance [7-8].

The microstructure of the cathode plays a critical role in determining the overall performance of PCFCs, particularly in influencing the ORR and electron flow within the cathode [5,9]. Key parameters such as pore size, pore distribution, and degree of porosity are directly linked to the electrode's efficiency. Optimizing these microstructural features is essential for improving the cathode's electrochemical performance. Among the various approaches investigated, the use of pore formers has proven to be one of the most effective methods for achieving the desired porosity. By introducing pore-forming agents, especially those sourced from natural organic matter, the development of porous ceramics with well-controlled pore structures becomes possible,

significantly enhancing the cathode's performance [10-11]. Despite advances in optimizing PCFC cathodes, there is still a need for sustainable and cost-effective pore formers to overcome the limitations of conventional materials. Previous studies have explored a variety of pore formers, including carbon-based materials, starch, and polymers, to improve the cathode microstructure [10]. However, these materials often come with drawbacks such as high costs, limited availability, or environmental concerns.

Biowaste, such as agricultural waste like rice straw, offers a promising solution in this context. Rice straw is an abundant agricultural waste, with global production exceeding 731 million tons annually [12]. Despite its value as a bio-resource, rice straw is often disposed of through open burning, which contributes to air pollution, global warming, and environmental degradation due to the emission of sulfur dioxide (SO_2) and carbon monoxide (CO) [13]. Utilizing rice straw as a pore-forming agent in PCFCs provides an environmentally friendly alternative to its disposal while enhancing cathode performance. The incorporation of rice straw during composite cathode fabrication holds significant potential to enhance the microstructure by increasing porosity, a key factor for optimizing gas transport efficiency. However, care must be taken to avoid the formation of secondary phases during heat treatment, as these can negatively affect cathode performance by disrupting the purity of the composite. Ensuring phase purity is critical because any deviation from the desired composition can introduce resistive interfaces and affect both the conductivity and durability of the cathode [14]. Therefore, controlling the sintering conditions and carefully managing the rice straw decomposition are essential steps in preventing the formation of detrimental secondary phases, which could undermine the overall performance and long-term stability of the composite cathode.

In addition to address environmental concerns, the use of rice straw aligns with the growing trend of utilizing waste materials in ceramic production. This study aims to explore the potential of rice straw as a pore-forming agent in PCFC cathodes, with a focus on

optimizing the microstructure and improving overall cell performance. By incorporating rice straw into the LSCF-BCZY composite cathode, this research not only offers a sustainable solution for agricultural waste disposal but also contributes to advancements in fuel cell technology.

■ EXPERIMENTAL SECTION

Materials

For the synthesis of $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ (LSCF) powder, the following chemical reagents were utilized, i.e., lanthanum(III) nitrate hexahydrate (99.995%, Acros), strontium nitrate (99+%, Acros), cobalt(II) nitrate hexahydrate (99%, Acros), and iron(III) nitrate nonahydrate (99+%, Acros). Citric acid monohydrate (CA, 99.5%, Merck) and ethylenediaminetetraacetic acid (EDTA, 99%, Acros) were used as combined chelating agents to stabilize the precursor solution. For the preparation of $\text{Ba}(\text{Ce}_{0.6}\text{Zr}_{0.4})_{0.9}\text{Y}_{0.1}\text{O}_{3-\delta}$ (BCZY) powder, the key chemicals included barium nitrate (99%, Acros), cerium(III) nitrate hexahydrate (99.5%, Acros), zirconyl nitrate hydrate (99.5%, Acros), yttrium(III) nitrate hexahydrate (99.9%, Acros), ethylene glycol (EG, 99.97%, Acros), CA, and EDTA.

Instrumentation

An analytical balance (XB 220A, Precisa) was used for all weighing purposes throughout the experiment. The precursor solution was stirred and heated using a hot plate stirrer (HS7, IKA® C-MAG). The pH changes of the aliquot were measured with a pH meter (ST3100-F, Ohaus). The sample drying process was conducted in a ventilation oven (Memmert), while calcination and sintering of the samples were carried out using a chamber furnace (Thermoconcept). A hydraulic press from Specac was used for electrolyte pellet fabrication. The diameter and thickness of the fabricated pellet were determined using a vernier caliper from Mitutoyo (accuracy ± 0.02 mm).

The physical characterization of the samples was conducted using a few instruments. Fourier-transform infrared (FTIR) spectroscopy was performed using a PerkinElmer FTIR spectrometer to analyze the functional groups present in the rice straw powder. The spectra were recorded in the range of $4000\text{--}400\text{ cm}^{-1}$ with a resolution

of 4 cm^{-1} . Phase formation of the oxide compound was confirmed through X-ray diffraction (XRD) analysis with a PANalytical X'Pert PRO MRD PW3040. The microstructure and morphology of the cathode powders were examined with scanning electron microscopy (SEM) using JEOL (JSM6460LA) and Nova NANO-SEM 450 instruments. The density of the samples was determined using a densimeter (Dahometer).

Procedure

The LSCF and BCZY ceramic powders were synthesized following the procedure outlined in our previous study [9]. To fabricate the composite cathode, equal weight proportions (1:1) of both ceramic powders were thoroughly mixed. The rice straw used in this study was collected from a paddy field in Perlis, Malaysia. It was chopped into smaller pieces and dried at $80\text{ }^{\circ}\text{C}$ for 24 h to eliminate residual moisture. Once dried, the rice straw was ground into fine powder and sieved through a $63\text{ }\mu\text{m}$ mesh to achieve uniform particle size. Different amounts of rice straw powder, ranging from 10 to 40 wt.%, were incorporated into the LSCF-BCZY composite cathode. The samples were labeled RS10, RS20, RS30, and RS40, corresponding to the percentage of rice straw powder used.

The resulting powder mixtures were thoroughly homogenized using an agate mortar and pressed into discs with a diameter of approximately 15 mm under a pressure of 5 tons. The compacted samples were heat-treated at $1000\text{ }^{\circ}\text{C}$ for 5 h to remove the incorporated pore-former. A temperature of $1000\text{ }^{\circ}\text{C}$ was selected as the heat-treatment temperature reported by a previously reported study on the thermal degradation of rice straw [15]. Furthermore, this temperature was chosen to investigate potential chemical reactions and phase transformations among the composite cathode constituents under elevated thermal conditions, with the resulting phase transformations analyzed using XRD spectroscopy. The prepared samples were further characterized through SEM and density analysis. The porosity was estimated using image analysis with ImageJ 1.45p software, employing the thresholding technique to evaluate the pore structure.

■ RESULTS AND DISCUSSION

Characterization of Rice Straw Powder as the Pore Former

FTIR was performed to analyze the chemical composition of rice straw before its application as a pore-forming agent in ceramic materials. This technique is instrumental in identifying the functional groups and organic components present in the rice straw, which significantly influence its decomposition behavior during the sintering process. Understanding these characteristics is essential for predicting the effectiveness of rice straw as a pore former and its interaction with the ceramic matrix.

As shown in Fig. 1, the FTIR spectrum of rice straw powder reveals several prominent peaks corresponding to specific functional groups, identified at 3246.55, 1707.92, 1258.40, and 737.10 cm^{-1} . The broad and strong band at 3246.55 cm^{-1} , located within the range of 3200–3500 cm^{-1} , is attributed to the hydrogen-bonded O–H stretching vibration, indicating the presence of cellulose, hemicellulose, and lignin in the rice straw. Additionally, the strong peak at 1707.92 cm^{-1} is characteristic of C=O stretching, typically arising from carboxyl, ester, or ketone groups in lignin and hemicellulose. The peak at 1258.40 cm^{-1} corresponds to the C–O–C stretching vibration of aryl-alkyl ether, a functional group commonly present in lignin, while the peak at 737.10 cm^{-1} indicates the presence of aromatic C–H bending vibrations, which is the characteristic of aromatic rings in lignin. Despite the known silica content in rice straw, the FTIR spectrum does not exhibit distinct peaks in the 1000–1200 cm^{-1} region, which are typically attributed to Si–O–Si stretching vibrations in silica. This absence of silica peaks could be due to the amorphous nature of silica in rice straw, which produces broad and weak signals that may be overshadowed by the strong absorption bands of organic functional groups, such as C–O and C–O–C vibrations. Furthermore, the low silica concentration in the sample or its uneven distribution could contribute to the lack of detectable signals. This observation is consistent with the dominance of lignocellulosic components in rice straw and the limitations of FTIR in detecting inorganic compounds. These functional groups and their corresponding peaks

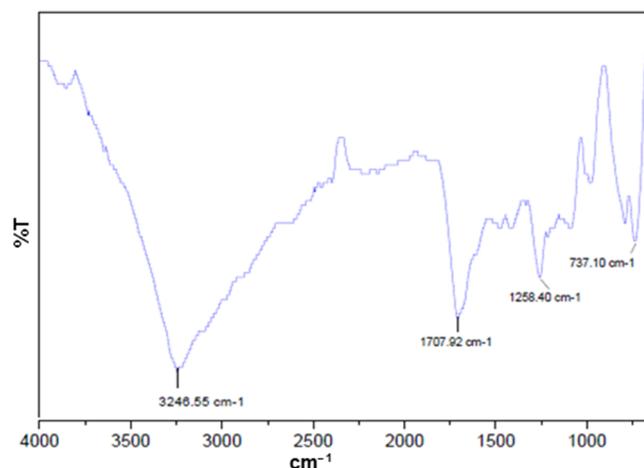


Fig 1. FTIR spectrum of the rice straw powder

reflect the primary chemical constituents of rice straw: cellulose (32–47 wt.%), hemicellulose (19–32 wt.%), and lignin (5–24 wt.%), consistent with findings from previous studies [16–18].

The absence of silica peaks highlights the predominance of organic components in the rice straw sample, making it highly suitable for use as a pore-forming agent in ceramic material processing. The organic matter, primarily composed of cellulose, hemicellulose, and lignin, undergoes thermal decomposition during sintering, releasing gases and leaving behind voids that form pores within the ceramic matrix. This behavior aligns with previous research conducted by Dele-Afolabi et al. [10], which demonstrated the efficacy of organic materials as pore-forming agents in fuel cell cathode materials. Overall, this characteristic is advantageous for achieving uniform porosity in ceramics without the risk of silica interference, which could alter the mechanical or thermal properties of the final product.

Fig. 2 presents a SEM image of the rice straw powder at a magnification of 2000 \times . This SEM image reveals a heterogeneous distribution of particles characterized by irregular shapes, including rounded, angular, and irregular forms, which vary in size and surface texture. Such variability is typical of natural biomass materials, often reflecting fragmentation patterns influenced by the plant's fibrous structure and chemical composition [19–20].

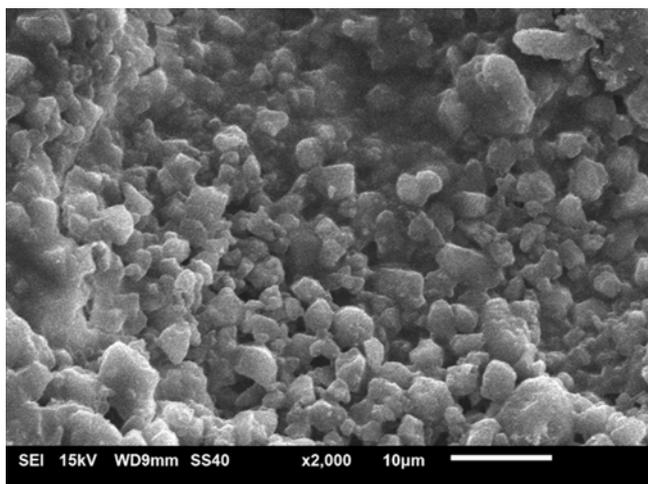


Fig 2. SEM micrograph of the rice straw powder

The morphology of rice straw powder, characterized by irregular particle shapes, varying sizes, and rough surface textures, plays a critical role in its potential as a pore-former in ceramic matrices. These morphological features enable the creation of non-uniform, interconnected pores, enhancing the overall porosity of the ceramic material. The interconnected pore network increases surface area and pore connectivity, facilitating gas diffusion during sintering and contributing to the functional properties of the final product. Such porosity is particularly advantageous for applications like fuel cells, filtration systems, and thermal insulation.

Furthermore, the irregularity in particle morphology contributes to a more uniform pore distribution, which can lead to better structural integrity in the final material. The diverse particle sizes allow graded porosity to form, enabling a balance between strength and permeability. Overall, the unique characteristics of rice straw powder highlight its promising role in enhancing the performance of ceramic materials through improved porosity and structural quality.

Effect of Biowaste Incorporation on the Crystalline Structure of the Composite Cathode

The XRD patterns of the LSCF-BCZY composite, incorporating varying concentrations of rice straw powder as a pore former and subjected to heat treatment at 1000 °C, are presented in Fig. 3. The distinct peaks corresponding to pure LSCF and BCZY powders are clearly identifiable in the composite cathode samples containing 10, 20, 30,

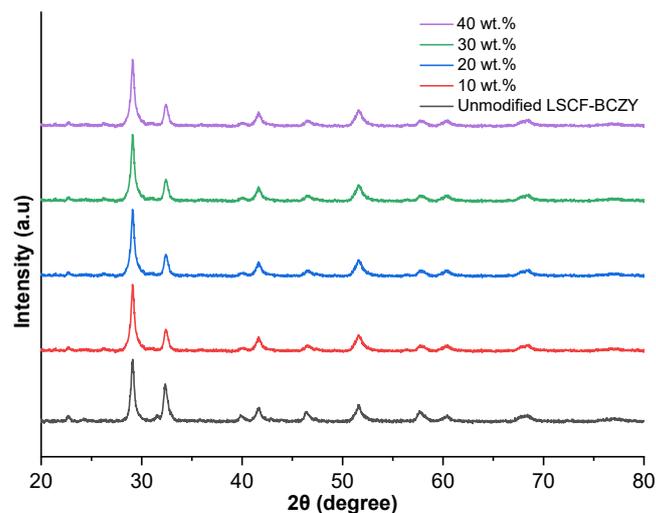


Fig 3. XRD diffraction pattern of unmodified LSCF-BCZY and the composite cathode incorporated with various amounts of rice straw powder

and 40 wt.% rice straw. Importantly, the addition of rice straw does not modify the original XRD patterns of pure LSCF and BCZY. This observation confirms that rice straw powder does not chemically interact with the composite cathode material and is entirely combusted during heat treatment. Furthermore, the absence of the secondary phase indicates that the addition of rice straw does not compromise the crystal structure or the purity of the cathode material.

Maintaining a pure phase within the composite cathode is crucial for achieving optimal electrochemical performance. Any chemical reactions between the cathode components and the pore former that result in secondary phase formation could jeopardize the cathode's structural integrity and overall performance [21]. The XRD analysis corroborates that no such reactions occurred, demonstrating that rice straw serves its purpose as a pore-forming agent without interfering with the crystalline structure or purity of the cathode. This characteristic makes rice straw highly suitable for composite cathode fabrication, as it ensures the desired structural and functional properties are preserved.

Effect of Different Amounts of Pore Former on the Microstructure of the Composite Cathode

The morphology of the composite cathode, which incorporates varying amounts of rice straw powder as a

pore former, was examined using SEM. Fig. 4 presents SEM images of both the unmodified sample and the composite cathode prepared with different rice straw loadings. The unmodified sample (Fig. 4(a)) reveals a relatively smooth surface with few visible pores or cavities, indicating a compact and uniformly distributed material. However, the lack of a prominent porous structure suggests limited airflow and ion diffusion pathways, which may impede fuel cell performance.

In contrast, the incorporation of 10 wt.% rice straw powder (Fig. 4(b)) results in a noticeable increase in both the number and size of visible pores. Specifically, the pores in the modified composite ranged from approximately 5 to 15 μm in diameter, as observed in the SEM images, compared to smaller, less distinct pores in the unmodified sample. The pore size range is comparable to findings in previous studies, where pore sizes of 5 to 10 μm were formed using pore formers such as polymethyl methacrylate, potato starch, and ammonium oxalate [22]. This increase in pore size, number, and degree of porosity highlights the effectiveness of rice straw as a pore-forming agent during sintering.

As the rice straw content increases from 10 to 30 wt.% (Fig. 4(c) and Fig. 4(d)), the microstructure

undergoes significant changes. In Fig. 4(c), at 20 wt.% rice straw powder, the composite exhibits a denser porous network with pores appearing more interconnected while maintaining structural integrity. The pores in this sample are more uniform in size and shape compared to the 10 wt.% sample, reflecting a balanced microstructural development. Meanwhile, at 30 wt.% of rice straw powder (Fig. 4(d)), the pore network becomes increasingly pronounced, characterized by larger pore sizes and greater void formation. This enhanced porosity is attributed to the thermal decomposition of the organic rice straw particles, which generate interconnected voids that facilitate gas diffusion and expand the active surface area [15,23].

However, at a 40 wt.% (Fig. 4(e)), while the material demonstrates a substantial increase in porosity and interconnected pores that facilitate gas diffusion, concerns arise regarding structural integrity. Similar to findings in previous studies, the presence of non-uniform pore shapes and the potential collapse of thin pore walls during high-temperature sintering could compromise the mechanical stability of the composite cathode [24]. Consequently, further investigation is necessary to optimize rice straw content, mitigate pore

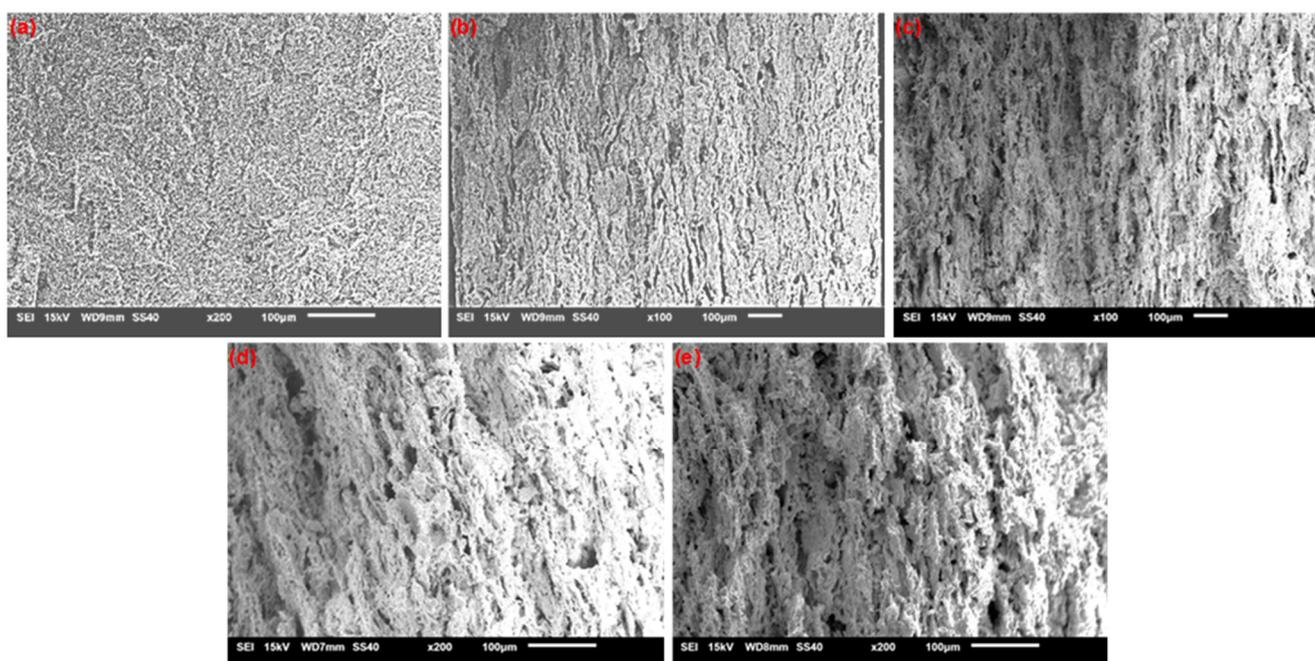


Fig 4. The SEM images of the cross-sectional view of (a) the unmodified composite cathode and the composite cathode modified with (b) 10, (c) 20, (d) 30, and (e) 40 wt.% of the rice straw powder

deformation, and establish a well-defined porous network that balances performance and durability.

The porosity levels of the composite cathode, as determined by image analysis software, are summarized in Table 1. The results indicate a significant increase in porosity with the addition of rice straw as a pore former. Specifically, the initial porosity of the unmodified sample is 5.53%, which is notably low for a functional cathode, where optimal porosity typically ranges from 20 to 30%. Upon incorporating 10 wt.% rice straw, the porosity increases to 13.33%, and this trend continues, reaching 20.07% with 30 wt.% rice straw. The highest porosity, at 27.74%, is observed with the addition of 40 wt.% rice straw. This progressive increase in porosity aligns with observations from the SEM images, which shows that the microstructure evolves toward a more porous network as the rice straw content increases. The porosity levels achieved in this study (13.33–27.74%) align with the range considered optimal for cathodes (20–30%), as noted in the previous studies [25].

In parallel with the porosity assessment, the effect of rice straw addition on the density of the composite cathode was evaluated, with results summarized in Table 2. The density of the unmodified sample is 1.33 g/cm³. A clear correlation is observed between the content of the pore former and the density: as the rice straw content increases from 10 to 40 wt.%, the density decreases progressively. This reduction in density supports the porosity findings, which show that when the porosity increases, the density of the composite cathode correspondingly decreases. These results indicate that the rising porosity is directly linked to a reduction in the material's overall density.

Based on these findings, it can be presumed that incorporating 20–30 wt.% rice straw provides sufficient porosity for effective cathode operation. However, further analysis of cathode performance is essential to validate this finding. While the addition of 40 wt.% rice straw achieves porosity within the suggested range [25], morphological observations raise concerns about potential pore collapse and mechanical stability at this higher loading. While pore formation is crucial for enhancing gas diffusion and improving cathode functionality [26], excessive porosity may compromise

Table 1. The porosity of LSCF-BCZY composite cathode with different amounts of rice straw powder

Rice straw amount (wt.%)	Porosity (%)
0	5.53
10	13.33
20	15.47
30	20.07
40	27.74

Table 2. The density of LSCF-BCZY composite cathode with varying amounts of incorporated rice straw powder

Rice straw amount (wt.%)	Density (g/cm ³)
0	1.33
10	1.26
20	1.11
30	1.02
40	0.93

material performance. Over-formed pores or cavities can weaken the structural integrity and mechanical strength of the cathode, which are critical for maintaining high-performance levels in protonic ceramic fuel cells. Therefore, achieving a balance when tailoring the microstructure of the composite cathode is essential to ensure sufficient porosity for optimal function without sacrificing mechanical properties. Future studies should focus on evaluating the composite cathode's mechanical strength, structural integrity, and long-term durability to confirm its suitability for practical applications.

■ CONCLUSION

This study has demonstrated that rice straw is an effective pore-forming agent for fabricating LSCF-BCZY composite cathodes, significantly enhancing porosity while maintaining the original crystalline structure. The increase in porosity with higher amounts of rice straw was accompanied by the formation of interconnected porous networks, potentially improving gas diffusion and expanding the active surface area. The absence of secondary phases confirms that rice straw does not chemically interact with the composite cathode, ensuring reliable cathode performance. Future research should explore these modified cathodes' mechanical and

electrochemical properties to assess their potential in practical applications fully.

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■ CONFLICT OF INTEREST

The authors have no conflict of interest to declare.

■ AUTHOR CONTRIBUTIONS

Ismariza Ismail, Nur Ashafieka Abdullah, and Norizah Abd Karim conducted the experiment. Shazlina Johari handled the data analysis & interpretation. Ismariza Ismail and Muhammad Mahyiddin Ramli were responsible for writing and revising the manuscript. All authors have approved the final version of this manuscript.

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