

Lanthanum Strontium Cobalt Ferrite (LSCF) perovskites by Sol-Gel Method for Potential Application in Solid Oxide Fuel Cells (SOFC)

Perovskitas LSCF (Ferritas de Lantânio, Estrôncio e Cobalto) obtidas pelo método sol-gel para potencial aplicação em células à combustível de óxido sólido (SOFC)

Perovskitas LSCF (Lanthanum, Strontium and Cobalt Ferrites) obtidas por el método sol-gel para su potencial aplicación en celdas de combustible de óxido sólido

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Abstract

Fuel cells are one of the most efficient and effective solutions to environmental problems and high energy demand. They are devices which change chemical energy into electrochemical energy, allowing much higher efficiency than conventional thermomechanical conversion methods. Lanthanum Strontium Cobalt Ferrite (LSCF) perovskites have been widely studied for application as cathodes in solid oxide fuel cells (SOFC) due to their high electrical conductivity, high thermal and chemical stability, low difference in thermal expansion coefficient, and physico-chemical compatibility with the other cells components. The aim of this work was to synthesize perovskitas type $\text{La}_{0.7}\text{Sr}_{0.3}\text{Co}_{0.5}\text{Fe}_{0.5}\text{O}_3$ by sol-gel method and evaluate the potential for application as a cathode for fuel cell. The results obtained by X-ray powder diffraction (XRD) indicate that the sol-gel method calcined at 900°C obtained an amount of the perovskite phase above 95%. The Field Emission Gun-Scanning Electron Microscope (FEG-SEM) images of LSCF film produced with 4 layers showed a better quality. Thus, the results obtained by XRD and FEG-SEM, indicate that the sol-gel method calcined at 900°C has a potential application as cathode in solid oxide fuel cells.

Keywords: Perovskite; LSCF; Fuel cell; Cathode.

Resumo

As células à combustível são uma das soluções mais eficientes e eficazes para problemas ambientais e atender a alta demanda de energia. São dispositivos que transformam energia química em energia eletroquímica, permitindo uma eficiência muito maior do que os métodos convencionais de conversão termomecânica. As perovskitas de Ferrita de Lantânio, Estrôncio e Cobalto (LSCF) têm sido amplamente estudadas para aplicação como cátodos em células a combustível de óxido sólido (SOFC) devido à sua alta condutividade elétrica, alta estabilidade térmica e química,

baixa diferença no coeficiente de expansão térmica e compatibilidade físico-química com os demais componentes das células. O objetivo deste trabalho foi sintetizar perovskitas tipo $\text{La}_{0.7}\text{Sr}_{0.3}\text{Co}_{0.5}\text{Fe}_{0.5}\text{O}_3$ pelo método sol-gel, e avaliar o potencial de aplicação como cátodo para célula à combustível. Os resultados obtidos por difratometria de Raios X (DRX) indicam que o método sol-gel calcinado a 900°C obteve uma quantidade da fase perovskita acima de 95 %. As imagens de microscopia eletrônica por emissão de campo (FEG-MEV) do filme LSCF produzidas com 4 camadas apresentaram melhor qualidade. Assim, os resultados obtidos por DRX e FEG-MEV, indicam que o método sol-gel calcinado a 900°C tem potencial aplicação como cátodo em células a combustível de óxido sólido.

Palavras-chave: Perovskita; LSCF; Células à combustível; Cátodo.

Resumen

Las pilas de combustible son una de las soluciones más eficientes y eficaces a los problemas medioambientales y de alta demanda energética. Son dispositivos que transforman la energía química en energía electroquímica, lo que permite una eficiencia mucho mayor que los métodos convencionales de conversión termomecánica. Las perovskitas de ferrita de lantano, estroncio y cobalto (LSCF) han sido ampliamente estudiadas para su aplicación como cátodos en celdas de combustible de óxido sólido (SOFC) debido a su alta conductividad eléctrica, alta estabilidad térmica y química, baja diferencia en el coeficiente de expansión térmica y compatibilidad fisicoquímica con los demás componentes de las células. El objetivo de este trabajo fue sintetizar perovskitas tipo $\text{La}_{0.7}\text{Sr}_{0.3}\text{Co}_{0.5}\text{Fe}_{0.5}\text{O}_3$ por el método sol-gel, y evaluar el potencial de aplicación como cátodo para celdas de combustible. Los resultados obtenidos por DRX indican que el método sol-gel calcinado a 900°C obtuvo una cantidad de fase perovskita superior al 95%. Las imágenes FEG-MEV de película LSCF producida con 4 capas mostraron una mejor calidad. Así, los resultados obtenidos por DRX y FEG-MEV, indican que el método sol-gel calcinado a 900°C tiene una aplicación potencial como cátodo en pilas de combustible de óxido sólido.

Palabras clave: Perovskita; LSCF; Pila de combustible; Cátodo.

1. Introduction

Efficient power generation which respects the environment is a major challenge to engineers and scientists (Ufa et al. 2022). In response to the critical need for clean energy technology, some possible solutions have evolved, including the reduction of energy consumption by improving energy efficiency, reduction of fossil energy consumption, and an increase in the supply of sustainable energy, such as renewable sources and fuel cells (Boudghene Stambouli & Traversa 2002; Yatoo & Skinner 2022; Xu et al. 2022). Fuel cell is an energy conversion device which generates electricity and heat, by the electrochemical combination of a gaseous fuel (hydrogen) and an oxidant gas (air oxygen) (Boudghene Stambouli & Traversa 2002; Stambouli & Traversa 2002; Liu et al. 2006).

The cathode of a fuel cell is the interface between air and the electrolyte; its main functions are to catalyze the oxygen reduction reaction and to lead the electrons from the external circuit to the site for the reduction reaction (Boudghene Stambouli & Traversa 2002; Cheng et al. 2022; He et al 2020). As other materials used in solid oxide fuel cells, the cathodes must meet some general criteria such as, low cost and manufacture easiness, minimal differences among thermal expansion coefficients of the various components of the cell, phase and microstructural stability during cell application, chemical stability, compatibility with others components of the cell, which keep in contact, during its production and application, mixed high electrical conductivity, ionic and electronic, stable microstructure, however porous, throughout the cell procedure and high catalytic activity to reduce the oxygen and be stable in highly oxidizing atmospheres (Boudghene Stambouli & Traversa 2002; Xu et al. 2022). In addition to the advanced structured materials, research nowadays is also going on the development of different layered structures such as perovskite materials (Andrade et al. 2020). In these systems, perovskite structure is altered by multiple substitutions at the A or B-site with the general notation of ABO_3 . A-site cations are coordinated to the oxygen while B and B-site cations occupy alternating sites in the case of perovskite materials (Klafke et al. 2021; Anderson et al. 1993). Fast oxygen ion diffusion and high catalytic activity are common phenomena due to higher possibility to accommodate defects in the perovskite materials than perovskite materials (Albuquerque et al. 2021; Arandiyán et al 2021). They exhibit excellent compatibility with the solid electrolyte such as yttria-stabilised zirconia (YSZ) of solid oxide fuel cells (Kaur & Singh 2020).

La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-δ} perovskites (LSCF) were identified as possible cathode materials to replace La_{1-x}Sr_xMnO_{3-δ} (LSM). This is due to LSCF high catalytic activity for the oxygen reduction reaction, as well as its excellent ionic and electronic conductivity through a wide range of temperatures (Wang et al. 2020; Fatah et al. 2021; Zhao et al. 2022; Costilla-Aguilar et al. 2021; Liu et al. 2006). The aim of this work was to synthesize perovskites type LSCF by sol-gel method and evaluate the potential for application as a cathode for fuel cells.

2. Methodology

2.1 Synthesis of LSCF and preparation of ceramic films

The perovskites were prepared and characterized in the laboratories of the postgraduate program in materials science and engineering at the Federal University of Rio Grande do Norte. Lanthanum Strontium Cobalt Ferrite powder with compositions La_{0.7}Sr_{0.3}Co_{0.5}Fe_{0.5}O₃ were prepared by sol-gel method. The starting materials used were La(NO₃)₃·6H₂O (Aldrich), Sr(NO₃)₂, Co(NO₃)₃·6H₂O (Aldrich) and Fe(NO₃)₃·9H₂O (Synth), ethylenediamine (Vetec) and acetic acid (Vetec) and acetylacetone (Vetec).

It was initially performed calculations to determine the quantities of reactants in the synthesis process of 1 g for the composition. After weighing of the reactants to the desired stoichiometric ratios: 0.8819 g of iron nitrate, 0.7708 g of cobalt nitrate, 1.3233 g of lanthanum nitrate and 0.272 g of strontium nitrate were dissolved, respectively, in 50 ml of distilled water, under magnetic stirring and heating at 80°C. After complete dissolution of salts, the organic solution, consisted of 1.77 ml of ethylenediamine, 1.5 ml of acetic acid and 2.7 ml of acetylacetone, it was added with molar ratio of 1:3 from metallic salt/organic load, still under magnetic stirring and heating until complete homogenization for about 3 h, resulting in a brown color pasty solution. Soon after the completion of the synthesis by sol-gel method, the obtained solution was placed in a furnace at 100°C during 24 h and the resulting powder was deagglomerated in a mortar. After, the obtained powder was precalcined to a 350°C temperature for about 2 h and the heating rate of 10°C/min in a EDG 7000 muffle furnace model. The calcination treatment was carried out at 500, 700 and 900°C for 2 h (heating rate = 10°C/min). The ceramics films were made with 0.8 g of YSZ (yttria-stabilized zirconia), pressed by a single-acting uniaxial press, with a pressure of 100 MPa, then, to reduce to a minimum gradients of material compaction, acquired during uniaxial pressing, all films were subjected to isostatic pressing, with a pressure of 100 MPa. After pressing, the pellets were sintered at 1400, 1500 and 1600°C for 6 h (heating rate = 10°C/min).

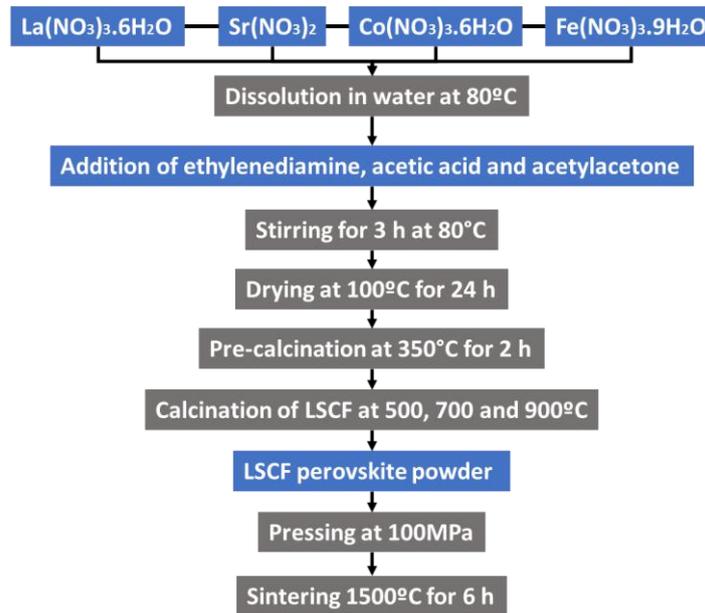
2.2 Characterization of LSCF

The powders were characterized by X-ray diffraction (XRD) and Field Emission Gun Scanning Electron Microscopy (FEG-SEM) equipped with an Oxford Link-Isis energy dispersive X-ray spectroscopy (EDS) analyzer.

The crystalline structure of the catalysts was analyzed by X-ray diffraction (XRD) using a Shimadzu XRD-7000 X-ray diffractometer with CuK α radiation ($\lambda = 1.5409 \text{ \AA}$), operating at 2 °/min with voltage and amperage of 40 kV and 30 mA. The refinement of the structure was carried out by applying the Rietveld treatment, using the MAUD software. The Joint Committee on Powder Diffraction Standards (JCPDS) was used to designate the crystalline phases with the Inorganic Crystal Structure Database (ICSD) database.

The morphological characteristics of the perovskites were observed, and Field Emission Gun Scanning Electron Microscopy (FEG-SEM) equipped with an Oxford Link-Isis energy dispersive X-ray spectroscopy (EDS) analyzer. Previously, the surface of the perovskites was coated with a gold layer of approximately 70 nm and using a ZEISS microscope, model Supra 35 VP, the surface chemical microanalysis of perovskite was observed and analyzed.

Figure 1 – Experimental scheme of LSCF perovskite synthesis and film preparation.



Source: Authors (2022).

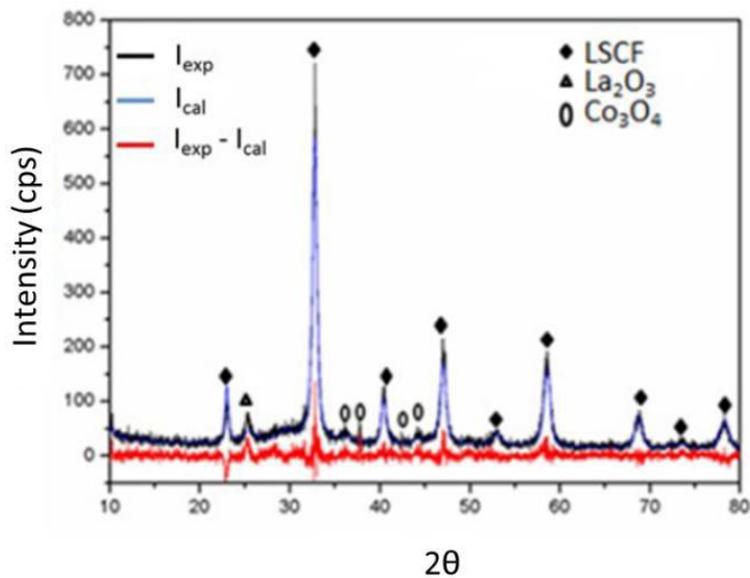
3. Results and Discussion

The X-Ray diffraction was used in this project to confirm the formation of the crystalline phase of interest (Lanthanum Strontium Cobalt Ferrite) and observing the possible presence of undesired phases.

Figure 1 shows the diffraction of X-Ray to LSCF calcined powder by 2 h at 500, 700 and 900°C. As it can be observed, the calcined powder at 900°C was the one with a lower formation of secondary phases, presenting in its mostly peaks corresponding to the material of interest. We can also observe that the diffraction peaks of the LSCF crystalline phase become more intense at an increase of calcined temperature, which suggests a gradual increase in crystallite size, being verified by the refining Rietveld method. Table 1 presents the percentage and size of crystallite to all identified crystalline phases.

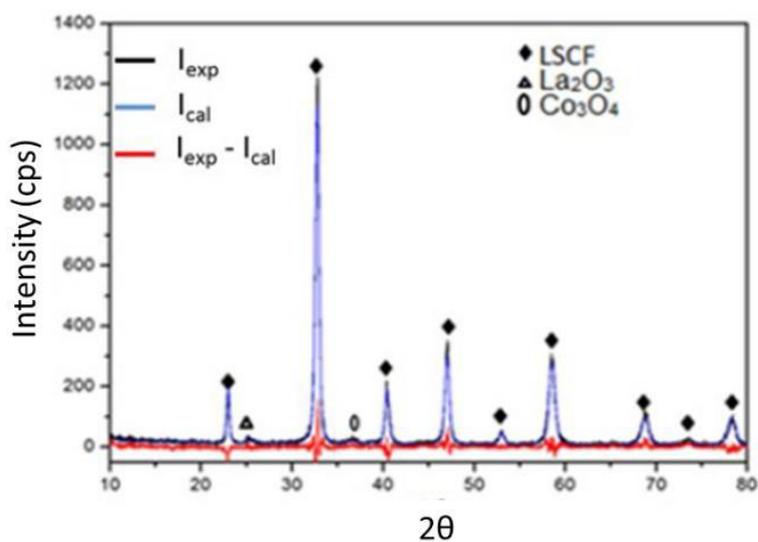
All phases obtained crystallite size at nanometer scale. The LSCF calcined at 900°C was the sample that presented the highest amount of perovskite formed (above 95%wt.) and, therefore, was the treatment selected to prepare the material to produce ceramic films. Thus, to evaluate the potential of the LSCF as a cathode, the surface was analyzed. Figures 2a and 2b are micrographs of the powders. At calcined powder at 900°C, it is found typical morphology, with particles in the form of micrometric clusters of nanoparticles, and nano-porosity is particularly noticeable in the round area of the LSCF particles.

Figure 2 – X-Ray diffraction powder from $\text{La}_{0.7}\text{Sr}_{0.3}\text{Co}_{0.5}\text{Fe}_{0.5}\text{O}_3$ composition obtained by sol-gel method and calcined at 500°C for 2 h.



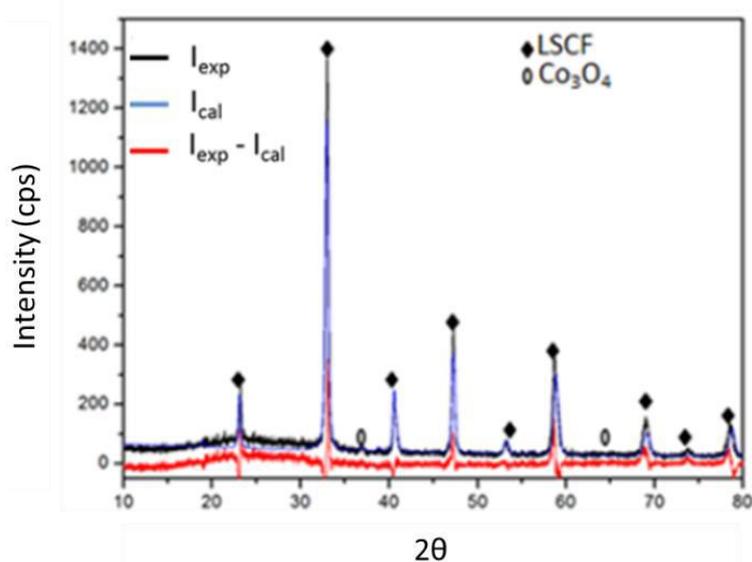
Source: Authors (2022).

Figure 3 – X-Ray diffraction powder from $\text{La}_{0.7}\text{Sr}_{0.3}\text{Co}_{0.5}\text{Fe}_{0.5}\text{O}_3$ composition obtained by sol-gel method and calcined at 700°C for 2 h.



Source: Authors (2022).

Figure 4 – X-Ray diffraction powder from $\text{La}_{0.7}\text{Sr}_{0.3}\text{Co}_{0.5}\text{Fe}_{0.5}\text{O}_3$ composition obtained by sol-gel method and calcined at 900°C for 2 h.



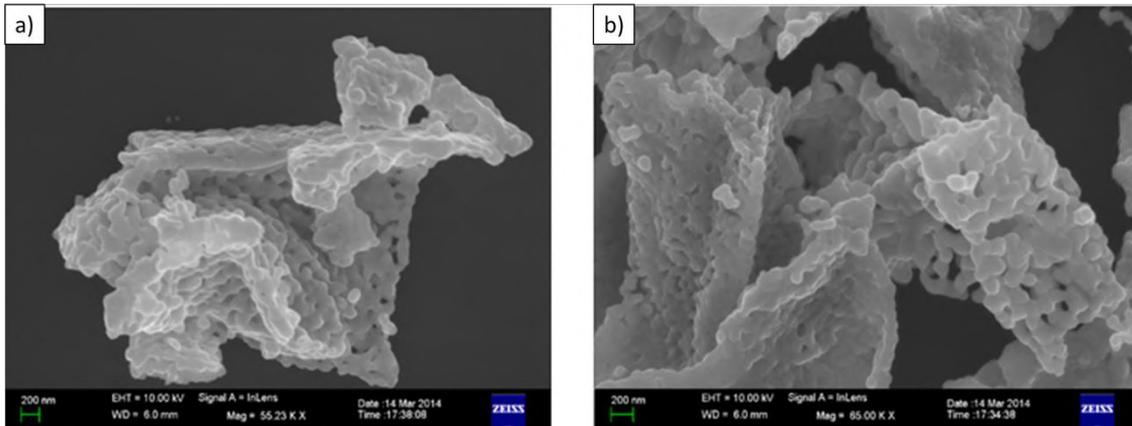
Source: Authors (2022).

Table 1 – Percentage and size of crystallite identified in the analysis of X-Ray diffraction.

Temperature ($^\circ\text{C}$)	Phase	Card #	CIF #	Quant. (% wt.)	Crystallite size (nm)
500	LSCF	01-086-1663	2017565	92.9	20.9
	Co_3O_4	01-080-1540	69373	5.35	99.9
	La_2O_3	01-083-1349	100209	1.75	99.9
700	LSCF	01-086-1663	201765	94.3	43.9
	Co_3O_4	01-080-1540	69373	5.7	99.9
900	LSCF	01-086-1663	2017565	95.8	61.1
	Co_3O_4	01-080-1540	27498	4.2	99.9

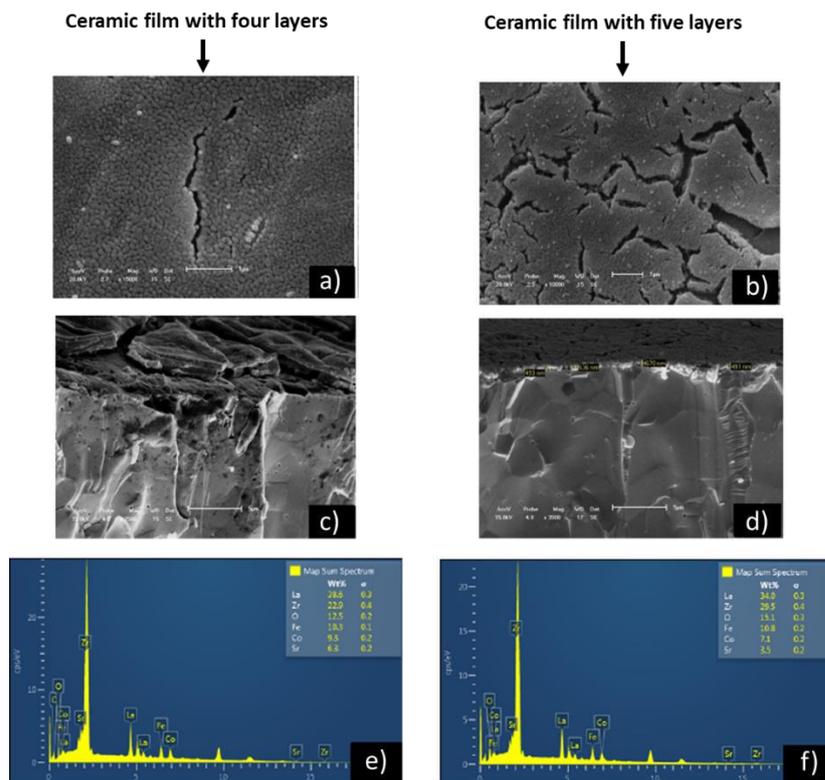
Source: Authors (2022).

Figure 5 – X-Micrography done in FEG-MEV to LSCF samples calcined at 900°C (a) Mag=55.23 KX; (b) Mag = 65.00KX.



Source: Authors (2022).

Figure 6 – Scanning electron microscopy images of (a) surface (a and b), cross-section (c and d) and EDS chemical analysis (e and f) of LSCF films with 4- and 5-layer deposition, respectively, in YSZ and sintered at 1500°C.



Source: Authors (2022).

The morphological analysis of the surface and the cross section, as well as the chemical analysis by EDS, of the thin films obtained by spin-coating, with depositions of 4 and 5 LSCF layers on the YSZ substrates are shown in Figure 6. Comparing the micrographs it was possible to identify that the deposition with 5 layers promoted a greater amount of cracks on the surface of the film, despite presenting a more compact layer. Thus, the LSCF film produced with 4 layers showed a better quality. However, the procedure can be improved by optimizing the process parameters. According to the literature, the most suitable surface microstructures for application as fuel cell cathodes have small particle size, high porosity and high

specific surface areas, allowing rapid oxygen diffusion through the cathode (Piao et al. 2008). Thus, the micrographs of the cross sections shown in Figure 6 reveal good film/substrate adhesion and allow the quantification of thickness values as being between 357 nm and 1.59 μm . Finally, the chemical analysis by EDS obtained for the films deposited in stages I and II confirm the presence of the elements La, Sr, Co, Fe and O, the identification of the element Zr is attributed to the substrate YSZ.

4. Conclusion

From the synthesis by sol-gel method it was possible to obtain lanthanum cobaltite and ferrite perovskite strontium-doped (LSCF) with crystallite size in nanometric scale, structural and morphological characteristics suitable to porous electrodes manufacture with potential application as cathodes fuel cells of solid oxide. X-ray diffraction showed that the diffraction peaks of the LSCF crystalline phase become more intense with increasing calcination temperature, which suggests a gradual increase in crystallite size and an increase in crystallinity of LSCF powders, as confirmed by the Rietveld refinement method. The LSCF powder calcined at 900°C showed the lowest formation of secondary phases and nanometric porosity. The calcination temperature was an important parameter for the development of the half-cell, as it influences several microstructural characteristics of the cell, such as the grain/pore size distribution of the electrodes and the adhesion of the electrode with the electrolyte. The SEM images indicate the good adhesion of the cathode to the electrolyte, due to the contact between the grains and the electrolyte being very homogeneous on the entire surface analyzed at the cathode/electrolyte interface.

As a suggestion for future and complementary work, the authors recommend studying the effects on the structure of perovskites by altering the synthesis route by combustion and trying to make synthesis without the need for calcination.

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