Research Article

A Study on Synthesis and Characterization of Dy-Doped $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ via the Coprecipitation Method

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The perovskite Lanthanum Strontium Cobalt Ferrite (LSCF) is investigated as the cathode material used in intermediate-temperature solid oxide fuel cells (IT-SOFCs). In the present study, $\text{La}_{0.6-x}\text{Dy}_x\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ ($x = 0, 0.3, 0.6$) was synthesized through the coprecipitation method. The obtained precipitate was calcined at 500, 700, 900, and 1000°C. Phase characterization of the synthesized LSCF and LDySCF powder before and after heat treatment at 700°C was carried out by X-ray diffraction (XRD) analysis. XRD patterns revealed that the perovskite phase was obtained at 700°C in all calcined samples. Chemical bond study to investigate the synthesis process was conducted using the Fourier transform infrared spectroscopy technique. Thermal analysis of DTA and TG has been utilized to investigate how the calcination temperature affects the perovskite phase formation. According to the STA results, the perovskite phase formation started at 551°C and completed at 700°C. The density values of synthesized powders were 6.10, 6.11, and 6.37 g·cm⁻³ for the undoped and doped samples calcined at 700°C. Powder morphology was studied by field emission scanning electron microscopy (FE-SEM). The micrographs showed the spherical-shaped particles with the average particle size of 24–131 nm.

1. Introduction

The perovskite oxides with the general formula of ABO_3 illustrate properties such as electronic conductivity, ionic conductivity, and catalytic activity that are the main reasons to use them for the different parts of a solid oxide fuel cell fabrication [1]. LaFeO_3 is a perovskite with orthorhombic crystal structure which has potential to make the cathode in solid oxide fuel cells, especially when it is doped as $\text{La}_{1-x}\text{Sr}_x\text{Co}_{1-y}\text{Fe}_y\text{O}_{3-\delta}$ (LSCF) [2, 3]. Among the various LSCF compounds, $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ (LSCF6428) is usually selected as a cathode material due to its high ionic and electronic conductivities [1, 4]. It has been shown that, in LSCF-based cathodes, defects of A site play an important role in improving cell efficiency by increasing electrical conductivity; hence, the doping strategy leads to reach the stoichiometry of LSCF by incorporating dopants in A site replaced La cations [3]. Banerjee et al. obtained the rhombohedra structured LSCF

powder through the combustion synthesis method. They have reported that the maximum content of defects in the A sites resulted in the minimum amount of resistance (0.02 Ω·cm) in the symmetric cells at the constant temperature of 800°C [3]. Another prominent property of LSCF as a cathode is the catalytic activity. Researchers investigated the effect of noble elements such as Pd on the catalytic activity of the LSCF powder [5]. One important reaction in a solid oxide full cell is the oxygen reduction reaction (ORR); Lu Zhang et al. investigated the effect of CaO additive on ORR reaction kinetics. They added CaO nanoparticles to the porous LSCF structure via the infiltration method. They observed that there was no reaction between CaO and LSCF indicating compatibility of LSCF and CaO within the moderate operating temperature range. Their impedance spectrum observations indicate that CaO nanoparticles can effectively reduce the polarization resistance of the LSCF cathode and LSCF-SDC composite cathode as well [6].

Several synthesis methods for LSCF have been reported which effected on various aspects of the quality of powders that eventually determined the efficiency of the cathode in the SOFCs. Therefore, the production process enhancement is a challenge that researchers focused on it. For instance, in comparison to the citrate method, the high content of oxygen vacancy could be produced via the hydrothermal method [7]. Sanzhao Song et al. produced the LSCF powder via the molten salt technique. They have claimed that their way of synthesis method is a suitable way for the porous-structured material production in energy conversion field equipment [8]. In another research work, LSCF powder synthesized from the ultrasound-assisted synthesis method led to forming an equiaxial LSCF phase after calcinations at temperature of 800°C [9]. In another comparative research work, researchers synthesized the genuine LSCF-CuO nanopowder through both the solid-state reaction and sol-gel method in order to figure out how the synthesis method affected the quality of finally synthesized powders. The rhombohedra perovskite structure formed after heat treatment in temperatures of 800°C and 600°C for solid-state reaction and sol-gel methods, respectively. According to their report, the lower calcination temperature for the obtained sol-gel powder can be attributed to the polymerization process that occurs during the sol-gel method. Also, they have deduced that the CuO nanoparticles acted as catalysts reduced the activation energy needed for perovskite phase formation in both synthesis methods. It is worth mentioning that the low coverage of CuO particles on LSCF also enhanced the oxygen reduction reaction kinetics [10]. Another proposed idea is to combine different methods in purpose of acquiring proper quality achievement such as the study has conducted by Fansuri et al. They have mixed solid state and mechanochemical routes to attain powder with fine particle size after optimum time of milling [11]. Alireza babaei et al. declared that the rigorous washing of the coprecipitation-yielded precipitates enhanced the purity of the calcined powder [12]. The aim of the present study is to acquire the fine and homogenous powder using the coprecipitation method and then investigate how dysprosium oxide influences the quality of the obtained powder.

2. Materials and Methods

In order to synthesize $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_3$ (LSCF), stoichiometric amounts of Sr (NO_3)₂, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, and Co (CH_3CO_2)₂·4H₂O were dissolved in distilled water separately, and La_2O_3 (and Dy_2O_3 in the compositions containing Dy) was dissolved in diluted nitric acid. All materials are 99.99% pure and purchased from Merck. After that, all the abovementioned solutions were mixed together at 65°C under vigorous stirring for 30 minutes. Ammonium carbonate 0.2 M was poured into the solution until the pH reached the value of ~10 causing a light brown precipitate to emerge. After completing the precipitation process, the obtained precipitate was washed several times until pH reduced to 7 and then dried and heat treated in the furnace at a temperature of 500–1000°C for 2 h. The phase identification was carried out by using an X-ray diffractometer

using the Cu K α X-ray ($\lambda = 1.54 \text{ \AA}$) by means of the Siemens instrument model and Fourier transform infrared spectroscopy with the PerkinElmer device model: spectrum 400 in the 4000–400 cm^{-1} wave number range, respectively. Thermal gravimetric analysis (TGA) and differential thermal analysis (DTA) were performed on a NETZSCH STA 409 PC/PG thermo analyzer in ambient atmosphere, at a heating rate of 10°C/min. Study on the microstructure and morphology was carried out by using a MIRA3 TESCAN field emission scanning electron microscope (FE-SEM). Powder densities were measured using a helium pycnometer (Accupyc 1330, Micromeritics). Figure 1 implies the synthesis process schematically.

3. Results and Discussion

The XRD patterns of the coprecipitation-yielded precipitate of supposedly LSCF before and after calcinations at different temperatures are shown in Figure 2.

According to Figure 2, the XRD pattern of dried precipitate represents some metal hydroxide precipitations were formed in advance by increasing pH during the synthesis process. Accordingly, after heat treatment of the dried coprecipitation-yielded powder at 500°C, the LaFeO_3 perovskite phase (JCPDS card No. 82–1958) was emerged besides of La_2O_3 , SrCO_3 , Fe_3O_4 , and Co_3O_4 . The XRD pattern of the sample calcined at 700°C reveals that the perovskite LSCF (JCPDS card 82–1961) [13] was formed simultaneous with La_2O_3 and a little amount of SrCO_3 as secondary phases. Presenting of La_2O_3 and SrCO_3 as impurities have been reported in the synthesis process of LSCF powder in both coprecipitation [12, 14] and sol-gel [15] methods. Regarding emerging of the orthorhombic perovskite phase (500°C), orthorhombic-structured LSCF has been presented as well. At the temperature 900°C, the LSCF characteristic peaks with rhombohedra structure became stronger while the strontium carbonate peaks vanished; however, the low intensity of La_2O_3 peaks still presented. Since increasing temperature leads to enhanced diffusion of lanthanum and strontium cations to the perovskite structure, the amount of remained La_2O_3 decreased. Although, raising calcination temperature to 1000°C led to stronger perovskite and La_2O_3 peaks because of the grain growth which supported by the reported average crystallite size calculated from XRD pattern presented in Table 1. Therefore, the best calcination temperature for the precursor prepared by the coprecipitation method is chosen as 900°C due to the minimum amount of the remained secondary phase and increasing the crystallinity of the perovskite phase.

The average crystallite size of samples calcined at the temperatures of 700, 900, and 1000°C is calculated by the Scherer equation [16]:

$$D = \frac{0.94\lambda}{\beta \cos \theta} \quad (1)$$

Here, D is the average crystallite size, λ is the wavelength, β is the peak width at the half maximum amount, and θ is the diffraction angle.

FIGURE 1: The coprecipitation synthesis process flowchart.

FIGURE 2: The XRD patterns of the synthesized powder before and after calcinations at 500, 700, 900, and 1000°C.

TABLE 1: The average crystallite size of the LSCF powder calcined at 700, 900, and 1000°C calculated by the Scherrer equation.

Calcination temperature (°C)	700	900	1000
Average crystallite size (nm)	9	11	13

The FT-IR results of as-synthesized dried LSCF precipitated before and after heating at 900°C are presented in Figure 3.

According to Figure 3, a wide peak appeared at 3392.26 cm^{-1} in the dried precipitate spectrum, showing the OH stretching band, therefore, demonstrating water adsorbed [17]. The observed peaks in the range of $1471\text{--}1368\text{ cm}^{-1}$ wavenumber confirm the presence of the structural water (OH) and confirmed metal hydroxides formed [18]. The absorption band in the 852.04 cm^{-1} wavenumber illustrates the CO_3^{2-} existence. Therefore, a few amount of carbonate compounds formed during precipitation due to using the ammonium carbonate as the precipitant agent. There are two absorption bands in the 697.75 and 746.71 cm^{-1} wavenumbers, which referred to the La-O [19] bond and lepidocrocite [20]. The observed peak in 451.08 cm^{-1} indicates Sr-O and H-Sr-OH compounds are present [21]. According to the calcined sample curve, the absorption band at 600 cm^{-1} exhibited MO_6 octahedral site absorption bands which indicate the perovskite phase was formed [22]. The two absorption bands at 439.99 and 857.65 cm^{-1} are related to lanthanum oxide [23] and C-O band [21], respectively, which confirmed lanthanum oxide and strontium carbonate phases. The peaks in wavenumbers of 1472.54 and 3367.56 cm^{-1} are related to the environmental moisture adsorbed [18].

The DTA and TG curves of the coprecipitation-yielded precipitate during heating are displayed in Figure 4. According to the DTA results, the endothermic peak at 164°C is attributed to releasing adsorbed water [16].

Three exothermic peaks at 290 , 462 , 462 , and 551°C are seen which correspond to the following:

- (1) Metal hydroxide decomposition reaction according to the following equations [17, 24–26]:

- (2) The intermediate compound formation [24, 26–29]:

FIGURE 3: FT-IR spectra of the synthesized powder before and after calcinations at 900°C.

(3) Starting the LSCF formation, respectively [16, 30, 31].

Accordingly, the LSCF rhombohedra structure was obtained after the preformation of an orthorhombic-

structured phase (LaFeO_3) [32] simultaneous with the La_2O_3 , SrCO_3 , Co_3O_4 , and Fe_3O_4 compounds by increasing temperature. As the temperature increases, the orthorhombic structure of perovskite changes into a rhombohedra phase due to more stability of it and increasing diffusion. It should be noted that the LSCF rhombohedra structure was formed at 700°C completely. The proposed mechanism for the LSCF formation is as follows:

(i) Ammonium carbonate dissolves in distilled water:

(4)

(ii) Lanthanum oxide dissolves in diluted nitric acid:

(iii) Metal salts dissolve in distilled water [22]:

(iv) The hydrated metal oxide and hydroxide complex formation at 65°C and the pH ~ 8.5 as a result of the ammonium carbonate injection into the mixed

metal solution reactor in the coprecipitation process:

The washed and dried precipitate endures step-by-step transitions as follows:

(1) Water evaporation in the 100–164°C temperature range:

FIGURE 4: STA results of the precursor before calcination.

(2) Metal hydroxide decomposition reaction beginning at 290°C:

(3) Intermediate compound formation in the 462–551°C temperature range:

- (4) The lanthanum strontium cobalt ferrite formation beginning at 551°C:

Figure 5 summarizes the LSCF formation steps graphically:

The TG result (Figure 4) shows 40.48% weight lost during the heating process. This overall weight loss occurs approximately in four steps by increasing temperature. The first step starts at a temperature range of 164–330°C, and the calculated weight loss is about 13.01 wt.%, while the TG curve shows an approximate weight loss of 9 wt.%. When intermediate compounds were formed (330–550°C), it is expected that weight increases approximately 6.85% according to calculations based on chemical reactions, whereas the curve keeps a downward trend (12.5%). Incomplete and delayed reactions of the previous step coinciding with their subsequent reactions could be the reason for these inconsistencies. For instance, the onset of the formation of lanthanum oxide occurs at 600°C [24]; therefore, some of the LaOOH may remain unreacted during the perovskite phase formation and then hydrolyzed to La_2O_3 at higher temperatures. At 550°C and higher temperatures, the samples lose weight more slowly. This slow rate of weight loss is reasonable considering the formation of the stable perovskite phase.

The XRD patterns of the LSCF and the powders which contain 0.3 mol Dy and 0.6 mol Dy at 700°C are shown in Figure 6.

Phase characterization of the compounds containing 0.3 mol Dy and 0.6 mol Dy is corresponded with LaFeO_3 (JCPDS card No. 01-082-1961) and DyFeO_3 (JCPDS card NO. 00-046-0135) cards, respectively. It could be seen from the XRD pattern of Dy-doped LSCF compounds that the perovskite phase with rhombohedra structure is formed while a few amount of strontium carbonate exists too. The diffraction pattern peaks have shifted to larger angles compared to the peak position of the undoped LSCF pattern as a result of the diffusion of the Dy^{3+} cations into the unit cell. Increasing density and the peak displacement to larger diffraction angles implies the reduction of the distance between the crystalline plates led to reducing the volume of unit cell. Therefore, it could have concluded that the Dy^{3+} cations are replaced by La^{3+} cations (A sites in the perovskite structure) regarding the smaller cationic radius of the Dy^{3+} (0.91 Å) compared to the cationic radius of the La^{3+} (1.03 Å). It also shows that the dysprosium cations occupy less space of the lattice and provide enough space for the La^{3+} cations in A site and prevent the retention of unreacted La_2O_3 [1]; however, according to Table 2, density of the sample which contains 0.3 mol Dy has not changed significantly. Based on the density relation ($\rho = (M/V)$), it was expected by replacing Dy into La ($M_{\text{Dy}} = 162.5 \text{ g}\cdot\text{mol}^{-1}$ and

$M_{\text{La}} = 138.9 \text{ g}\cdot\text{mol}^{-1}$). In theory, if some amount of Dy^{3+} cations are located in the interstitial spaces but not in the site of lanthanum cations in the position of the A sites, distortion caused by Dy^{3+} in placement in interstitial spaces makes some of the strontium cations unstable due to their relatively large radius (cationic radius = 1.12 Å) and different oxidation states, so strontium cations move out of the network. Therefore, a few amount of strontium carbonate detected in the XRD pattern of the sample containing 0.3 mol Dy is reasonable. As evidenced by the XRD pattern and the increased density of the sample which contains 0.6 mol Dy, the crystalline structure changes into orthorhombic and the volume of the unit cell reduces. In general, the substitution of lanthanide cations with higher atomic number and lower ionic radius than lanthanum reduces the lattice parameter leading to a decrease in the unit cell volume and also causes a change in the structure from rhombohedra to orthorhombic [33]. According to that the pattern of the sample contains 0.6 mol Dy, the strontium carbonate peaks become stronger and the Dy_2O_3 peaks appear too. It can be concluded that a significant radius difference of the Dy^{3+} and Sr^{2+} cations compared to the La^{3+} and Sr^{2+} radius difference increases distortion. Also, decrease of volume structure prevents Sr^{2+} and some of the Dy^{3+} from penetrating the perovskite structure, intensifying strontium carbonate and Dy_2O_3 peaks. The calculated mean crystallite size for the compounds containing 0.3 and 0.6 mol Dy is 6 and 7 nm.

The FE-SEM micrographs of the calcined powders are illustrated in Figure 7. It can be observed that the porous agglomerated particle with spherical shape formed in all samples, and high calcination temperature which makes weak primary chemical bonds become stronger leads to agglomeration of particles [16]. The small particle size is another factor that facilitates agglomeration of particles as a result of the particle assembling occurred to decrease in the high surface energy. The particle size distribution is rather uniform, and the average amounts of particle size determined by Dgmizer software for the LSCF samples which were calcined at 700, 900, and 1000°C are 44, 82, and 131 nm, respectively. Figures 7(a)–7(c) confirm powder agglomeration with increasing calcination temperature in the LSCF powder. Figures 7(e) and 7(d) are compounds containing 0.3 mol Dy and 0.6 mol Dy, respectively. Although the calcination temperature in Figures 7(a), 7(d), and 7(e) is the same, there is a significant difference between the average particle sizes of the compounds containing the Dy and LSCF powder. The mean particle size of the samples has decreased with an increasing amount of Dy. Likewise, the calculated crystallite size decreased with increasing Dy content at the constant calcination temperature. This confirms that incorporation of Dy^{3+} into the structure decreases the average particle size and crystallite size. Obviously, the presence of Dy^{3+} by distorting the structure makes it difficult for other cations to penetrate into the primary nuclei [34]. Therefore, the growth of the nuclei is reduced, thus reducing the size of crystallites, and the particle size distribution of the powders containing Dy would be more uniform compared to the LSCF sample. The average particle size for the samples containing 0.3 mol and 0.6 mol Dy is 28 and 24 nm, respectively. Some reported average LSCF particle sizes from different references have been listed in Table 3.

FIGURE 5: Graphical summary of LSCF formation steps.

FIGURE 6: The XRD patterns of the LSCF and doped LSCF which contain 0.3 mol Dy and 0.6 mol Dy after heat treatment at 700°C.

TABLE 2: The measured density for the samples calcined at 700°C

Sample	LSCF	LDSCF	DSCF
Density ($\text{g}\cdot\text{cm}^{-3}$)	6.10	6.11	6.37

FIGURE 7: (a) The 700°C-calcined LSCF powder, (b) the 900°C-calcined LSCF powder, (c) the 1000°C-calcined LSCF powder, (d) the sample containing 0.3 mol Dy calcined at 700°C, and (e) the sample containing 0.6 mol Dy FE-SEM micrographs in 50 kx magnification.

TABLE 3: The average particle size for different synthesis methods at different temperatures.

Synthesis method	Calcination temperature (°C)	Average particle size (nm)	Reference
Coprecipitation	1000	90	[12]
Sol-gel	700	10–60	[31]
Microwave-assisted glycine nitrate combustion	800	114	[35]
Sol-gel	900	90	[36]

4. Conclusions

The LSCF light brown coprecipitation yielded precipitate at 65°C in pH 10. The synthesis of the $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_3$ compound in rhombohedral crystal structure was completed after calcinations at 700°C; during the heating process, the orthorhombic phase preformed at 551°C. In addition to the LSCF phase, a very little amount of La_2O_3 and SrCO_3 has been detected in the X-ray diffraction pattern of the powder calcined at 700°C. Raising calcination temperature to 900°C led to vanishing of SrCO_3 ; then, the purest LSCF powder with the spherical shape and the average particle size of 82 nm was obtained. Doping Dy-replaced La sites reduced the average particle size to 28 and 24 nm in the composition containing 0.3 and 0.6 mol Dy, respectively. The dysprosium also eliminated the secondary phase of the lanthanum-oxide-resulted pure LSCF phase in a lower calcination temperature of 700°C.

Data Availability

All data used to support this study are included in the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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