Influence of the Amount of Carbon during the Synthesis of LaFe0.8Co0.2O3/Carbon Hybrid Material in Oxygen Evolution Reaction

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ABSTRACT: The oxygen evolution reaction (OER) and the hydrogen evolution reaction occurred at the anode and cathode, which depends on the electronic structure, morphology, electrochemically active surface area, and charge-transfer resistance of the electrocatalyst. Transition metals like cobalt, nickel, and iron have better OER and oxygen reduction reaction activities. At the same time, transition-metal oxide/carbon hybrid has several applications in electrochemical energy conversion reactions. The rich catalytic site of transition metals and the excellent conductivity of carbon material make these materials as a hopeful electrocatalyst in OER. Carbon-incorporated LaFe0.8Co0.2O3 was prepared by a simple solution combustion method for the development of the best performance of the electrocatalyst. The catalyst can deliver 10 mA/cm² current density at an overpotential of 410 mV with better catalytic stability. The introduction of carbon material improves the dispersion ability of the catalyst and the electrical conductivity. The Tafel slope and onset potential of the best catalyst are 49.1 mV/dec and 1.55 V, respectively.

1. INTRODUCTION
The production of clean and sustainable energy has turned into a vital topic to overcome energy limitations. The main point of convergence to produce sustainable energy is energy conservation and environmental protection. Nowadays as a renewable energy, hydrogen is considered as a strong candidate for the best alternative for fossil fuels.¹ In the last few decades, water electrolysis has gained a lot of attention to convert renewable energy into hydrogen.² Oxygen evolution reaction (OER) is considered as a bottleneck in water electrolysis for the production of sustainable hydrogen energy.³ OER is a four electron–proton transfer reaction that results in sluggish kinetics, and it requires higher overpotential.⁴ To lower the overpotential and to increase the OER activity, suitable electrocatalysts are required. Noble metals such as Ru and Ir and their oxides (IrO₂ and RuO₂) are considered as the state-of-the-art catalysts toward OER.⁵⁶ The high cost and rarity of these noble metals make them unsuitable for industrial applications. Therefore, the researchers are focusing more on developing non-noble metal-based OER electrocatalysts with high activity.⁷ Among them, perovskite-type oxides are considered as a promising OER electrocatalyst.

Recently many perovskite oxides (ABO₃, where A is a rare earth or alkaline earth metal ion and B is a transition-metal ion) with an excellent OER activity in alkaline solutions have been developed at a lower cost.⁹ The physical, chemical, and catalytic properties of perovskite can be altered by the partial or complete substitution of A or B cations in the perovskite oxides.⁵¹⁰¹¹ The B-site cation in the perovskite oxides acts as an active site for OER.¹² The theoretical and experimental investigation on perovskites showed that the B-site transition metal with ε₈ occupancy approximately equal to 1 ameliorates the OER activity.¹³ The creation of oxygen vacancy is another method to enhance the electrocatalytic activity. This oxygen vacancy is associated with the valence state and electronic structure of B-site transition metal in the perovskites.¹² In addition to all these methods for enhancing OER activity, the incorporation of conductive carbon material (like carbon black, graphene, carbon nanotubes, etc.) increases the electrical conductivity, which is crucial for high-efficiency catalysis.⁵ In recent times, lanthanum-based perovskite oxides received more attention and exhibited remarkable performance in electrocatalysis because the various lanthanides present in the A-site bring about a minor effect on electrocatalysis.¹⁴ She et al. synthesized La₁−ₓSrₓFeO₃−δ (LSF − x, x = 0, 0.2, 0.5, 0.8, and 1) for OER. The strontium doping on the lattice of LaFeO₃ creates oxygen vacancy, which enhances the OER activity.¹⁵ Liu et al. developed a Co-doped LaMnO₃. The OER activity of LaMnO₃ was enhanced because of cobalt doping.¹⁶ Kim et al. used a hydrothermal method for the synthesis of LaCoO₃ with three different morphologies. Porous
LaCoO₃ hollow nanosphere with an amorphous surface structure favored the highest OER activity. Alkan et al. favorably used a spray flame method for the synthesis of LaCo₁₋ₓFeₓO₃ perovskite nanoparticles. They studied the effect of the incorporation of the Fe content on OER activity. Gao et al. developed oxygen vacancy-enriched LaFeO₃₋ₓ nanosheets as the electrocatalyst for Li−O₂ batteries. The synergistic effect of the structured nanosheets, the oxygen atom defects, and valence modulation of iron are responsible for the highest electrocatalytic activity. Apart from perovskites, there are other materials like CoₓMᵧ (M = P, N), transition-metal dichalcogenides, nickel−indium thiospinels, transition-metal nitrides, and carbon materials that are active in OER.

The cobalt containing perovskite-type materials are cost-effective, highly active, and excellent OER catalysts, but their conductivity is poor. The carbon materials like graphene, reduced graphene oxide, carbon nanotubes, and so forth are good conductors of electricity and have high surface area and opulent surface functional groups. Therefore, the incorporation of carbon materials as a catalyst support in the active perovskite family members can nullify the conductivity and surface area issues and promote the electron transport. In this work, we synthesized LaFe₀.₅Co₀.₅O₃ and LaFe₀.₅Co₀.₅O₃/carbon hybrid materials by solution combustion followed by the calcination method. The presence of iron and cobalt having electron occupancy close to 1 in the e level can form a desirable chemical...
bond between the transition metal and oxygen. The varying oxidation state and the different atomic radius of iron and cobalt promote the OER mechanism by increasing the rate of redox reaction of *OOH in the rate-determining step. For the first time, we found that the presence of graphite oxide during the combustion synthesis of LaFe0.8Co0.2O3 influences the porous network structure formation. This characteristic property enhances the conductivity and improves the corresponding onset potential of the OER. The hybrid material with 5 wt % graphite oxide on preparation showed better OER catalytic performance. This work provides a new strategy for incorporating porous structures in to perovskite family members. This material is a new candidate in the perovskite family for OER application.

2. RESULTS AND DISCUSSION

The schematic representation of the synthesis procedure for catalysts is depicted in Scheme 1. The PXRD patterns of LFCO, LFCO2.5, LFCO5, and LFCO7.5 are shown in Figure 1A. The LaFeO3 and LaCoO3 are in orthorhombic and rhombohedral structures respectively in its pure form.27 The substitution of cobalt in LaFeO3 (LaFe0.8Co0.2O3, LFCO) shows a phase closer to the orthorhombic crystal structure (PDF 00-088-0641). In all the samples, the peak positions are identical, but the intensity of the peaks increased with the addition of GO during the synthesis. This means that the crystallinity of the material is improved. The Fe 3+ ionic radii are greater than Co3+ ionic radii,28 and this causes the peak shift to higher diffraction angles in LFCO compared to LaFeO3. The characteristic and well-defined peaks of LFCO, LFCO2.5, LFCO5, and LFCO7.5 were observed at 2θ values of 22.7°, 32.3°, 39.88°, 46.43°, 52.24°, 57.73°, 67.74°, and 77.18°, corresponding to the (101), (121), (220), (202), (141), (240), (242), and (412) planes, respectively. Any impurity peaks related to La, Fe, or Co oxides or hydroxides were absent, which indicates that the synthesized material is in the pure perovskite phase. The peaks corresponding to graphite oxide or reduced graphite oxide are absent in LFCO2.5, LFCO5, and LFCO7.5. This may be due to the very low amount of the carbon containing species. The crystallite sizes of LFCO, LFCO2.5, LFCO5, and LFCO7.5 are calculated to be 36, 20, 22, and 19 nm, respectively, by using Scherrer’s equation:

\[
D_{\text{XRD}} = \frac{K\lambda}{\beta_{\text{hkl}} \cos \theta}
\]

where \(D_{\text{XRD}}\) is the crystallite size (nm), \(K\) is the constant (0.89), \(\lambda\) is the X-ray wavelength (1.5405 Å), \(\beta_{\text{hkl}}\) is the full width at half-maximum of the (110) plane, and \(\theta\) is the diffraction angle.

Figure 1B represents the Raman spectra of LFCO, LFCO2.5, LFCO5, and LFCO7.5. Like LaFeO3, the synthesized LFCO, LFCO2.5, LFCO5, and LFCO7.5 may give 24 Raman active modes (7Ag, 5B1g, 7B2g, and 5B3g).29 In these types of molecules, the vibration modes present below 200 cm\(^{-1}\) and above 300 cm\(^{-1}\) are due to the displacement of lanthanum ions and the motion of oxygen ions, respectively. The vibrations present in the intermediate region (200–300 cm\(^{-1}\)) are due to both La and O ions. The B-site ions (Fe or Co) are present in the inversion center. So the vibrations involving Fe3+/Co3+ ions movements are Raman inactive. In all the four samples, Raman bands are observed at 93, 148, 287, 424, 502, and 622 cm\(^{-1}\), and they correspond to Ag, B1g, A g, B3g, and two B2g modes, respectively.30 The other bands are not clear, and this may be due to the band overlapping or low intensity. At the same time, there is no well-defined Raman active mode present in the region of 1000–1500 cm\(^{-1}\), but there is a broad band observed in this region and is shown in Figure S1. The LFCO also shows a bump in the frequency region of 1000–1500 cm\(^{-1}\). This may be due to the presence of amorphous carbon present in the sample. The Raman spectra also show the same trend as XRD data that the intensity of all Raman active modes is increasing with increasing...
the amount of carbon in the sample. Along with this trend, the broad band present in the range of 1000–1500 cm\(^{-1}\) splits into two or more peaks, which may be due to the occurrence of graphitic carbon on the surfaces of LFCO2.5, LFCO5, and LFCO7.5. But compared to the amount of perovskite phase, the amount of carbon is less. Because of that, the intensity of the graphitic D and G bands is very less. The previous reports show that along with the D and G bands, D\(^1\), D\(^*\) (∼1150 to 1200 cm\(^{-1}\)), and D\(^{11}\) (∼1500 to 1550 cm\(^{-1}\)) bands were observed in graphitic materials. The D\(^*\) band is related to the disordered graphitic lattice because of the existence of sp\(^3\) bonds. Because of the annealing of the sample, the oxygen group present in the GO decreases and the intensity of the D\(^*\) band decreases.\(^{31}\)

ATR spectra of LFCO (Figure 1C) show a sharp peak at 548 cm\(^{-1}\), which is due to the antisymmetric stretching vibration of the B–O bond of BO\(_6\) octahedra of ABO\(_3\). In pure LaFeO\(_3\), the Fe–O stretching band is observed at 535 cm\(^{-1}\), whereas in LaCoO\(_3\), the stretching vibrations of Co–O bands are present at 529 cm\(^{-1}\) with a shoulder at 579 cm\(^{-1}\).\(^{35}\) Because of the partial substitution of cobalt ions in LaFeO\(_3\), the Fe–O (B–O) stretching band is slightly red-shifted to 548 cm\(^{-1}\) and is observed in LFCO. This means that the B–O bond strength increases. It happens due to the substitution of smaller ionic radii Co\(^{3+}\) (low spin/high spin) ion. The substitution of B ions having smaller ionic radii causes tilting of oxygen octahedron and reduces its volume. This means that smaller Co\(^{3+}\) substitution in the B-site reduces the volume of Fe\(_{0.7}\)Co\(_{0.3}\)O\(_6\) octahedra, and it strengthens the Fe–O/Co–O bond and hence increases its stretching band wavenumber. In LFCO2.5, LFCO5, and LFCO7.5, a sharp peak is observed at the same wavenumber (548 cm\(^{-1}\)) as that of LFCO. The B–O stretching band is present intact in LFCO2.5, LFCO5, and LFCO7.5, which means that the carbon species included in its preparation did not change the crystal structure of LFCO.\(^{33}\)

The thermal stabilities of the synthesized LFCO, LFCO2.5, LFCO5, and LFCO7.5 were analyzed by thermogravimetric analysis, which is shown in Figure 1D. This study shows that the synthesized material is highly stable and in the pure phase. There is no appreciable weight loss observed up to 1000 °C, which means that all the material can be used for high-temperature applications.

The surface morphologies of LFCO and LFCOS were analyzed by SEM and are shown in Figure 2. These SEM images are shown in two magnifications: (a) 20 000× and (b) 100 000×. Figure 2A,B corresponds to LFCO, and Figure 2D,E corresponds to LFCO5. The figure shows that both the samples are in porous structure with nonuniform pore size. By comparing Figure 2A,F, the LFCO5 have greater number of micropores compared to LFCO. This suggests that the presence of graphite oxide during the solution combustion reaction enhances the evolution of gas molecules (H\(_2\)O, CO\(_2\), and N\(_2\)) via the thermal decomposition of the species. It creates a large number of pores in its surface. The energy-dispersive X-ray (EDX) analysis of LFCO and LFCOS (Figure 2C,F) gives an idea that the La, Fe, and Co are in the 1:0.8:0.2 ratio. This implies the proper incorporation of cobalt in the FeO\(_6\) (BO\(_6\)) octahedra. The exact quantification of carbon present in the material is difficult through EDX analysis. The elemental composition of the LFCOS was also analyzed using ICP–AES. The elemental percentages for La, Fe, and Co are in the ratio of 1.0:0.7:0.2, closely matching with the results of EDX analysis.

The elemental mapping analysis of LFCO and LFCOS was studied and is shown in Figures S1 and 3, respectively. It suggests that the surface elemental distribution of La, Fe, and Co in both the samples is reasonably homogeneous. The elemental overlay (Figures S1B and 3B) shows that some of the areas are richer in oxygen species.

The high-resolution transmission electron microscopy (HR-TEM) and HAADF–STEM analyses provide much deeper information about the morphology of the LFCO5 (Figure 4). The LFCOS consists of irregular-shaped smaller units with a rough surface texture. These structures are interconnected with each other to form an elongated network structure. The magnified image (Figure 4B,C) shows that the grains are closer to the spherical shape in the nanometer regime. The bright spot present in Figure 4D corresponds to the LaO layer and dark spot corresponds to the FeO\(_2\) layer.\(^{34}\) This means that LaO and FeO\(_2\) layers are arranged in an ordered fashion in the (121) direction. The marked area in Figure S2 shows that some amorphous carbon materials are present on the interface of LFCO5 grains. This may improve the conductivity of LFCO. The elemental analysis (Figure 5) shows that all the elements are distributed in proper ratios. The SAED pattern of LFCO5 depicts that the materials are in the polycrystalline structure.

2.1. Oxygen Evolution Activity. Engineering of active electrocatalysts with adequate electronic and geometric properties influences the catalytic activity and stability toward OER. The electrochemical behaviors of LFCO, LFCO2.5, LFCO5, and LFCO7.5 toward OER were evaluated by using rotating disc electrode (RDE) as the working electrode, platinum wire as the counter electrode, and Ag/AgCl as the reference electrode. All the electrode potentials were converted to RHE by using the equation E (vs RHE) = E (vs Ag/AgCl) + E\(^\circ\) (Ag/AgCl) + 0.059 × pH for better comparison. The influence of the incorporation of carbon material (GO) during the synthesis of LFCO on the OER was investigated by the LSV technique. All the electrochemical studies were performed in a 1 M KOH solution in a sweep rate of 5 mV/s. Figure 6A shows the typical LSV curves of electrochemical activities of LFCO, LFCO2.5, LFCO5, and LFCO7.5 electrodes toward OER studies with a rotation speed of 1600 rpm. The OER polarization curves with iR compensation at a scan rate of 5 mV/s showed that LFCO5 exhibited higher OER performance. The LFCO5 reached the current density of 10 mA/cm\(^2\) at 1.64 V, whereas LFCO, LFCO2.5, and LFCO7.5 reached at 1.69, 1.68, and 1.65 V, respectively. In order to study the effect of calcination temperature on the OER performance, OER activity of the uncalcined sample was done. The results are given in Figure S5. Calcination temperature had only a minimal effect on the OER performance (Figure S5). Figure 6B represents the bar plot of
overpotential versus samples. It shows that the LFCO5 requires an overpotential of 0.41 V (vs RHE) to achieve a current density of 10 mA/cm², which was lower than those of LFCO (0.46 V), LFCO2.5 (0.45 V), and LFCO7.5 (0.42 V). In most of the perovskites, the OER activity in the alkaline medium is explored. In this work, we compared the OER activity of LFCO5 in both alkaline and acidic media. The results are shown in Figure S6. Even though, the overpotential at 10 mA/cm² is lower in the acidic medium, the current response is much lower compared to the basic medium. In addition, chemical stability is a major issue for OER in acidic conditions.

Figure 4. HR-TEM images of LFCO5 in different magnifications: (A) 20, (B) 10, (C) 10 nm, and (D) zoomed-in portion of the white box marked in (B).

Figure 5. (A) EDS–HAADF images of LFCO5, (B) lanthanum, (C) iron, (D) cobalt, (E) carbon, and (F) oxygen mapping of LFCO5, (G) overall elemental mapping, and (H) SAED pattern.
The enhancement in activity of the material toward OER is possible by increasing the reactive site number or its intrinsic activity. The perovskite materials with the B-site atoms having $\text{eg}^1$ configuration have good catalytic activity because its $M-O$ bond energy is favorable for OER. In the case of LaCoO$_3$, Co$^{3+}$ is in the $3d^6$ configuration. In the case of low spin, intermediate spin, and high spin state Co$^{3+}$, the numbers of electrons present in the $\text{eg}$ state are 0, 1, and 2, respectively. Theoretically, the Co$^{3+}$ in the octahedral field has crystal field stabilization energy and the exchange energy almost equal. So the interchange of these low spin to high spin states is easy. This low spin–high spin transition influences the Co–O bond length and bond strength. Because of this easy spin change, it may help the partial bond making and breaking during OER mechanism. But among first-row transition metals, cobalt is expensive compared to iron. So in this study, for preparing the cost-effective catalyst, only the 0.2% B-site occupancy of Co$^{3+}$ and 0.8% Fe$^{3+}$ occupancy are selected for catalyst preparation.

Another important parameter that influences the catalytic activity is the surface area of the material. For improving the surface area of the catalyst, LFCO$_2$, LFCO$_5$, and LFCO$_7.5$ were prepared in the dispersion of graphite oxide (GO). The incorporation of GO during the synthesis improves the number of pores because of the evolution of large amount of gas molecules and hence increases its surface area. In order to prove the superiority of the LFCO$_5$ catalyst, we prepared a simple mixture of LFCO and GO and performed the OER measurements. The OER performance of the simple mixture of LFCO and GO was inferior to that of LFCO$_5$. The electrochemically active surface area (ECSA) plays a vital role in understanding the OER activity of electrocatalysts. To further compare the relationship between the ECSA and the OER activity of the electrocatalyst, the ECSA of the material was investigated from the double-layer capacitance. The CV measurements at varying scan rates (150–350 mV/s) were performed in a non-Faradaic region (1.05–1.3 V vs RHE) to...
calculate the double-layer capacitances of LFCO, LFCO2.5, LFCO5, and LFCO7.5. Figure S4A–D shows the plots of scan rate-dependent cyclic voltammograms, and Figure 7A–D shows the plot of $\Delta j = j_a - j_c$ at 1.15 V against the scan rates. The slope of LFCO5 ($3.67 \times 10^{-4}$) was higher than those of LFCO ($2.81 \times 10^{-4}$), LFCO2.5 ($1.5 \times 10^{-4}$), and LFCO7.5 ($1.72 \times 10^{-4}$), which indicates that the LFCO5 has a large surface area.

To evaluate the electron-transfer resistance to understand the electrocatalytic properties of LFCO, LFCO2.5, LFCO5, and LFCO7.5, electrochemical impedance spectroscopy (EIS) was performed. The FRA analysis was conducted at an onset potential value of 1.55 V. The Nyquist plot and circuit diagram are shown in Figure 8A. This figure shows that the LFCO5 has low charge-transfer resistance and faster charge-transfer kinetics than pristine LFCO, LFCO2.5, and LFCO7.5, which indicates the superior charge transport kinetics. This result proves that LFCO5 has an increased conductivity compared to LFCO, and it promotes the electrocatalytic activity. Figure 8B shows the mechanism of OER in the alkaline medium. The Tafel slope is another parameter used to evaluate the electrochemical activity and kinetics of OER. The materials with decreasing Tafel slope indicate the improvement in the catalytic activity. Figure 9A shows that LFCO5 exhibited a lower Tafel slope of 49.1 mV/dec in 1 M KOH, which is lower than that of LFCO (93 mV/dec), LFCO2.5 (89 mV/dec), and LFCO7.5 (75.1 mV/dec). The lower Tafel slope of LFCO5 indicates that it has faster electrochemical kinetics. The long-term stability of electrodes is very essential for practical application. As shown in Figure 9B, the stability was tested by the chronopotentiometry method under a constant current density of 10 mA/cm² for the most active catalyst LFCO5 in 1 M KOH. After applying a constant current for 10 h, the potential almost remains constant.
unchanged, suggesting a stable performance and long-term viability of LFCOs in alkaline solution. We also compared the Tafel slope of different perovskite catalysts, and the comparison is given in Figure 10. The small Tafel slope, high current density at lower overpotential, lower charge-transfer resistance, and stable performance make the LFCO5 a promising electrode for OER.

![Figure 10. Comparison of the Tafel slope of the LFCO5 catalyst with other perovskites in the literature.](image)

3. CONCLUSIONS

In summary, we synthesized porous LaFe_{0.8}Co_{0.2}O_{3} doped with different amounts of carbon and evaluated the OER activity of the samples. The experimentally determined OER activity of the porous carbon-doped LaFe_{0.8}Co_{0.2}O_{3} was significantly better than the undoped LaFe_{0.8}Co_{0.2}O_{3}. The incorporation of different percentages of GO during the synthesis of LaFe_{0.8}Co_{0.2}O_{3} enhances the porosity of the synthesized material and minimizes the conductivity issues. The catalyst ink preparation showed the easy dispersion formation for LFCO2.5, LFCO5, and LFCO7.5 compared to LFCO. The LFCO shows a lower overpotential of 0.41 V compared to LFCO (0.46 V), LFCO2.5 (0.45 V), and LFCO7.5 (0.42 V) at a current density of 10 mA/cm². The Tafel slope of LFCO5 (49.1 mV dec⁻¹) was also small compared to other electrocatalysts used in this study. The high ECSA, low charge-transfer resistance, easy interconversion of the cobalt spin state in the octahedral environment, interconnected porous structure, B-site exposed grains, and the synergic effect between the Co³⁺ and Fe³⁺ ions enhanced the electrocatalytic activity of the LFCO5 toward OER. Importantly, the excellent stability, catalytic activity, and the conductivity of the LFCO opens up a new synthesis strategy for excellent OER catalysts.

4. EXPERIMENTAL SECTION

The entire chemicals used in this work were used without further purification. Lanthanum nitrate, ferric nitrate, cobalt nitrate, glycine, isopropanol, and potassium hydroxide were purchased from Merck. Nafion was purchased from Sigma-Aldrich, USA. The ultrapure type 1 water from Millipore is used for all the materials. The ultrapure type 1 water from Millipore is used for all the materials. Graphite oxide was prepared in our laboratory by modified Hummers’ method.²⁶

4.1. Preparation of LaFe_{0.8}Co_{0.2}O_{3}/Carbon Hybrid Material. LaFe_{0.8}Co_{0.2}O_{3} and LaFe_{0}Co_{2}O_{3}/carbon hybrid materials were synthesized by the solution combustion method. For the preparation of LaFe_{0.8}Co_{0.2}O_{3}, the stoichiometric amounts of 0.005 moles of lanthanum nitrate, ferric nitrate, cobalt nitrate, and glycine were dissolved in a minimum quantity of water with a fuel to oxidizer ratio of 1. The solution was stirred for 30 min to obtain a homogeneous mixture. The mixture was placed in a hot magnetic stirrer at 200 °C, and a sticky viscous solution was obtained. At this stage, the magnetic bead was removed and temperature was raised to 300 °C till the combustion reaction became complete. The obtained powder was ground well and calcined at 700 °C for 5 h (LFCO). Different amounts of (2.5, 5, and 7.5 wt %) graphite oxide was sonicated in a minimum quantity of water and added to a mixture of lanthanum nitrate, ferric nitrate, cobalt nitrate, and glycine (0.005 mol) taken in a separate beakers for synthesizing LaFe_{0.8}Co_{0.2}O_{3}/carbon hybrid materials. The mixture undergoes combustion at 300 °C, and the powder was calcined at 700 °C for 5 h. Based on the different weight percentages of GO, the samples were labeled as LFCO2.5, LFCO5, and LFCO7.5 for 2.5, 5, and 7 wt %, respectively.

4.2. Preparation of the Electrode. The catalyst ink was prepared by sonicating 1:3 volume ratio of isopropanol and water for 5 min. To this solution, 5 mg of catalyst (LFCO, LFCO2.5, LFCO5, and LFCO7.5) was added and sonicated for 30 min. Then, 5 μL of nafion was added and sonicated for 5 min. The glassy carbon electrode was cleaned properly by using alumina powder. Catalyst ink (5 μL) was drop-casted into the 3 mm glassy carbon electrode and dried at room temperature. This electrode was used as a working electrode for electrochemical studies.

4.3. Characterization. The powder X-ray diffractions of the prepared LFCO, LFCO2.5, LFCO5, and LFCO7.5 nanoparticles were recorded using a X-ray diffractometer (Rigaku Miniflex 600, Cu Kα) at a 1.54056 Å wavelength. Raman spectra of the materials were analyzed by a micro-Raman spectrometer, HORIBA France, LABRAM HR Evolution, with a 633 nm excitation laser. ATR spectra of the synthesized materials were recorded using an Agilent Technologies Cary 630 FT-IR spectrometer with the ATR technique. Thermal stability was analyzed using a STA445 Coincidence Thermal Analyzer NETZSCH with a chemical balance of Shimadzu AX200. The phase composition and morphology of the material were studied by using a JEOL SEM 5600 LV instrument using gold sputtering. The elemental compositions of the synthesized materials were studied using elemental mapping and EDX. The particle size, elemental mapping, and EDX of the LFCO5 were studied using TEM. The HR-TEM image, SAED pattern, HAADF elemental mapping, and EDX of LFCO were examined by Thermofisher TEM TALOS F200SG2 200 KV with an ultrabright FEG gun with a 4 K X 4 K CMOS camera, column EDS detector. Elemental analysis was performed using the ICP–AES model JY2000 of Jobin Yvon make.

4.4. Electrochemical Measurements. Electrochemical studies were performed using Autolab potentiostat/galvanostat (Metrohm Autolab 302N with NOVA 2.1.4). The electrochemical experiments were performed using a three-electrode system. The working electrode as a perovskite catalyst coated with 3 mm glassy carbon electrode coupled with RDE was set up. Platinum wire and Ag/AgCl electrode were used as the counter and reference electrodes, respectively. Electrochemical impedance studies were performed with the FRA module. The electrolyte used in this study was 1 M KOH solution. The ECSA was calculated by using a cyclic voltammetric technique in the non-Faradic region (1.05–1.3 V vs RHE) with a scan rate of 150–350 mV/s. The linear sweep voltammetry (LSV) and hydrodynamic LSV (HLSV) were conducted in a potential window of 0.9–1.9 V (vs RHE) with a scan rate of 5 mV/s. The
potentials in LSV and HLSV are converted to RHE for better comparison of the reported values.

## ASSOCIATED CONTENT

#### Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c02074.

Raman spectrum, elemental mapping, HR-TEM, CV at different scan rates, and linear sweep voltammetry of LFCOS (PDF)

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### Notes

The authors declare no competing financial interest.

### ACKNOWLEDGMENTS

J.T. thankfully acknowledges the financial support from Kannur University as a Junior Research Fellow (JRF). This work has also been supported by the DST-FIST (SR/FST/College-289/2015), KSCSTE-SARD (KSCSTE/S253/2017/SARD), and DBT Star strengthening Grant (F. No. BT/HRD/11/01/2019) programs of Nirmalagiri College, Nirmalagiri, India. We acknowledge SRM institute of Science and Technology for providing "micro-Raman FACILITY".

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