

Characterization of $\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-\alpha}$ cathode powder prepared by a combined citrate-EDTA method

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Abstract

The $\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-\alpha}$ (LSCO) cathode powder was prepared by a combined citrate-EDTA method using nitrate based chemicals. Citric (CA) and ethylenediaminetetraacetic acid (EDTA) were used as chelating agents and ethylene glycol (EG) as a polymerizing agent. The formation of metal complexes of LSCO was analyzed using Fourier transform infrared (FTIR) spectroscopy. Various organic compounds such as carbonyl and carbonate groups were observed in FTIR spectra. The as-synthesized LSCO powder analyzed by thermogravimetric analysis (TGA) showed there were three stages of weight losses with a drastic weight loss observed between 150-550 °C. A complete decomposition of intermediate compounds was observed at 800 °C indicating the formation of metal oxide was formed. This result was further confirmed by X-ray diffraction (XRD) analysis which revealed the calcined LSCO powders at 800 °C and 900 °C formed a single perovskite phase. LSCO cathode slurry was prepared by adding polyvinyl pyrrolidone (PVP) as binder. This slurry was painted on pellet surfaces of $\text{BaCe}_{0.54}\text{Zr}_{0.36}\text{Y}_{0.1}\text{O}_{2.95}$ (BCZY) electrolyte. Scanning electron microscopy (SEM) results showed the LSCO cathode was well adhered on BCZY electrolyte. It is apparent that the binder plays an important role in making a good contact between the obtained high purity LSCO cathode and BCZY electrolyte.

Keywords: $\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-\alpha}$, $\text{BaCe}_{0.54}\text{Zr}_{0.36}\text{Y}_{0.1}\text{O}_{2.95}$, combined citrate-EDTA, PVP binder

Introduction

The need for clean and efficient energy conversion devices brings to the new level in development of fuel cells. Fuel cell is a device that converts chemical energy directly into electrical energy via electrochemical reactions. It is deemed to play an important role in future clean power generation. Among various kinds of fuel cells, solid oxide fuel cell (SOFC) get the most attention as it offers the highest energy conversion efficiency, low pollutant emission and excellent fuel flexibility but, high processing costs and operating temperatures hinder it for a broad commercialization. Currently, tremendous efforts directed towards reducing high operating temperatures to intermediate temperatures (IT) (500-800 °C) have been extensively done in order to accelerate the commercialization of SOFC. At these IT, it has been found that increasing in cathode polarization tends to limit the cell performance. Thus optimization of electrode materials especially

cathode part is essential as the performance of IT-SOFC strongly depends on cathode-electrolyte interface (Xu et al., 2005; Qiang et al., 2009; Sun et al., 2010).

A few approaches can be adopted to reduce the cathode polarization including by using materials which based on mixed ionic-electronic conductor (MIEC) as cathode material. They extend the active oxygen reduction sites from the typical electrode/electrolyte-gas triple-phase boundary (TPB) to the entire cathode surface, therefore greatly reducing the cathode polarization at IT. Conventionally, perovskite strontium-doped lanthanum manganite (LSM) was used as cathode material for SOFC. However, it is not suitable to be used as IT-SOFC cathode due to its low catalytic activity below 800 °C. Thus, new potential cathode candidates for IT-SOFC based on MIEC materials are being developed. One of the most promising materials is strontium-doped lanthanum cobalt oxide, $\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-\alpha}$ (LSCO) as it has shown very low polarization resistance, high electronic

conductivity and good catalytic activity at IT (Chen et al., 2003; Berenov et al., 2010; Fu, 2010).

LSCO powder has been synthesized by numerous methods including solid-state reaction (SSR), mechanical-synthesis, co-precipitation, solution combustion and Pechini methods. Among them, Pechini or modified-Pechini method has received a great attention. There are two basic chemical reactions involve in this method namely (i) complexation or chelation between metal ions and chelating agents and (ii) poly-esterification of complexes with ethylene glycol (EG). These reactions preserve the homogeneity of the metal salts in the solution into the gel. Here, chelating agents such as ethylenediaminetetraacetic acid (EDTA), citric acid (CA) and a polyalcohol play an important role in the evolution of crystalline phase and properties of final product. The advantages of this method are the high purity powder can be obtained at relatively low temperatures and excellent stoichiometry control (Tao et al., 2008; Shao et al., 2012).

Another approach to reduce the cathode polarization is the selection of additives such as electrode binder in optimizing the cathode structure or addition an ionically conducting second phase to form a composite cathode (Qiang et al., 2009). An electrode binder is usually a polymer. This polymer helps to keep the cathode materials bind onto electrolyte surfaces (Choudhury et al., 2011). The contact properties at electrode-electrolyte interface such as formation reaction products, interdiffusion, segregation and electronic structure changes will affect the oxygen exchange rates and impact the resulting IT-SOFC performance (Backhaus-Ricoult, 2008). Ethyl cellulose (EC) is the conventional electrode binder used in LSM cathode slurry. However, other polymers including polyvinyl pyrrolidone (PVP) are seldom served as electrode binder for cathode slurry. PVP binder is inexpensive and can further the manufacturing cost.

In this paper, LSCO cathode powder was prepared by a combined citrate-EDTA method. The conventional citrate-EDTA method is usually performed in an alkaline condition but in this present study, it was performed in acidic condition. A small amount of ammonium hydroxide is introduced into the precursor solution to aid pH adjustment and dissolution of metal salts. The reduction of the amount of ammonia solution used hinders the emission of some toxic gases such as NO_x during pre-heat treatment process. Besides that, the combined chelating agents used can further reduce the sensitivity of the synthesis process to the pH than the single chelating agent adopted in Pechini method. Additionally, combined chelating agents are also help to reduce the temperature requires for phase formation. The use of PVP binder to aid in making good contact between LSCO and $\text{BaCe}_{0.54}\text{Zr}_{0.36}\text{Y}_{0.1}\text{O}_{2.95}$ (BCZY) electrolyte is also discussed.

Materials and Method

Synthesis of LSCO Powder

The LSCO cathode powder was prepared by a combined citrate-EDTA method using $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (99.999% purity, ACROS), $\text{Sr}(\text{NO}_3)_2$ (99+% purity, ACROS), $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (99+% purity, ACROS), citric acid monohydrate (CA) (99.5% purity, MERCK), ethylenediaminetetraacetic acid (EDTA) (99% purity, ACROS), ethylene glycol (EG) (99.97% purity, ACROS), ammonium hydroxide solution (25% assay, HmbG®) and deionized water. Metal nitrates were employed both as metal precursors and oxidizing agents. CA and EDTA were used as a combined chelating agent to chelate metal cations while EG was used as surfactant for polymerization process. To yield 5 g of the final LSCO powder, the precursor solution was prepared by mixing a stoichiometric amount of the metal nitrate salts and dissolved in 100 mL deionized water. The red color of the precursor solution was stirred and heated in a water bath at 75 °C for an hour. Calculated amounts of CA and EDTA with molar ratio to overall metal cations content of 1.5:0.5:1 were added into the solution. The color of the solution turned to transparent red and dark violet color solution after CA and EDTA were added, respectively. After that, the pH of the solution was adjusted to be 0.5 by adding appropriate amount of ammonia hydroxide solution. Then, EG with molar ratio to overall metal cations content of 3:1 was added and the solution was continuously stirred and heated for several hours to evaporate the water. Next, the resulting viscous gel was dried at 150 °C for 12 hours and 250 °C for 5 hours. The obtained as-synthesized powder was calcined at respective temperature of 800 °C, 900 °C and 1000 °C with heating/cooling rate of 5 °C min⁻¹ for 5 hours to yield black powder. A flow chart showing the various steps involved in the synthesis of LSCO powder by a combined citrate-EDTA method is shown in Figure 1.

Fabrication of symmetrical cell

A dense BCZY electrolyte pellet was prepared as previously reported by Osman et al. (2009). About 0.65 g of BCZY powder was compacted at 5 tons pressure using a hydraulic press in a mould of 13 mm in diameter. The obtained green BCZY pellet was sintered at 1400 °C for 10 hours. Both sides of the sintered BCZY pellet were polished with 1000# SiC grit paper and ultrasonically cleaned. The pure phase powder of LSCO calcined at 800 °C was mixed in ethanol solvent with ratio of 1:2. PVP binder with ratio to LSCO powder of 1:10 was added into the mixture and stirred for 30 minutes. Then, the cathode slurry was homogeneously dispersed by an ultrasonic. The

obtained homogeneous cathode slurry was painted on both sides of the sintered and polished BCZY pellet and the symmetrical cell was first heated up at 500 °C to eliminate the binder and then to 950 °C for 2 hours. Next, the cell was cross-sectioned and mounted in a sample holder using epoxy resin. The mounted cell was polished using 1200#, 1500# and 2000# SiC grit papers. Contamination on the surface was ultrasonically cleaned in ultrasonic water bath after each step. Afterward, the cell was polished using 6 µm diamond polish to ensure a scratch free surface. The polishing process was finished by using 1 µm diamond polish and the sample was dried in air for few hours. Finally the sample was etched in 0.2 mol dm⁻³ nitric acid, HNO₃ for 30 seconds. The same procedure was also applied for the sintered BCZY pellet painted with platinum (Pt) ink which serves as standard sample. The BCZY pellets painted with LSCO and Pt ink were denoted as LSCO/BCZY/LSCO and Pt/BCZY/Pt, respectively.

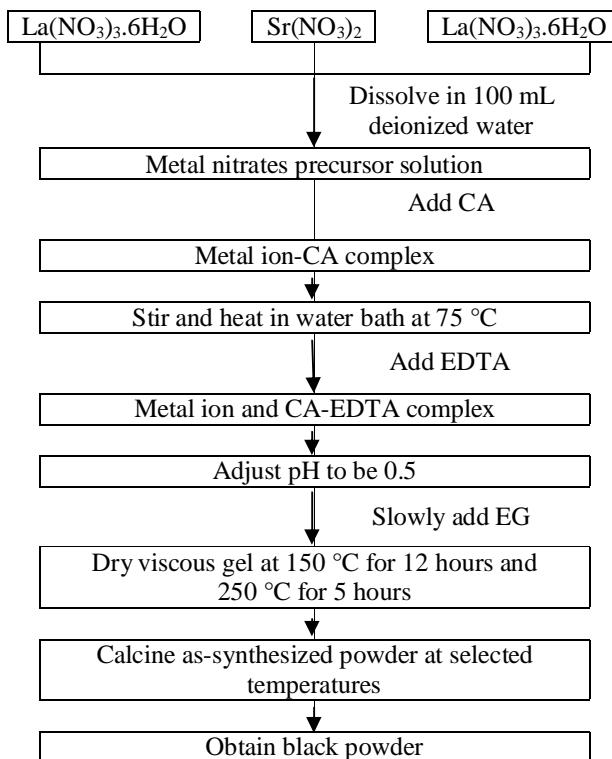


Figure 1. Steps of LSCO powder synthesis by a combined citrate-EDTA method

Characterization

Thermogravimetric study of the gel dried at 150 °C was carried out using a Thermal Analysis (TA) instrument (model SDT Q600) in a flow of synthetic air (flow rate of 100 mL min⁻¹) under heating/cooling rate of 5 °C min⁻¹ in an alumina pan. The temperature range was

between room temperature and 1000 °C. The weight changes were recorded as a function of temperature. Fourier transforms infrared (FTIR) spectra of uncalcined precursors and calcined LSCO powders were obtained using Nicolet FTIR spectrometer (Thermo Electron Corporation) at room temperature. The spectra of various organic complexes present were registered from 4000-400 cm⁻¹ by averaging 32 scans with a resolution of 4 cm⁻¹ for each spectrum. X-ray diffractometry study was carried out on an X-ray diffractometer (XRD 6000 Shimadzu) using a monochromatic CuK_α radiation source ($\lambda = 0.1540558$ nm) with Ni-filtered in order to examine phase formation of the calcined LSCO powders. The X-ray diffractometer was operating at 40 kV and 30 mA using a step procedure of 0.02° s⁻¹ for 20 range from 20° to 80°. The percentage of perovskite phase present in the calcined LSCO powders was calculated using following equation (Wongmaneerung et al., 2009):

$$\text{Perovskite phase (wt\%)} = \left[\frac{I_p}{I_p + I_m} \right] \times 100\%$$

where I_p and I_m refer to the maximum intensity of the perovskite and impurities phase, respectively. Scanning electron micrographs of the Pt/BCZY/Pt and LSCO/BCZY/LSCO interfaces were obtained using a JEOL JSM-6460 LA Scanning Electron Microscope (SEM) operating at 15 kV. SEM study was carried out at magnification of 2000 times using backscatter electron mode.

Results and Discussion

Thermal analysis

Figure 2 depicts the TG curve of the gel precursor dried at 150 °C obtained at heating/cooling rate of 5 °C min⁻¹ in air from 25 °C to 1000 °C. The TG curve showed there were three stages of the weight loss between 25-150 °C, 150-550 °C and 550-800 °C. A small weight loss of about 1.8% at the first temperature region up to 150 °C (A) is resulted from dehydration of water, alcohol and decomposition of nitrates. Since the boiling point of water is 100 °C, all the moisture is decomposed at this stage. The drastic weight loss which is about 71.18% observed at 150-550 °C (B) is corresponded to the decomposition of metal-chelate complex. The decomposition results into releasing of water (H₂O), carbon dioxide (CO₂) and NO_x gaseous during heating as the chelating and polymerizing agents used are the sources of carbon, hydrogen and nitrogen. In the third region at 550-800 °C (C), TG curve indicates weight loss of 18.49% due to the decomposition of carbonate species to yield oxide

compound. From 800-1000 °C, TG curve almost flatten indicating the possibility of oxide phase is formed. The results are also in agreement with the work done by Doorn et al. (1998) and Tao et al. (2008). The percentages of weight loss at each stage are tabulated in Table 1.

FTIR analysis

Table 2 is summary of the assignments of peak locations for each group present in FTIR spectra of various organic complexes presence in the precursors and calcined LSCO powders in Figure 3. From the FTIR spectra, the peaks around 3400 cm^{-1} and 1640 cm^{-1} appear in all spectra are characteristic of absorbed water or hydroxyl group in alcohol, O-H stretching and O-H bending, respectively. From Figure 3(a), formation of metal complexes with chelating and polymerizing agents during synthesizing process can be observed. When stoichiometric amounts of metal nitrate salts were dissolved in deionized water, a peak at 1348.41 cm^{-1} appeared. This peak represents the nitrate group that contains in each of the metal salt (C-N stretching). It also shows the peaks in the vicinities of 1700 cm^{-1} and 1200 cm^{-1} which can be attributed to monodentate ligand of metal with carbonyl groups (COO^-) after citric acid and EDTA were added. With one molecule of citric acid, one metal ion can be chelated by three bonds and with one molecule of EDTA; one metal ion can be chelated by six bonds (Galceran et al., 2007). After EG was added, some additional peaks appeared at 1083.70 cm^{-1} , 1039.58 cm^{-1} and 876.14 cm^{-1} are attributed to the C-C-O structure from ethylene glycol in the polymerization process. These peaks are also reveal the existence of carbonate species (Yang et al., 2005).

Figure 3(b) shows the FTIR spectra of as-synthesized LSCO precursor heated at $150\text{ }^\circ\text{C}$ and calcined LSCO powders at 800 , 900 and $1000\text{ }^\circ\text{C}$. As seen from it, citric acid, EDTA and EG have been completely destroyed during the calcination process since no characteristics peaks of them were found in the powders calcined at $800\text{ }^\circ\text{C}$ and $900\text{ }^\circ\text{C}$ as compared to the spectrum of the as-synthesized LSCO powder heated at $150\text{ }^\circ\text{C}$. However, the powder calcined at $1000\text{ }^\circ\text{C}$ still possesses the peaks. The small peaks appear at 550 cm^{-1} and 450 cm^{-1} are mainly due to formation of metal oxide bond (Yang et al., 2005 and Nityanand et al., 2011).

Table 1. Weight loss percentage of as-synthesized LSCO dried at $150\text{ }^\circ\text{C}$

Stage	Temperature range ($^\circ\text{C}$)	Weight loss (%)
1	$25 - 150$	1.78
2	$150 - 550$	71.18
3	$550 - 800$	18.49

Table 2. Peak locations and assignments for the various vibration modes present in the precursors and calcined LSCO powders

Peak location (cm^{-1})	Peak assignment
$\sim 3380 - 3450$	O – H stretching
$\sim 1630 - 1660$	O – H bending
$\sim 1348 - 1470$	C – N stretching, NO_3^-
~ 1700 and ~ 1200	COO^- (carbonyl group) stretching
$\sim 1000 - 1100$	C – C – O bond stretching
$\sim 840 - 930$	CO_3^{2-} (carbonate group)
$\sim 450 - 550$	M – O (metal oxide bond) stretching

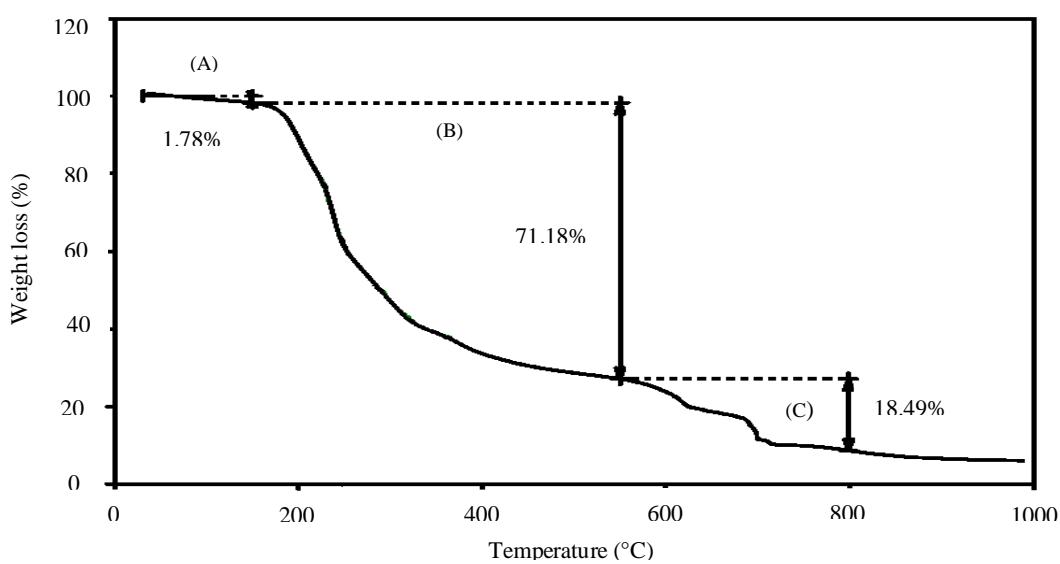


Figure 2. TG curve of the as-synthesized LSCO precursor heated at $150\text{ }^\circ\text{C}$

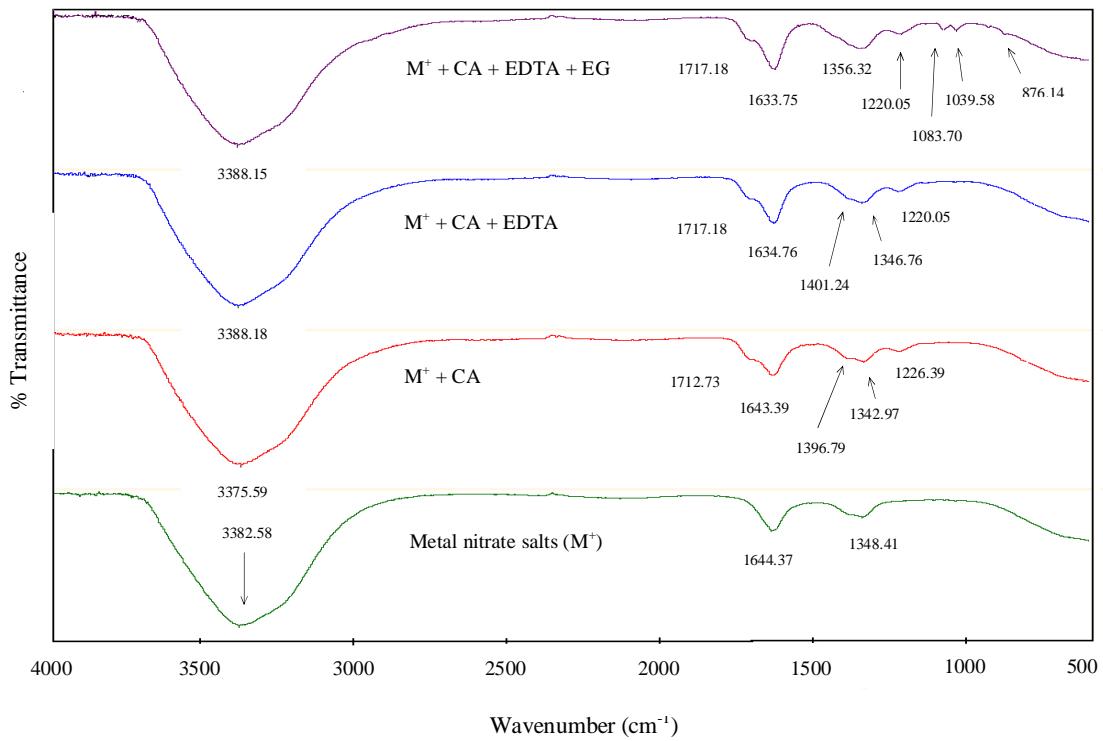


Figure 3(a). FTIR spectra of various organic complexes presence during synthesis

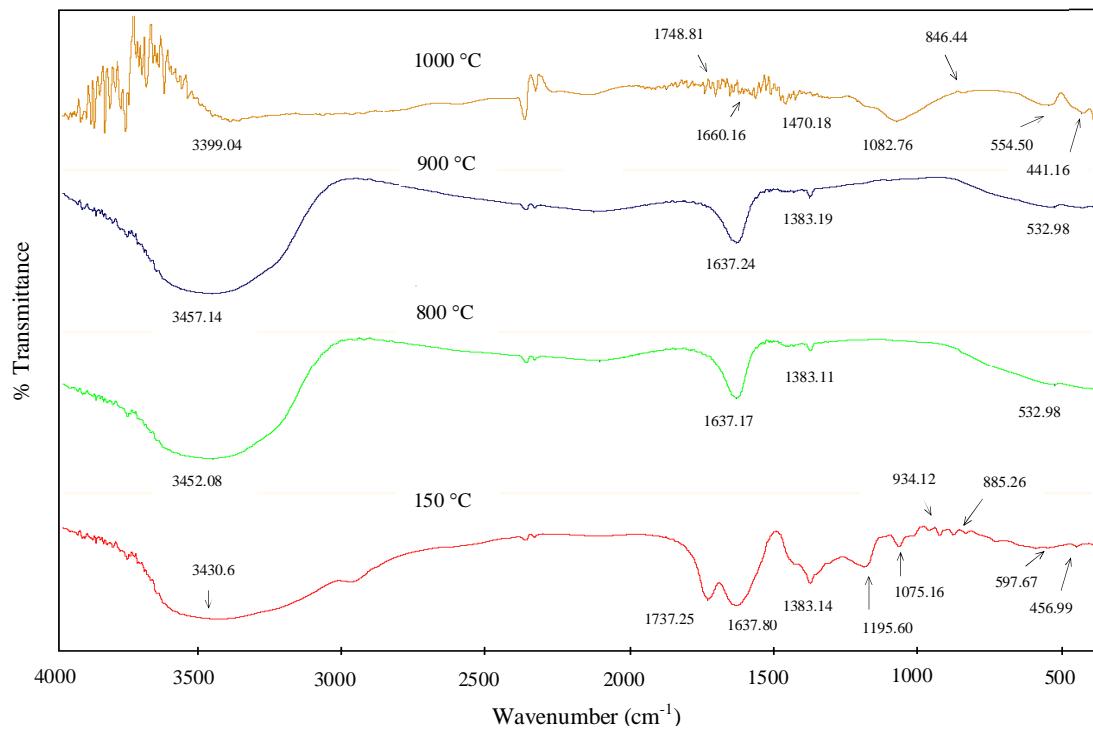


Figure 3(a). FTIR spectra of various organic complexes presence during synthesis

XRD analysis

Figure 4 shows the XRD patterns of LSCO powders calcined at three calcination temperatures for 5 hours at heating/cooling rate of $5\text{ }^{\circ}\text{C min}^{-1}$. Intensity of the peaks in all XRD spectra is increased as the calcination temperatures increased. The strongest reflections of the XRD patterns that matched with JCPDS file 48-0121 were found in all spectra which indicate the formation of perovskite phase. The presence of some additional reflections is also observed in some XRD spectrum. These reflections correlate to trace of impurity phases such as cobalt oxide (CoO) (JCPDS file 43-1004), strontium oxide (SrO) (JCPDS file 6-0520) and strontium cobalt oxide (SrCoO_x) (JCPDS file 49-0692). Based on the XRD patterns, it is found that a single perovskite phase of LSCO was obtained at $800\text{ }^{\circ}\text{C}$ and $900\text{ }^{\circ}\text{C}$. The result is in line with the TGA results as previously discussed which revealed that a complete decomposition of intermediate compounds was found at $800\text{ }^{\circ}\text{C}$. However, when the calcination temperature increased to $1000\text{ }^{\circ}\text{C}$, an amount of impurity phases such as CoO , SrO and SrCoO_x appeared. This indicates the decomposition of LSCO perovskite phase as the powder was calcined above the optimum condition (Kashif *et al.*, 2009). It also explained why the various organic complexes are still present as observed in the FTIR spectra. The XRD results and calculated percentages of the perovskite and impurity phases formed are presented in Table 3.

Table 3. Summary of XRD analysis of LSCO powders after calcined at selected temperatures

Calcination temp. ($^{\circ}\text{C}$)	Crystalline phase	Amount (wt%)	
		Perovskite	Impurity
800	$\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-\alpha}$	100.00	-
900	$\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-\alpha}$	100.00	-
1000	$\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-\alpha}$ SrO, CoO	72.00	28.00

SEM analysis

Figure 5 shows the SEM micrograph of the surface contact between Pt/BCZY electrolyte and LSCO cathode/BCZY electrolyte. The SEM analysis was performed on a half-cell from a cross-sectional view. Three distinct regions corresponding to the Pt, LSCO cathode and BCZY electrolyte can be clearly seen from the SEM micrograph. Figure 5(a) depicts the contact formation between platinum and BCZY electrolyte which served as reference electrode since it is a pure electronic conductor due to its high catalytic and stability in oxidative and reductive atmosphere. It is observed that platinum does not well adheres onto the electrolyte surface because they are in different class of materials which are metal and ceramic. The joint between metal-ceramic is difficult to bring in a good contact unless appropriate parameters such as temperature, time and pressure are provided (Allen & Borbridge, 1983 and Tomsia, 1993).

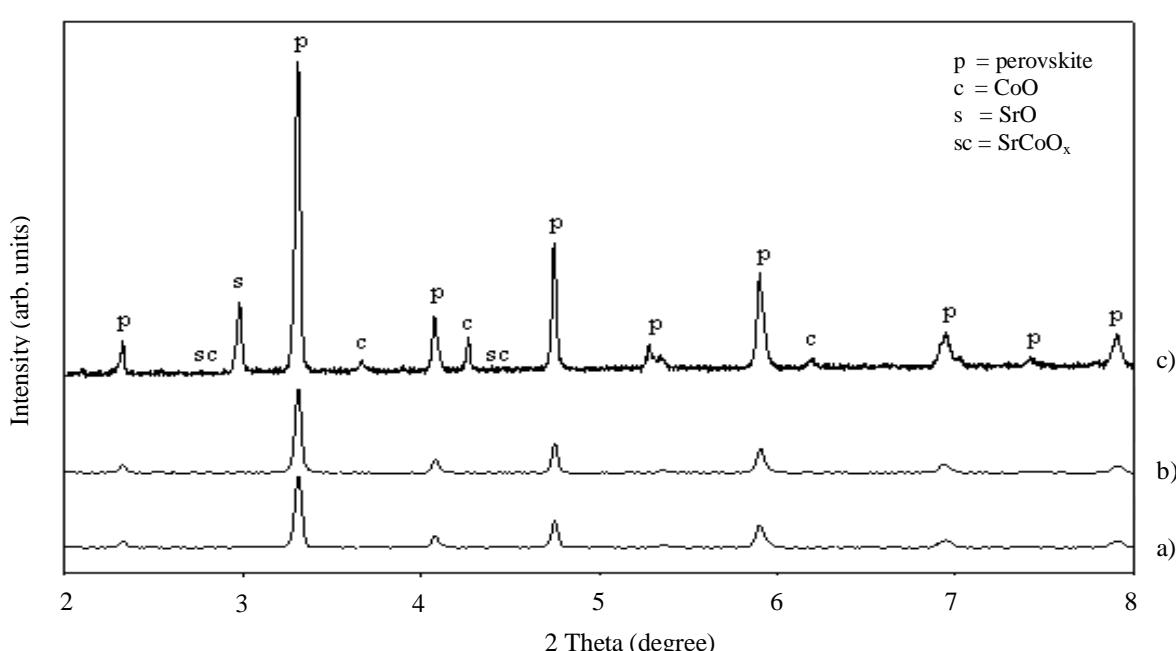


Figure 4. XRD patterns of LSCO powders calcined at temperatures: $800\text{ }^{\circ}\text{C}$ (a); $900\text{ }^{\circ}\text{C}$ (b) and $1000\text{ }^{\circ}\text{C}$ (c)

The LSCO cathode was well knitted with BCZY electrolyte as observed in Figure 3(b) using polyvinyl pyrrolidone (PVP) as a binder. PVP has amide group in its structure. The available lone electron pairs of oxygen and nitrogen atoms in their structures can interact with the metal ions in the LSCO and BCZY materials. During the early stage of evaporation process of solvent which is the ethanol, metal ions in both materials adsorb on the binder surface. After the cell was first heated up to 500 °C, the PVP binder was burnt out as its melting point is about 150-180 °C and the aggregation at cathode/electrolyte interface took place (Shao *et al.*, 2000). It is apparent that the used of binder has significant effect in making a contact between LSCO cathode and BCZY electrolyte

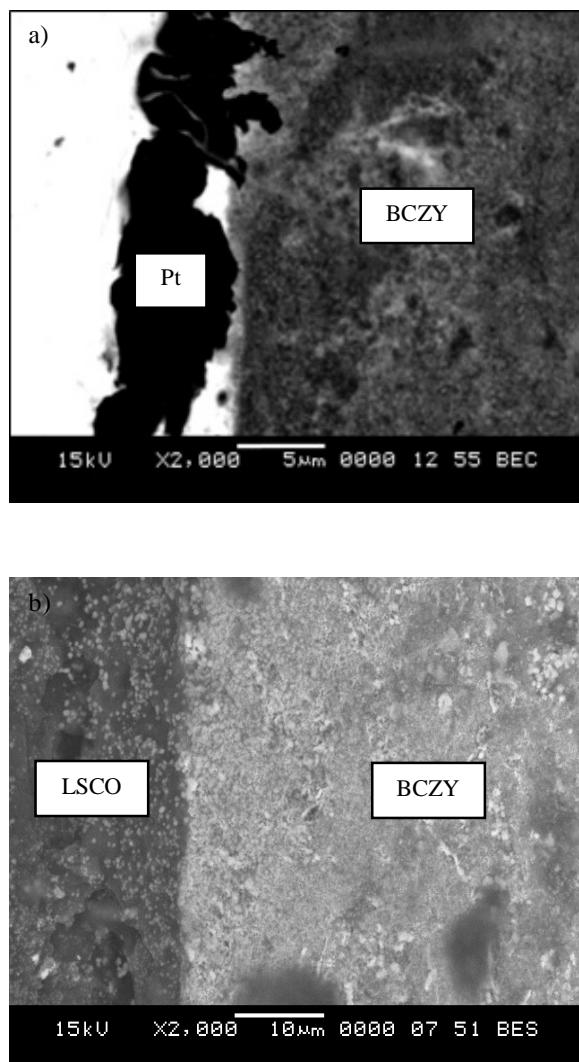


Figure 5: Cross-sectional view of SEM micrograph at interface between Pt/BCZY (a) and between LSCO/BCZY (b)

Conclusions

The $\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-\alpha}$ (LSCO) cathode powder was synthesized by a combined citrate-EDTA method. Based on TGA result, a complete decomposition of intermediate compounds was found at 800 °C. XRD analysis confirmed that the obtained LSCO powder is a single perovskite phase after calcined at temperatures of 800 °C and 900 °C. The PVP binder which is an electrode binder observed to help in making good contact between LSCO and BCZY electrolyte. Further works on the effects of using various electrode binders in forming good contact between LSCO and BCZY have been extensively studied and the progress will be reported elsewhere.

Acknowledgment

This research was funded by Excellent Fund Grant from Universiti Teknologi MARA and Fundamental Research Grant Scheme (FRGS) from the Malaysian Ministry of Higher Education.

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