

Synthesis and Characterization of Zn-doped LiCoO_2 Material Prepared via Glycine-nitrate Combustion Method for Proton Conducting Solid Oxide Fuel Cell Application

(Sintesis dan Pencirian LiCoO_2 Terdop Zn Melalui Kaedah Pembakaran Glisina- Nitrat untuk Aplikasi Sel Fuel Oksida Pepejal Konduktor Proton)

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ABSTRACT

LiCo₂-based materials are well-known, widely used as cathode materials in lithium ion batteries and currently are also used in low temperature proton conducting solid oxide fuel cells (H⁺-SOFCs) application. Dopants such as Zn are introduced in LiCo₂-based materials to improve the properties and performance of the materials for H⁺-SOF application. In this study, Zn-doped LiCo₂, LiCo_{0.6}Zn_{0.4}O₂ (LCZO) powder was synthesized via glycine-nitrate combustion method followed by various characterizations. The precursor LCZO powder dried at 100°C was subjected to thermogravimetric analysis (TGA). The phase formation and morphology of the calcined LCZO powder at 600°C were examined by an X-ray diffractometer (XRD) and a tabletop scanning electron microscope (SEM), respectively. The TGA result revealed that the thermal decomposition of the intermediate compounds in the precursor LCZO powder was completed at 800°C through three main phases of weight losses. A pure phase of LCZO was not completely produced after the calcination at 600°C due to the presence of secondary phases as confirmed from the XRD analysis. The identified secondary phases that present are confirmed as ZnCo₂O₄ and ZnO as documented in JCPDS file no. 00-001-1149 and JCPDS file no. 01-079-0205 consecutively. The SEM images showed that the impure calcined LCZO powder possessed homogeneous and fine particles.

Keywords: Solid Oxide Fuel Cell; LiCo_{0.6}Zn_{0.4}O₂ cathode; Glycine-nitrate combustion method; single phase

ABSTRAK

Bahan berasaskan LiCo₂ merupakan salah satu bahan yang kerap digunakan dalam aplikasi bateri ion litium dan kini bahan tersebut telah mula diaplikasikan untuk kegunaan sel fuel oksida pepejal kekonduksian proton (proton conducting solid oxide fuel cell, H⁺-SOFC). Penggunaan bahan dopan seperti Zn diperkenalkan ke dalam bahan asas LiCo₂ untuk meningkatkan sifat dan prestasi terhadap bahan tersebut untuk aplikasi H⁺-SOFC. Dalam kajian ini, Zn terdop LiCo₂, LiCo_{0.6}Zn_{0.4}O₂ (LCZO) telah disintesis melalui kaedah pembakaran glisina nitrat diikuti pelbagai pencirian. Pencirian termogravimetrik (TGA) telah dijalankan terhadap serbuk awalan LCZO yang telah dikeringkan pada suhu 100°C. Pembentukan fasa dan morfologi bahan LCZO yang dikalsin pada suhu 600°C dan 700°C dianalisis melalui pembelauan sinar-X (XRD) dan imej mikrograf mikroskop elektron pengimbas (SEM), masing-masing. Hasil analisis termogravimetri (TGA) menunjukkan bahawa penguraian sebatian perantara ini terurai sepenuhnya pada suhu 800°C melalui empat fasa kehilangan berat. Serbuk LCZO yang terbentuk selepas dikalsin pada suhu 600°C tidak menunjukkan ketulenan yang sepenuhnya disebabkan kemunculan fasa kedua seperti yang dibuktikan daripada analisis XRD. Kemunculan fasa kedua yang telah dikenal pasti ialah ZnCo₂O₄ dan ZnO sepertimana yang telah direkodkan dalam JCPDS file no. 00-001-1149 dan JCPDS file no. 01-079-0205 secara berturutan. Imej SEM menunjukkan bahan terkalsin LCZO adalah homogen dengan saiz partikel yang halus.

Kata kunci: Sel Fuel Oksida Pepejal ; Katod LiCo_{0.6}Zn_{0.4}O₂; Kaedah pembakaran glisina nitrat

INTRODUCTION

Solid oxide fuel cell (SOFC) is a type of electrochemical energy conversion device that possesses high energy conversion efficiency and environmental friendly (Agun et al. 2014). Due to these favorable advantages, SOFC received an undoubtedly great recognition and the exploration works

on new electrolyte and electrode materials have been done actively to improve the fuel cell life and reliability (Ding, Xie, and Xue 2011). However, the commercialization of this technology was restrained by high production cost (Baldinelli et al. 2016). The reduction of temperature is a way to reduce the high operating cost of SOFCs (Rahman et al. 2010). This approach increased the opportunity of using various cheaper

materials with improved reliability (Paydar, Shariat, and Javadpour 2016; Carpanese et al. 2017; Chen et al. 2012).

In general, SOFCs are made up of two electrodes (cathode and anode) partitioned by an electrolyte dedicated to transport oxide ions from the cathode to anode or protons from the anode to cathode (Bertei et al. 2012). In SOFCs application, the performance of cathode is very significant to improve their overall performance (Xia et al. 2016). For instances, proton conducting composite cathodes are able to assist the transportation of protons and electronic defects effectively (Xi et al. 2014). These characteristics improved the reaction sites for effective charge transfer during reduction reaction in the cathode (Sun et al. 2011). Currently, the cathode materials developed for H⁺-SOFCs are divided to single phase cathodes composed of mixed ionic and electronic conductor (MIEC) and dual phase cathodes composed (MIEC) and a proton conductor (Wang et al. 2013).

New oxide cathode materials with a good catalytic activity at lower operating temperature are critically needed to improve the overall performance of SOFCs. Recently, the lithiated transition-metal oxide materials were proposed as potential cathode materials because of their remarkable electrochemical performance (Zhang and Tao 2011). Zhang et al. showed that a single cell of NiO-GYDC|Ce_{0.8}Gd_{0.05}Y_{0.15}O_{1.9}(GYDC)|lithiated NiO-GYDC fabricated by one-step dry-pressing and co-firing at 1200°C for 4 h presented a low electrode polarization resistance (0.54 Ω cm²) at 600°C (Zhang et al. 2010). This result demonstrated that the lithiated NiO is a potential cathode material for operation with ceria-based electrolytes in IT-SOFCs. Cathodes modified by the lithiated materials such as the layered LiNi_{0.8}Co_{0.15}Al_{0.05}O₂ (Ruan, Zhu, and Teng 2016) LiNiCuZnO_x composite (Fan et al. 2013) and LiFeO₂-LiAlO₂ composite (Lan and Tao 2015) were also developed to improve the electrochemical performance of the cathodes. In addition, Tan et al. produced a modified lithiated NiO cathode, Li-Ni-M (M= Cu, Fe, Co) for low temperature SOFC application (Tan et al. 2013). It appeared that the cobalt oxide modified lithiated NiO (Li-Ni-Co) cathode showed the highest power density of 0.38 mW cm⁻² at 560°C. The most recent lithium based cathode material was layer-structured LiNi_{0.8}Co_{0.2}O₂ (LNCO) where this material possessed a triple conducting characteristic (oxygen ionic, protonic and electronic) for the application of proton conducting SOFC (Fan and Su 2016). The LNCO cathode allowed a simultaneous conduction of extrinsic proton, intrinsic oxide ion and electron in the cathode (Kim et al. 2014).

In this study, the performance of lithiated materials was improved by introducing potential dopants to the B site of ABO₂ structure while maintaining the A sites with lithium. Zinc (Zn) was selected as a dopant because it facilitated the expansion of lattice volume, formation of crystal structure and formation of extra space for lithium ion intercalation/de-intercalation in the material. On top of that, the Zn doping was found to improve the diffusion and lower the charge transfer resistance of lithium ions mainly because of the pillar effect of the doped Zn atoms (Tan et al. 2013).

To date, lithium cathode based materials are limited particularly to the H⁺-SOFC application. In the meantime, the characterization of this material has been prioritized in many studies. Thus, a clear understanding of the Zn substitution mechanism in LiCoO₂, its effect on the crystal structure of LiCoO₂ and how this structural modification is related to the electrochemistry properties of the LiCoO₂ for H⁺-SOFCs that will be studied later.

METHODOLOGY

POWDER PREPARATION

LiCo_{0.6}Zn_{0.4}CoO₂ (LCZO) powder was synthesized by glycine-nitrate combustion method. The precursor materials used in the synthesis process were lithium nitrate (LiNO₃), cobalt (II) nitrate hexahydrate (Co(NO₃)₂·6H₂O) and zinc nitrate hexahydrate (N₂O₆Zn·6H₂O). A stoichiometric amount of the precursor materials was dissolved in deionized water and stirred for 40 min on a hotplate with stirrer. After that, the calculated amount of glycine (C₂H₅NO₂) as a fuel was added into the precursor materials solution. The mixture was then continuously stirred at room temperature for another 12 h. Next, the temperature of the hotplate was accordingly raised from room temperature up to 250°C – 300°C to initiate the combustion process. Finally, the obtained black ash precursor powder was further dried at 120°C for 12 h in a drying oven. The obtained raw powder was grounded using a mortar agate to produce a fine powder with reduced agglomeration. The dried fine precursor powder was then calcined at 600°C with a heating or cooling rate of 10°C min⁻¹ in a high temperature furnace (Berkeley Scientific, USA) for 5 h.

POWDER CHARACTERIZATION

The thermal decomposition behavior analysis of the precursor LNCO powder was performed using a thermogravimetric analyzer/differential scanning calorimetry (TGA/DSC, SING Mettler Toledo, United States). The analysis was conducted from room temperature up to 1000°C at a heating rate of 10°C min⁻¹ under air with a flow rate of 50 ml min⁻¹. An X-ray diffractometer (XRD, Bruker AXS D8 Advance, Germany) with CuK_α (λ = 1.5406 Å) radiation source was used to confirm the phase and structure of the calcined powder. The XRD was operated at 40 kV, 40 mA and fitted with a 1-D fast detector (Lynx-Eye). The XRD spectrum was recorded for 2θ ranging from 20° to 90° with a step size of 0.025°. The morphology of the calcined powder was captured by a tabletop scanning electron microscope (SEM, Hitachi TM-1000, Japan).

RESULTS AND DISCUSSION

Figure 1 shows the TGA/DTG curves of the precursor powder. Three noticeable phases of weight losses are recorded in the TGA curve and corroborated with the formation of three major endothermic peaks (P1, P2 and P3) in the DTG curve.

The first phase (P1) of weight loss (7 % of weight loss) at the temperature below 100°C is mainly due to the removal of adsorbed water or moisture.

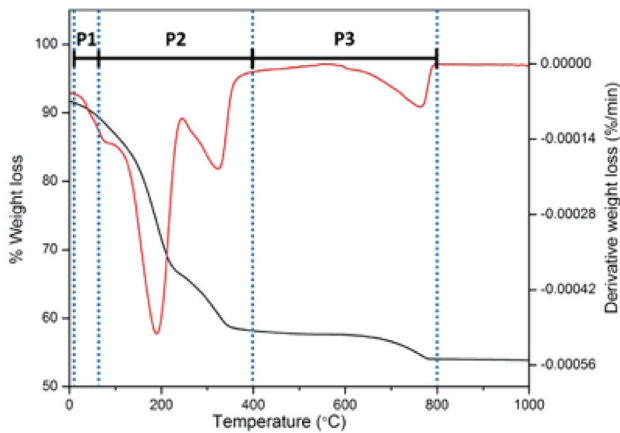


FIGURE 1. TGA/DTG curves of precursor LCZO powder calcined at the temperature of 600°C using a heating rate of 10°C min⁻¹

A well-defined step in the temperature range of 150°C to 400°C in the second phase (P2) with a weight loss of 24 % can be correlated to the decomposition of remaining nitrate species and hydroxyl ions (Ruan et al. 2016). As can be seen in P2 region, the main curve is believed due to combustion reaction of metal nitrate (in this case is Zn nitrate) and glycine while the latter curve could be due to the partial reduction of metal oxides to metal (Ashok et al. 2015). Meanwhile, a weight loss of 17 % recorded at the third phase (P3) in the temperature region of 500°C to 700°C is related to the decomposition of carbon residue which formed as intermediate compounds during the combustion reaction (Matheswaran et al. 2017). At the temperature above 700°C, the weight loss is expected due to the complete decomposition of carbon and the initiation of the formation of desired compounds. Above 800°C, the TGA and DTG curves become plateau (no endothermic peak), indicating that the required LCZO may form in this region and the decomposition process took about 2.33 hours.

From TGA analysis, the most appropriate temperature range should be above 800°C. However, this material melted at the temperature of 800°C. Thus, the temperature of 600°C was chosen as this calcination temperature was commonly used within lithium based materials (Rajammal et al. 2017 and 2016). A total weight loss of 48% is observed from the TGA and DTG for the LCZO powder and this is comparable to the materials synthesized by the same method reported in literature (Ashok et al. 2015). The XRD pattern of the calcined powder at 600°C is shown in Figure 2. The XRD pattern was matched with the Joint Committee on Power Diffraction Standard LiCoO₂ (JCPDS file no. 000-050-0653) with a rhombohedral structure (R-3m, 166). The peaks are indexed to their miller indices (*hkl*) of (101), (006), (012), (104), (015), (107), 018) and (110) accordingly. The peaks are slightly shifted to higher 2θ values is attributed to the shrinkage of lattice parameter as a result of ionic radii difference (Rajammal et al. 2016).

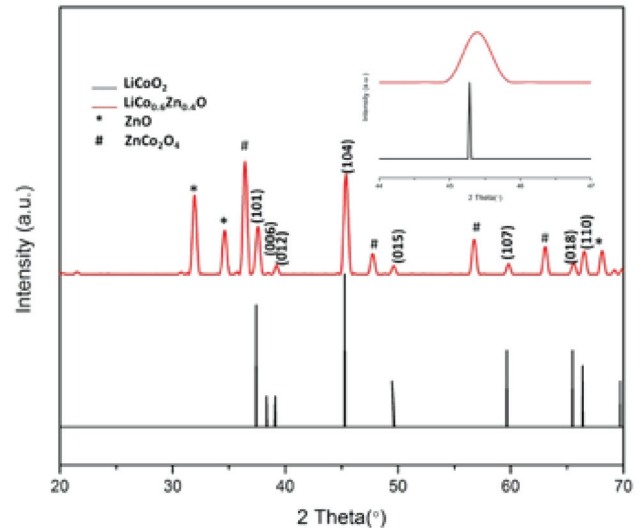


FIGURE 2. The XRD pattern of the calcined LCZO powder at 600°C for 5 h

The intensity of the diffraction peaks become broader after the doping due to the formation of smaller crystal size (Arumugam et al. 2010; Mohd et al. 2015). However, the XRD result showed that the calcined powder is impure with the formation of secondary peak such as ZnCo₂O₄ (JCPDS file no. 00-001-1149) and ZnO (JCPDS file no. 01-079-0205) as confirmed from the formation ZnCo₂O₄ and ZnO peaks (Figure 2). For ZnCo₂O₄ peaks formed, the structure can be classified in the cubic system with an Fd-3m (227) space group having the cell parameter of 8.1080 Å. On the other hand, ZnO is related to the hexagonal crystal system with a P63mc (186) space group having cell parameters of 3.2417 Å, 3.2417 Å and 5.1876 Å, respectively. In addition, the mixing time of initial solution of LCZO are determined as an important factor to obtain a pure phase material. A longer mixing time can improve the collision between the glycine and nitrate particles for homogenous combustion (Baharuddin, N et al. 2017). The SEM analysis was used to study the surface morphology of calcined LCZO powder. Figures 2 (a) and (b) below show that the LCZO powder calcined at 600°C. The figures showed that the LCZO powder is composed of homogenous and fine particles as confirmed by the XRD sharp peaks.

CONCLUSIONS

The cathode material of LiCo_{0.6}Zn_{0.4}CoO₂ (LCZO) powder for proton conducting SOFC application has been successfully synthesized by glycine-nitrate combustion method with LiCoO₂ as a precursor material. Although, the calcined LCZO powder at 600°C possessed homogeneous particles, but it showed multiple phases consisting of LiCoO₂ and impure phases of ZnCo₂O₄ and ZnO. A pure single phase of LiCo_{0.6}Zn_{0.4}CoO₂ cannot be achieved in this study as a result of low calcination temperature and lack of mixing time. The

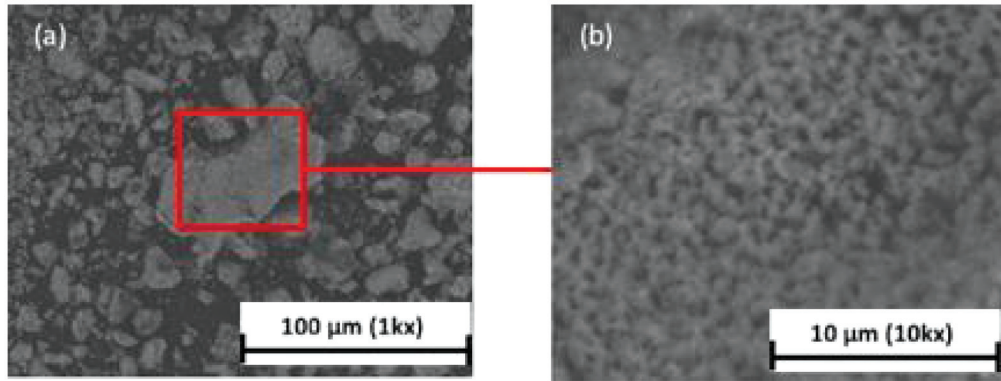


FIGURE 3. The SEM images of LCZO particles calcined at 600°C at the magnifications of (a)1kx and (b)10kx

sample is expected to gravitate to form the independence phases of ZnO and ZnCo_2O_4 with incomplete formation of LCZO at the low calcination temperature. In our future studies, the calcination temperature of LCZO powder is optimized in addition to the improvement of mixing time to obtain a single phase LCZO material.

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