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# Elucidation of Morphological Behavior on the Samarium Strontium Cobaltite Carbonate Composite Cathode for LTSOFC

Siti Fairus MOHAMMAD[1,a*], Sufizar AHMAD[1,b], Hamimah ABDUL RAHMAN[1,c] and Andanastuti MUCHTAR[2,d]

[1]Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, 86400 Batu Pahat, Johor, Malaysia.

[2]Fuel Cell Institute, Universiti Kebangsaan Malaysia, 43600 UKM Bangi, Selangor, Malaysia.

[a]fairusmohd86@gmail.com, [b]sufizar@uthm.edu.my, [c]hamimah@uthm.edu.my

**ABSTRACT.** Samarium strontium cobaltite, $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$ (SSC) and samarium doped ceria, $Sm_{0.2}Ce_{0.8}O_{1.9}$ (SDC) carbonate or (SDCC) was used as the new composite cathode powder materials. Composites cathode powder samples with different on weight percentages, 50:50 wt.% of SSC:SDCC, 60:40 wt.% of SSC:SDCC and 70:30 wt.% of SSC:SDCC were chosen to be use in this research work. The prepared samples of composite cathode powders were characterized by using high energy ball milling (HEBM) techniques for LTSOFC application. Characterizations of the composite cathode were performed through energy dispersion spectroscopy (EDS) and field emission scanning electron microscopy (FESEM) analysis. From EDS analysis shows SSC-SDCC composite cathode powders produced via high energy ball milling techniques (HEBM) or mechanochemical activation indicated that all major elements such as sodium, cobalt, samarium, cerium and strontium were well distributed among the composite cathode powders. Nonetheless, the FESEM images revealed HEBM technique can be used to prepare composite cathode powders with high homogeneity. These results showed that SSC-SDCC composite cathode powders, content with 50 wt.% of SDCC revealed a smallest of particle size with good homogeneity of particle to be us as cathode materials for LTSOFC applications.

***Keywords:*** *SSC, SOFC, Carbonate composite electrolyte, High Energy Ball Milling;*

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## 1. INTRODUCTION

The demand for secure, sustainable and clean energy sources has stimulated countless interests in the electrochemical energy storage and conversion technologies such as fuel cells, batteries and super capacitors. Solid oxide fuel cells (SOFCs) are considered as one of the most promising energy exchange devices which show advantages of high efficiency, fuel flexible and low emission. Development of SOFCs which devoted on issues associated with reduced operational temperature and presents significant solutions regarding (anode, cathode and electrolyte) material selection, synthesis, and processing. Due to reliability issues at high operating temperature, currently, SOFCs research have been motivated towards the cell development of low temperature (LT) to intermediate temperature (IT) [1,2]. The stability of the LTSOFC performance with excellent of power density is the main key with operating temperature within the range of 400 °C to 600 °C (Morandi, 2013). However, the cathode has been the center of the focus in the electrode development largely. In order to maintain sufficient high electrochemical activity to enable SOFC operating at lower temperatures, it is also critically important to have new cathode materials with low polarization loss [4]. Rare earth cobalt oxide such as samarium strontium cobalt oxide, $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$ (SSC) has attracted much attention as cathode due to high mixed ionic and electronic conductivities at low temperature that can improve the cell durability and stability issues. This promising material been revealed as a potential cathode material reveals a good electro-catalyst for oxygen reduction compared to BSCF and particularly compatible with the ceria carbonate based electrolytes [5,6]. Unfortunately, SSC itself is having high reactivity with some electrolytes when operating at high temperature [7,8]. Previous author had proven this disadvantage can be diminish with incorporation samarium doped ceria (SDC) electrolyte with pure of SSC (H Lv, Wu, Huang, Zhao, & Hu, 2006). The introduction of certain amount from alkaline salts into samarium cerium oxide or SDC in order to develop new SDC-carbonate (SDCC) were improve the electrical performances and stability of the chemical part compared with pristine of SDC. Currently, there has no reported regarding study on the preparation of SSC-SDCC composite cathodes. Furthermore, the electrical properties might be improves with this new composite cathode by expecting the increase in triple phase boundary value (TPB) [9,10]

Beside the selection of cathode materials used, the processing of starting composite powder with, such as milling process strongly influence the composite cathode powder properties and subsequently the cell performance. Preparation of composite cathode powders with high energy ball milling (HEBM) or mechanochemical activation oriented technique potentially produces a fine and well distributed powder (Gao, Zhao, Zhou, Ran, & Shao, 2011). Furthermore, the information regarding preparation of SSC-SDCC by using this technique is still rare. Homogenous and fine powders might be obtained from this new SSC-SDCC composite cathode which prepared using this outstanding HEBM technique [12,13]. In the present study, morphological studies of new composite cathode powder microstructure will be investigated. These characterizations will be done by EDS analysis and FESEM.

## 2. MATERIALS AND METHODS

***2.1 Powder preparation.*** In this study, three groups of samples were prepared. Began with producing composite electrolyte which has been prepared by employing suitable amounts of 20 wt.% from the binary carbonates (67 mole.% lithium carbonate, $Li_2CO_3$: 33 mole% sodium carbonate $Na_2CO_3$) and 80 wt.% from the $Sm_{0.2}Ce_{0.8}O_{1.9}$ powders. The mixed electrolyte powder was milled together by employing wet ball milling technique (Fritsch Pulveristte, Germany) with low speed of rpm in ethanol for 1 day. After a complete mixed, the powders were further dried in an oven. This process was done in order to eliminate excess ethanol with temperature at 100 °C for 1 day. After the composite electrolyte powder produced, it was grounded using agate mortar and followed by calcinations process in a furnace with temperature at 680 °C for 1 hour. In order to prepare composite cathode powders, the commercial $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$ raw powder (Kceracell, Korea) containing different weight percentages of $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$ and $Sm_{0.2}Ce_{0.8}O_{1.9}$ carbonate as presented in Table 1 were then mixed together followed with milling process. HEBM from Fritsch Pulveristte 6, was applied in order to obtain SSC based composite cathode powders using of bowl from zirconium oxide, in an ethanol as the milling medium at high speed of rpm for 2 hours. Finally the similar procedure was employed until all the composite cathode powder was grounded, before it will be used for further characterization as mention in the next section.

**Table 1** List of composite cathode powders

| Sample recognition | SSC (wt.%) | SDCC (wt.%) |
|---|---|---|
| SSC-CE55 | 50 | 50 |
| SSC-CE64 | 60 | 40 |
| SSC-CE73 | 70 | 30 |

***2.2 Characterization.*** For investaging the nature of all the samples, EDS analysis of SSC-SDCC powders were analyzed using scanning electron microscopy-energy dispersion spectroscopy mapping (SEM-EDS mapping) (JEOL-JSM 6380LA, Japan). While FESEM image has been conducted via field emission scanning electron microscopy, (JEOL, JSM-7600F, Japan) The average composite particle size was attained by measuring of 50 particles via Image J 1.48 software, while

## 3. RESULTS AND DISCUSSION

Morphology at Fig. 1 shows the elements distributions and EDS spectrum of SSC-CE55, SSC-CE64 and SSC-CE73 composite cathode powders after high energy ball milling process. All of SSC based composite cathode powders indicated that all major elements such as sodium (Na), samarium (Sm), strontium (Sr), cobalt (Co) and cerium (Ce) were homogenously and well distributed among the composite cathode powders [14]. However, for lithium (Li) was not detected from EDS due to its low atomic mass. The implementation of HEBM with high speed method able to disperse the elements homogeneously [13]. From EDS spectrum shows the intensity of the elements which has been contained in the SSC-SDCC composite cathode powder. Quantitative results revealed the amount of each element is given in percentages atom. SSC-CE55 display highest atomic percentage for Na elements followed by SSC-CE64 and SSC-CE73. However, for SSC cathode powders, SSC-CE73 gives a highest atomic percentage compared with SSC-CE64 and SSC-CE55. This phenomenal illustrations that the amount for carbonate content and the cathode content is in line with the carbonate compositions. The homogeneity of particle distributions of each element was very crucial in order to provide each of the elements optimize functioning during oxidation reduction process as well as increased the effectiveness of electrochemical reactions that takes part in cathode [15].

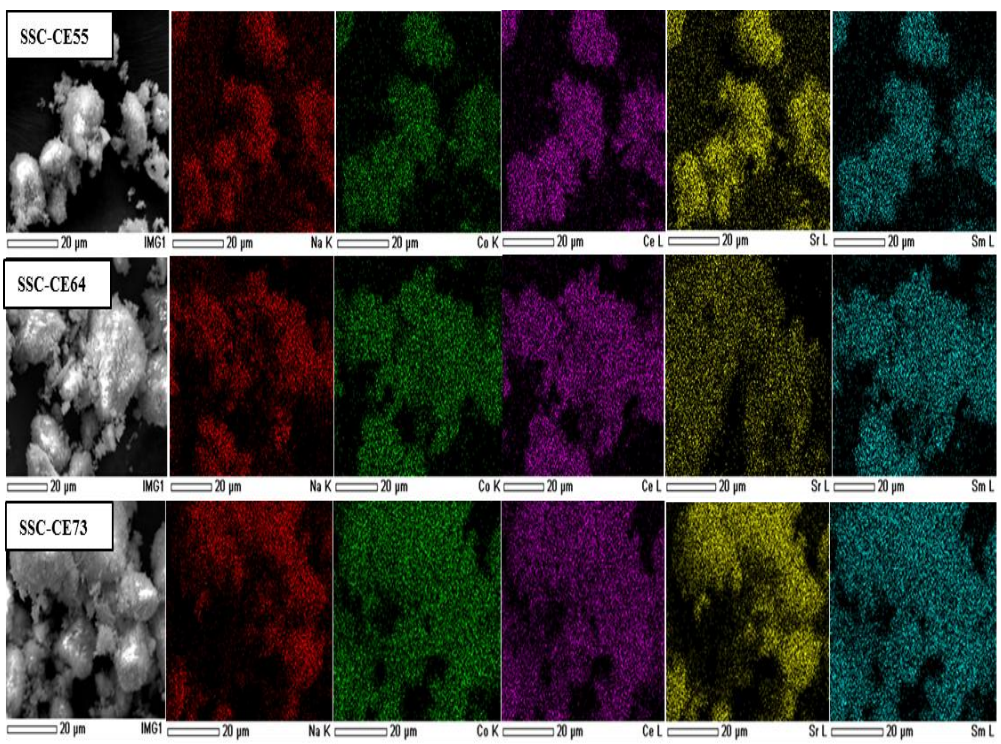


**Fig. 1** EDS spectrum of SSC-CE55 composite cathode powder

The FESEM morphologies of the SSC-SDCC composite cathode powders for all samples were in nano-submicron size, as shown in Fig. 2. The powder morphology of the raw commercial SSC, calcined SDCC, and uncalcined SSC-CE55, SSC-CE64 and SSC-CE73 composite cathode powders, are in nano-scale. All the samples of SSC based composite cathode powders prepared via HEBM at different SSC loadings (50 wt.% to 70 wt.%)

 

display a good powder distribution and fine equiaxed agglomerates (Fig. 2) [15]. The particles of the composite cathode powders obtained were suggestively tiny compared to the starting raw powder of pure SSC. This changes in the particles size as compared to the initial value, can be a measure of the HEBM effectiveness. Analysis of data obtainable in Table 2, allows us to conclude that reduction in particles size as evidenced by the reduction of average composite cathode powders particle size. The mechanical energy created during the ball milling process occurred possible to break particles into tiny pieces. This observation will promote an enlargement on surface area of SSC based composite cathode powders within a shorter processing time. The increased surface area indicates good porosity behaviour and remarkable the expansion of TPB area which subsequently magnify the cell performance of SSC-SDCC composite cathode [16,17,18].

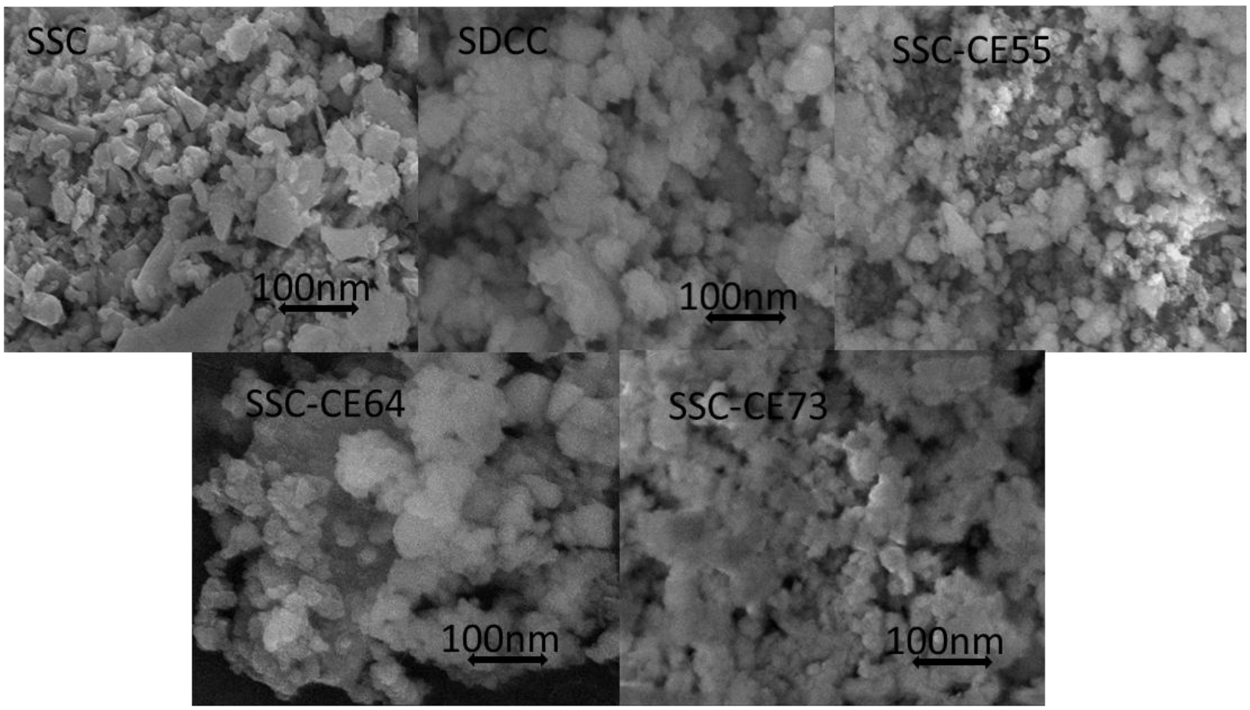


**Fig. 2** FESEM micrograph of SSC, calcined SDCC and SSC-SDCC composite cathode powders

**Table 2** Mean value of particle size for raw and composite cathode powders

| Sample recognition | Mean value of composite particle size ($D_{FESEM}$, nm) |
|---|---|
| Pure SSC | 89.01 |
| Calcined SDCC | 226.66 |
| SSC-CE55 | 72.10 |
| SSC-CE64 | 77.30 |
| SSC-CE73 | 78.00 |

## 4. SUMMARY

Preparation of SSC based composite cathode powder with incorporation of carbonate were optimized using HEBM technique. EDS analysis display SSC based composite cathode powders produced via ball milling demonstrations all major elements such as sodium, cobalt, samarium, cerium and strontium were distributed in good manner. FESEM images were confirmed that HEBM can be used to prepare composite cathode powders with high homogeneity. HEBM technique dramatically influenced the particle size and morphology of the composite cathode powders and has been revealed that SSC with 50% of $Sm_{0.2}Ce_{0.8}O_{1.9}$ carbonate content has high potential to be us as cathode powder materials for LTSOFC applications. However, further detailed analysis will be accomplished in future to determine the effects of carbonate content based on the calcination process, sintering, porosity, thermal expansion coeeficients, electrochemical characterization and single cell performance testing.

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