

**UNIVERSITI TEKNOLOGI MARA**

**A STUDY ON CHELATING AGENTS  
ROLE IN THE SYNTHESIS OF  
CERATE – ZIRCONATE POWDER**

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Thesis submitted in fulfillment  
of the requirements for the degree of  
**Master of Science**

**Faculty of Applied Sciences**

September 2014

## AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

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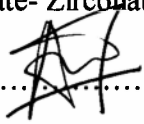
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Cerate- Zirconate Powder

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## ABSTRACT

Ceramics powder based on cerate-zirconates has a great potential application to be used as a solid electrolyte in electrochemical devices such as solid-oxide fuel cell, hydrogen sensor and steam electrolyzer. A conventional method used to prepare this type of ceramic powder is solid-state reaction (SSR) method but it needs high temperature processing to obtain high purity powders. In order to overcome this drawback, wet chemical methods (WCMs) such as sol-gel technique are widely applied in research laboratory because the method is able to produce high purity powders at lower temperature compared to SSR method. In this study, powder of  $\text{BaCe}_{0.54}\text{Zr}_{0.36}\text{Y}_{0.1}\text{O}_{2.95}$  (BCZY) was synthesized by a sol-gel process assisted with chelating agent. There are six chelating agents used; citric acid, tartaric acid, glycolic acid, triethylenetetramine (TETA), nitriloacetic acid (NTA) and ethylenediaminetetraacetic acid (EDTA) which have different electron donating groups. BCZY powder was also prepared by using a combination chelating agents of citric acid–EDTA for comparison study. The samples were dried at 325 °C and heated at various calcination temperatures. The resulting powder properties were characterized using thermogravimetric analysis (TGA), Fourier transform Infrared (FTIR) spectroscopy, X-Ray diffractometer (XRD), X-Ray Fluorescence Spectroscopy (XRF) and Energy dispersive X-Ray (EDX) spectroscopy. Thermal behaviour of dried samples was studied using TGA, and the results showed that all the samples almost completely decomposed at  $\approx 1000$  °C. It was found that the organic compounds released during thermal decomposition of samples increased with the increase of molecular weight of chelating agents. All the samples needed to be treated at relatively higher temperature to remove the carbonates impurities except for one prepared with TETA as proven by FTIR analysis. The sample prepared with TETA exhibits a high crystalline of BCZY powders as confirmed by XRD measurement. XRF and EDX analysis revealed that all the elements composition present in the sample is almost similar to the calculated stoichiometric ratio. As a conclusion, TETA was found to be the best chelating agent in co-operating with metal nitrate salts to produce a pure phase of BCZY powders. Therefore, a study on the role of chelating agent in this type of ceramic powder contributes significant new knowledge in synthesizing BCZY powder at relatively low temperature than SSR.

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