# UNIVERSITI TEKNOLOGI MARA

# A STUDY ON THE ROLE OF SURFACTANT IN THE SYNTHESIS OF CERATE-ZIRCONATE CERAMIC POWDER

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# ABSTRACT

In this study, the powder of $BaCe_{0.54}Zr_{0.36}Y_{0.1}O_{2.95}$ (BCZY) was synthesized via a modified sol-gel process assisted with a surfactant. Three different classes of surfactants were used which are; non-ionic (polyoxyethylene (10) oleyl ether, Brij97, polyoxyethyleneoctyl phenyl ether, Triton X-100, polyethylene glycol, PEG6000 and polyoxyethylene (20) sorbitan monooleate, Tween80), cationic (dimethyl dioctadecyl ammonium chloride, DiDAC, hexadecyltrimethylammonium bromide, HTAB and benzalkonium chloride, BKC) and anionic (aluminium lauryl sulfate, ALS and sodium dodecyl sulfate, SDS). The prepared samples were characterized using Thermogravimetric Analyzer (TGA), Fourier Transform Infrared (FTIR) Spectroscopy, X-ray Diffractometer (XRD) and Scanning Electron Microscopy/Energy Dispersive X-ray (SEM/EDX). TGA results clearly showed that all samples prepared using surfactant exhibits lower thermal decomposition temperature ($T_{td}$< 800 °C) compared to control sample ($T_{td}$> 900 °C). The attachment of surfactant molecules onto the BCZY particle surface was confirmed by the presence of stretching band at ~2900 – 2800 $cm^{-1}$ in the FTIR spectrum. In addition, the presence or absence of carbonate compound is proven by FTIR results with the appearance of the stretching band at ~1400 $cm^{-1}$ and for metal-oxide (M-O) bond at 700 − 400 $cm^{-1}$ after the heating treatment. XRD results for the sample prepared with Brij97, Triton X-100 and DiDAC successfully formed single perovskite phase of BCZY at T= 1100 ºC. However, samples prepared with other surfactants consist of a mixture of secondary phases such as $BaCO_3$, $BaCeO_3$, $BaZrO_3$, $(Ce,Zr)O_2$, $ZrO_2$ and $CeO_2$ even after calcined at T= 1100 ºC. In addition, the lowest synthesizing temperature obtained for the formation of single BCZY perovskite phase was at T= 950 °C using Brij97 as a surfactant which lower than the control sample (T= 1100 °C). This might due to the presence of steric hindrance and repulsive forces between the chelating agent and Brij97 which could assist the formation of BCZY ceramic at a lower temperature. SEM analysis also revealed that the employment of Brij97, Triton X-100 and DiDAC produced homogeneous and fewer agglomerate particles with particle size ranging from 20 nm to 100 nm. The SEM micrograph showed all samples prepared with cationic surfactants exhibited cubic particles due to its hydrophilic head charges indicate that surfactant also affects the morphology of the final product. As a conclusion, the employment of surfactant in a modified sol-gel method able to reduce the synthesizing temperature of BCZY ceramic powder as well as its particle size. Therefore, a study on the behavior of surfactant on cerate-zirconate ceramic powder may contribute to a significant new knowledge in the synthesis of BCZY powder at a relatively lower temperature than a conventional sol-gel method.

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# INTRODUCTION

## 1.1 BACKGROUND

The continuation of the uses of fossil fuel energy resources raises few challenges such as depletion of fossil fuel reserves, global warming and other ecological concerns. In order to solve this issue, there has been a significant effort to use clean and renewable energy sources. One of the most promising alternative energy generating system used nowadays to substitute the conventional fossil fuel-based system is the fuel cell. A fuel cell is an electrochemical device that able to convert chemical energy directly into electrical energy. There are several types of fuel cell that have been developed by researchers such as solid oxide fuel cell (SOFC), polymer electrolyte membrane fuel cell (PEMFC), phosphoric acid fuel cell (PAFC) and etc. Among these different sorts of fuel cells, SOFCs have many advantages such as high powder density and high energy efficiency (Choi et al., 2013). Unfortunately, SOFCs generally operate at high-temperature environment (> 800 °C) and leads to several technical problems associated with mechanical and chemical stability of SOFCs. Thus, the development of alternative electrolyte has been extensively studied by researchers in order to reduce the working temperature of SOFCs.

Generally, there are two classes of SOFC which are oxygen ion ($O^{2-}$) conducting electrolyte and proton ($H^+$) conducting electrolyte. The main difference between these two classes of SOFC is the side in the fuel cell which water is produced (either at anode or cathode) as indicated in Figure 1.1. The conventional oxygen conducting based electrolyte such as yttria-stabilized zirconia (YSZ) and gadolinia doped ceria (GDC) are mostly used since they show a reasonable ionic conductivity at a temperature above 800 °C. However, proton conducting based electrolyte such as strontium cerium oxide ($SrCeO_3$), barium zirconium oxide ($BaZrO_3$) and barium cerium oxide ($BaCeO_3$) have been introduced as a new candidate material to replace the conventional oxygen conducting based electrolyte due to its high ionic conductivity at a lower temperature then oxygen ion conducting fuel cell.