

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS AND
CHARACTERIZATION OF Y³⁺
DOPED Ba(Ce,Zr)O₃ CERAMIC
MATERIAL AIDED WITH
FUNCTIONALIZED ACTIVATED
CARBON**

NURUL AFIFAH BINTI MAHMUD

Thesis submitted in fulfilment
of the requirements for the degree of
Master of Science
(Applied Chemistry)

Faculty of Applied Science

July 2022

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.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student : Nurul Afifah binti Mahmud

Student I.D. No. : 2016588695

Programme : Master of Science (Applied Chemistry) – AS757

Faculty : Applied Sciences

Thesis Title : Synthesis and Characterization of Y^{3+} Doped
Ba(Ce,Zr)O₃ Ceramic Material Aided with
Functionalized Activated Carbon

Signature of Student :

Date : July 2022

ABSTRACT

Yttrium doped barium cerate-zirconate (BCZY) has been extensively utilized as an electrolyte material for proton ceramic fuel cells (PCFCs). Recently, much research has reported on the utilization of the BCZY material as a composite electrode component for PCFCs application. Therefore, more research has been conducted by tailoring the nanostructure of the BCZY materials to improve their quality and performance. In this work, two steps were employed to modify the structure of BCZY powders by utilizing activated carbon (AC) from empty fruit bunches as a dispersing agent during the synthesizing processes. A dispersing agent assisted in separating particles and improved the microstructure of ceramic powder. The first step was the functionalization of AC by using an acid treatment method to remove impurities and activate the functional group on the surface of AC. The acid reagents used for the treatment were hydrogen peroxide (H_2O_2), nitric acid (HNO_3), sulphuric acid (H_2SO_4) and a mixture of $\text{HNO}_3/\text{H}_2\text{SO}_4$. The second step was the addition of AC as a dispersing agent to the BCZY powders synthesized by a modified sol-gel method. The pristine BCZY, modified BCZY with untreated AC (*u*-AC BCZY) and modified BCZY with functionalized AC (*f*-AC BCZY) powders were physically and chemically characterized. The functionalized AC with $\text{HNO}_3/\text{H}_2\text{SO}_4$ (*f*-AC) produces a more stable suspension than other samples indicating the presence of oxygen-containing groups attached to the surface of AC based on the dispersion test. This finding was confirmed by Fourier transform infrared (FTIR) spectroscopy analysis, and the *f*-AC has been chosen as a dispersing agent. For the characterization of BCZY powders, the FTIR and X-ray diffraction (XRD) results confirmed the decomposition of the intermediate compounds in all powders, promoting the formation of a single BCZY perovskite phase. For pristine BCZY, *u*-AC BCZY, and *f*-AC BCZY, their surface areas are $5.61 \text{ m}^2/\text{g}$, $7.6 \text{ m}^2/\text{g}$ and $7.77 \text{ m}^2/\text{g}$ and particle sizes are $\sim 80 \text{ nm}$, $\sim 50 \text{ nm}$, and $\sim 60 \text{ nm}$, respectively. The addition of AC as a dispersing agent has reduced the particle size of BCZY powder. Meanwhile, energy-dispersive X-ray (EDX) data showed the presence of silica impurity in *u*-AC BCZY. However, the presence of an impurity in *u*-AC BCZY interrupted the ion conduction. In addition, the conductivity of dense pristine BCZY ($>91\%$) and *u*-AC BCZY ($>92\%$) pellets are $3.89 \times 10^{-3} \text{ Scm}^{-1}$ and $4.83 \times 10^{-4} \text{ Scm}^{-1}$, while the porous *f*-AC BCZY pellet ($<74\%$) is $9.86 \times 10^{-4} \text{ Scm}^{-1}$, respectively. Low conductivity of the *f*-AC BCZY pellet may result from pores that interrupting the charge carrier's conduction. Thus, *f*-AC BCZY is more suitable as a composite electrode component than as an electrolyte material.

ACKNOWLEDGEMENT

In the name of Allah S.W.T. The Most Merciful and Most Gracious.

Assalamualaikum W.B.T.

Alhamdulillah. Thanks to Allah because of His Bless, I can finally complete this thesis entitled 'Synthesis and Characterization of Y^{3+} Doped $Ba(Ce,Zr)O_3$ Ceramic Material Aided with Functionalized Activated Carbon.

Special thanks and gratitude to my supervisor, Assoc. Prof. Dr. Abdul Mutalib bin Md Jani for his excellent guidance throughout this journey. Thanks for all the information, ideas, criticisms, encouragement, and excellent mentorship given during all stages of this project. Also, thanks to my co-supervisor, Prof. Dr. Nafisah Osman. The supervision and support that was given were truly helped to ensure the smooth progression of this research. The cooperation is appreciated.

I would like to say thank you to my laboratory members in UiTM Cawangan Perlis for their willingness to help throughout this research. Thanks to the staff and laboratory assistants of UiTM Cawangan Perlis, who give excellent cooperation in completing this thesis. Also, thanks to the Physics and Chemistry for Materials Research Groups (PCMaG) members for the great discussion and valuable information. Their name cannot be disclosed, but I would like to acknowledge their kindness in helping me throughout this research.

My deepest thanks and appreciation to my family members for their cooperation, never-ending encouragement, constructive suggestions, and full support to complete this research from the beginning until the end. Special thanks to my mother, Siti Robiah binti Che Nik, and my husband, Mohamad Razif bin Mat Zain, for being supportive and tolerant. To my cheeky princess, Nuha Safiyyah, your presence brought me to stay strong and motivated through this journey. Thanks to all my friends and those who had supported me directly or indirectly while completing this research. Thank you.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF TABLES	ix
LIST OF FIGURES	x
LIST OF PLATES	xii
LIST OF SYMBOLS	xiii
LIST OF ABBREVIATIONS	xiv
LIST OF NOMENCLATURE	xvi
CHAPTER ONE INTRODUCTION	1
1.1 Research Background	1
1.2 Problem Statement	3
1.3 Objectives	4
1.4 Significance of Study	4
1.5 Scope and Limitation of the Study	5
1.6 Organization of the Thesis	5
CHAPTER TWO LITERATURE REVIEW	6
2.1 Proton Ceramic Fuel Cells (PCFCs)	6
2.2 Material for Proton Ceramic Fuel Cells (PCFCs)	7
2.3 Properties of Doped-Ba(Ce,Zr)O ₃ Ceramic Material	8
2.3.1 Electrolyte Material	9
2.3.2 Composite Cathode Material	11
2.3.3 Composite Anode Material	13
2.4 Synthesis of Cerate-zirconate Material	14
2.4.1 Solid-State Reaction (SSR) Method	14