

**THE EFFECT OF TiO_2 NANOPARTICLES ON PROPERTIES OF
CARBOXYMETHYL CELULOSE-LITHIUM PERCHLORATE SOLID
BIOPOLYMER ELECTROLYTE**

NABILAH HANI BINTI NORIHAN

**Final Year Project Report Submitted in
Partial Fulfilment of the Requirements for the
Degree of Bachelor of Science (Hons) Chemistry
in the Faculty of Applied Sciences
Univeristi Teknologi Mara**

JANUARY 2019

ABSTRACT

Biopolymer is a promising green alternative to replace petroleum-based synthetic polymer in solid biopolymer electrolyte (SBE). However, the low ionic conductivity may limit the future applications. In this study, combination of carboxymethyl cellulose (CMC) as host matrix and lithium perchlorate (LiClO_4) SBE have been fabricated with addition of TiO_2 nanoparticles and the effect on chemical, electrical, thermal and mechanical properties were investigated. Various concentration of LiClO_4 (5, 10, 15, 20, 25, 30 and 35 wt%) and TiO_2 (1, 2, 3 and 4 wt%) was prepared by solvent casting method. The samples were characterized by Differential scanning calorimetry (DSC), X-ray diffraction (XRD), Fourier Transfer Infrared Spectroscopy (FTIR), Electrical Impedance Spectroscopy (EIS), Universal Testing and Scanning Electron Microscope (SEM). Incorporation of 35 wt% LiClO_4 shows the highest conductivity with a value of $5.13 \times 10^{-6} \text{ Scm}^{-1}$. Upon the addition of 3 wt% TiO_2 the ionic conductivity increased to $1.162 \times 10^{-5} \text{ S cm}^{-1}$. DSC and XRD studies show that the addition nanoparticles reduce the crystallinity of SBE which improve the ion mobility and thus cause the increment of ionic conductivity. Nevertheless, the addition of nanoparticles had different effect on the mechanical properties of SBE; they reduce the percent elongation at break but improve the tensile strength. The FTIR spectra shows the complexation of Li^+ with all electron donor atoms in CMC structure but nanoparticles only interact with $-\text{OH}$ groups that can be considered as Lewis acid-base interactions. The morphology of the film by SEM showed the homogenous dispersion of nanoparticles in the SBE matrix.

TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENTS	iii
TABLE OF CONTENTS	iv
LIST OF TABLES	vi
LIST OF FIGURES	vii
LIST OF ABBREVIATIONS	x
ABSTRACT	xi
ABSTRAK	xii
CHAPTER 1 INTRODUCTION	
1.1 Background of Study	1
1.2 Problem Statement	3
1.3 Significance of Study	4
1.4 Objectives of Study	5
CHAPTER 2 LITERATURE REVIEW	
2.1 Solid Polymer Electrolyte	7
2.2 Solid Biopolymer Electrolyte	8
2.3 Cellulose-based Solid Biopolymer Electrolyte	9
2.4 Composite Polymer Electrolyte	21
CHAPTER 3 METHODOLOGY	
3.1 Materials	28
3.2 Preparation of composite SBE	28
3.3 Characterizations	
3.3.1 Fourier Transfer Infrared Spectroscopy (FTIR)	31
3.3.2 Electrical Impedance Spectroscopy (EIS)	31
3.3.3 X-ray Diffraction (XRD) Technique	32
3.3.4 Different scanning calorimetry (DSC)	32
3.3.5 Universal Testing	32
3.3.6 Scanning Electron Microscope (SEM)	32
CHAPTER 4 RESULTS AN DISCUSSION	
4.1 CMC and CMC-x (LiClO ₄) System	
4.1.1 DSC	34
4.1.2 XRD	37
4.1.3 EIS	38
4.1.4 FTIR	40
4.2 Effect of TiO ₂ on CMC-35 wt% (LiClO ₄) System	

4.2.1	DSC	45
4.2.2	XRD	47
4.2.3	EIS	48
4.2.4	FTIR	51
4.2.5	Tensile	54
4.2.6	SEM	56

CHAPTER 5 CONCLUSION AND RECOMMENDATIONS	58
---	-----------

CITED REFERENCES	59
-------------------------	-----------

APPENDICES	65
-------------------	-----------

CURRICULUM VITAE	76
-------------------------	-----------

LIST OF TABLES

Table	Caption	Page
2.1	Previous studies on CMC as biopolymer electrolyte with details on the preparation and ionic conductivity.	14
2.2	Previous studies on SPE with addition of nanoparticles and ionic conductivity.	23
3.1	SBE samples composition with complete quantities.	29
4.1	Glass transition temperature (T_g), melting temperature (T_m), recrystallized enthalpy (ΔH_m) and percentage of crystallinity (X_c) values of neat CMC and CMC-x (LiClO_4).	36
4.2	The ionic conductivity of neat CMC and CMC-x (LiClO_4) in ambient temperature.	39
4.3	FTIR for CMC-x (LiClO_4).	43
4.4	Glass transition temperature (T_g), melting temperature (T_m), recrystallized enthalpy (ΔH_m) and percentage of crystallinity (X_c) values of CMC-35 wt% LiClO_4 -x (TiO_2).	46
4.5	The ionic conductivity of neat CMC-35 wt% LiClO_4 -x (TiO_2) at room temperature.	50
4.6	FTIR for CMC-35 wt% LiClO_4 - x (TiO_2).	53
4.7	Mechanical properties of neat CMC and CMC-35 wt% LiClO_4 -x (TiO_2).	55