The Effect of Aluminium Oxide and Cerium Oxide as Dispersoids in Chitosan-Lithium Acetate Complexes

By

LEOW HOI MIEN

A DISSERTATION
SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENT
FOR
THE DEGREE OF MASTER OF TECHNOLOGY
(MATERIAL SCIENCE)
UNIVERSITY OF MALAYA
KUALA LUMPUR
2001

Declaration

I hereby declare that the work reported in this dissertation is my own work unless specified and duly acknowledged by quotations.

Date: 31st March 2001

(Leow Hoi Mien)

ACKNOWLEDGEMENT

Firstly, I would like to thank my parents for giving me the courage and confidence to complete this dissertation. Without their support, guidance and advice, this dissertation would not have been written.

I would like to extend my gratitude to my supervisor, Assoc. Prof. Dr. Kariem Arof, lecturer at the Physics Department, Faculty of Science, University of Malaya, who has always being supportive and the motivation, guidance, advice and encouragement throughout this work. Not forgetting Dr. Wan Jeffrey Basirun, my co-supervisor who has been giving his advice in this work.

I also wish to convey my appreciation to En. Hashim, En. Yusof, Ms. Ho, Pn. Zurina, En. Muhd Zu and En. Azmi for their help in the research and technical problem.

I would like to take this opportunity to thank my fellow friends for being there and always be. Not forgetting my supervisor and colleagues who have been so supportive during my absence in work for this writing.

Last but not least, my heartiest thanks to my family for their love and moral support, and for making this thesis a success.

ABSTRACT

In this study, chitosan with 85% deacetylation was used with $LiOAc.2H_2O$ as doping salt and Al_2O_3 or CeO_2 as dispersoid. They were mixed in certain weight percentages and dissolved in 100ml of 0.5% acetic acid solution. These solutions were poured into petri dishes and left at room temperature for film formation. Infrared Spectroscopy has shown that there is interaction between chitosan and LiOAc with and without dispersoid. The peaks are shifted, broadened, split and differs in intensity. The electrical conductivity of all samples was calculated using the bulk resistance value obtained from the complex admittance plot. The highest electrical conductivity is $8.92 \times 10^{-9} Scm^{-1}$ exhibited by the 2wt% Al_2O_3 added film at room temperature, which is 28 times higher than the film without dispersoid. Samples containing various CeO_2 content exhibit conductivity value with the same order of magnitude. The dielectric behavior and modulus formalism shows that the samples of Al_2O_3 and CeO_2 are ionic conductors. Using XRD, it was observed that the LiOAc peak was depressed when small amounts of Al_2O_3 (up to 2wt%) were added. The crystallinity of films increased when more dispersoid is added. The same observation was noted for samples containing CeO_2, but the changes are not as significant compared to Al_2O_3. All samples are thermally stable up to 350°C. Surface morphology changes with different wt% of Al_2O_3.

Page

Declaration

Acknowledgement i

Abstract ii

CONTENTS iii

CHAPTER 1: INTRODUCTION

1.1 Background 1

1.2 Chitosan 6

1.5 Objective of the present works 8

CHAPTER 2: LITERATURE SURVEY

2.1 Solid Polymer Electrolytes 11

2.2 Conductivity Mechanism 15

CHAPTER 3: EXPERIMENTAL METHODS

3.1 Sample preparation 25

3.2 Fourier Transformed Infrared Spectroscopy 27

3.3 Impedance Spectroscopy 28

3.4 X-ray Diffraction 34

3.5 Thermal Analysis 37

3.5.1 Thermogravimetry Analysis 37

3.5.2 Differential Scanning Calorimetry 39

3.6 Scanning Electron Microscopy 42

CHAPTER 4: FTIR CHARACTERIZATION FOR COMPLEX FORMATION

4.1 Introduction 44

4.2 Fourier Transformed Infrared Spectroscopy Analysis 45

4.3 Summary 52

CHAPTER 5: ELECTRICAL PROPERTIES FOR COMPLEX FORMATION

5.1 Introduction 53

5.2 Conductivity Variation with dispersoid concentration/composition 54

5.3 Dielectric Behavior 65

5.4 Summary 74

CHAPTER 6: XRD CHARACTERIZATION FOR COMPLEX FORMATION

6.1 Introduction 75

6.2 X-ray Diffraction Analysis 76

6.2.1 Aluminium oxide 76

6.2.2 Cerium oxide 80

6.3 Summary 83

CHAPTER 7: THERMAL PROPERTIES FOR COMPLEX FORMATION

7.1 Introduction 84

7.2 Thermogravimetric Analysis 84

7.3 Differential Scanning Calorimetry 87

7.4 Summary 92

CHAPTER 8: SURFACE STRUCTURE OF CHITOSAN-LITHIUM ACETATE COMPLEXES WITH Al_2O_3 THROUGH SCANNING ELECTRON MICROSCOPY

8.1 Introduction 93

8.2 Scanning Electron Microscopy 94

CHAPTER 9: SUMMARY AND CONCLUSION 97

REFERENCES 101