PREPARATION AND CHARACTERIZATION OF CHITOSAN-BASED ELECTROCHEMICAL CELLS

by

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DECLARATION

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

Date,

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Abstract

In this study a chitosan acetate (AC) film prepared from chitosan with a high molecular weight i.e. 2 x 10^6 g/mol was found to exhibit the highest ionic conductivity of (2.14 ± 0.14) x 10^{-7} S/cm. The film was prepared by the solution cast technique. 1 g chitosan was dissolved in 100 ml of 1% v/v acetic acid solution. Ethylene carbonate (EC) was found to be a better plasticizer than propylene carbonate (PC). The film AC + 0.4g EC exhibits the highest electrical conductivity of (1.03 ± 0.01) x 10^{-5} S/cm. Finally, the film of (AC + 0.4g EC) to $LiCF_3SO_3$ ratio of 80:20 was found to exhibit the highest conductivity of 3.0 x 10^{-4} S/cm at room temperature. The electrical conductivity of all samples were calculated using the bulk resistance value obtained from the complex impedance plot in the frequency range between 1 kHz to 5 MHz. Conductivity was also studied as function of temperature between 30 °C to 85 °C. The plot of ln σT versus 10^3/T for each sample seems to obey Arrhenius rule. The regression value (r^2) is between 98 % to 99 % indicating a good straight line fitting of the points obtained. The activation energy, B_1 was calculated from the Arrhenius plots and the increase in σ can be explained in terms of the decrease in B_1 and vice versa. The highest conducting polymer-plasticizer-salt complex has the lowest B_1 of 0.28 eV. The modulus formalism shows that the samples are ionic conductors. X-ray diffraction (XRD) confirms the disruption of the crystalline phase of chitosan acetate upon the addition of plasticizers, complexation between the salt and chitosan and their transformation to the amorphous phase for samples that exhibit increasing electrical conductivity. Infrared spectroscopy (IR) shows the occurrence of chitosan-salt complexation, which involves an interaction between the cation of the salt and the lone pair electrons of amide groups in the chitosan. Complexation is confirmed by the shifting of the -NH_2 bands at 1590 cm^{-1} to lower wavenumbers. Complexation is further confirmed by X-ray photoelectron spectroscopy (XPS) technique. The lithium signal can be deconvoluted into two peaks. The peak at ~ 54.0 eV in attributed to Li-N interaction. The nitrogen signal can be deconvoluted into three peaks. The peak at 403.1 eV is attributed to Li-N interaction. Transference number measurements indicate that the anion of the salt, $CF_3SO_3^-$ is the major ion conducting species in the complexed film exhibiting the highest electrical conductivity. The highest conducting film was used as an electrolyte in the fabrication of chitosan-based secondary polymer cells. However, the performance of these cells needs a lot of improvement as electrode-electrolyte contact poses serious problems, which magnifies the internal resistance of the cells.

Contents Pages

Acknowledgements i

Abstract ii

Contents iii

Chapter 1

1. Introduction

1.1 Why Polymer? 1

1.2 Criteria for Polymer Electrolyte 3

1.3 Polymer Electrolytes and Their Conductivity Values 4

1.4 Conductivity Mechanism in Polymers 6

1.5 What is Chitosan? 14

1.6 General Characteristics of Chitosan 17

1.6.1 Film forming ability 17

1.6.2 Optical properties 17

1.6.3 Chelation of metal ions 18

1.6.4 Glass transition temperature 18

1.7 Lithium Cells 19

1.8 Some Characteristics of Lithium Cells 22

1.9 Objectives of The Present Investigation 24

Chapter 2

2. Experimental Methods

2.1 Samples Preparation 27

2.1.1 To determine which molecular weight group of chitosan will give a film with the highest electrical conductivity 27

2.1.2 To determine the better plasticizer between ethylene carbonate (EC) and proylene carbonate (PC) in enhancing electrical conductivity of chitosan acetate film 28

2.1.3 To determine the salt content in a plasticized chitosan acetate film that gives the highest electrical conductivity 28

2.2 X-ray Diffraction (XRD) 29

2.3 Infrared Spectroscopy (IR) 30

2.4 X-ray Photoelectron Spectroscopy 31

2.5 Impedance Spectroscopy 36

2.6 Ionic Transference Number Measurement 44

2.7 Solid State Electrochemical Cell Fabrication 46

2.7.1 Charge/Discharge Characteristics 47

Chapter 3

3. Electrochemical Impedance Analysis

3.1 Complex Impedance Analysis 48

3.1.1 Highest molecular weight chitosan film exhibits highest electrical conductivity 48

3.1.2 Ethylene carbonate (EC) a better plasticizer than propylene carbonate (PC) in enhancing conductivity of chitosan acetate films 53

3.1.3 How plasticizer increases electrical conductivity and why EC the better plasticizer? 55

3.1.4 Effect of plasticizer on conductivity relaxation time 56

3.1.5 How plasticizer reduces conductivity relaxation time? 62

3.1.6 Effect of salt concentration on electrical conductivity of film 63

3.2 Complex Admittance Analysis 69

3.3 Ionic Conductivity Mechanism 74

3.4 Conductivity, Permittivity and Modulus Formalisms 86

Chapter 4

4. Material Characterization

4.1 X-ray Diffraction Analysis 95

4.2 Fourier Transformed Infrared Spectroscopy Analysis 101

4.3 X-ray Photoelectron Spectroscopy Analysis 106

4.4 Transference Number Measurement 111

Chapter 5

5. Electrochemical Cell Analysis

5.1 The Li/AC+EC+$LiCF_3SO_3$/V_2O_5 cell 113

5.2 The $LiCoO_2$/AC+EC+$LiCF_3SO_3$/V_2O_5 cell 119

Chapter 6

6. Summary and Conclusions 121

Reference 123

Papers Published and Presented by the Author in Related Areas 130