

MANAJEMEN SAINS DAN TEKNOLOGI
UNIVERSITAS PENDIDIKINGAN GURU
2007

1100051284 Perpustakaan Sultanah Nur Zahirah (UMT)
Universiti Malaysia Terengganu

c/n 5104

LP 18 FST 5 2007



1100051284

Preparation and fourier transform infrared spectroscopy study on chitosan based oleic acid doped polymer electrolytes / Siti Aishah Abdullah.

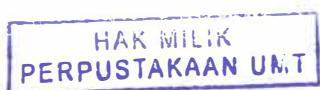


PERPUSTAKAAN
UNIVERSITI MALAYSIA TERENGGANU (UMT)
21030 KUALA TERENGGANU

100051284

1100051284

Lihat sebelah



**PREPARATION AND FOURIER TRANSFORM INFRARED SPECTROSCOPY
STUDY OF CHITOSAN BASED OLEIC ACID DOPED POLYMER
ELECTROLYTES**

By

Siti Aishah binti Abdullah

Research Report submitted in partial fulfillment of
the requirements for the degree of
Bachelor of Applied Science (Physics Sciences)

DEPARTMENT PHYSICAL SCIENCES
FACULTY OF SCIENCE AND TECHNOLOGY
UNIVERSITY MALAYSIA TERENGGANU
2007

1100051284



UNIVERSITI MALAYSIA TERENGGANU

UNIVERSITI MALAYSIA TERENGGANU

21030 KUALA TERENGGANU, TERENGGANU, MALAYSIA

Tel. : 09-668 4100

Faks : 09-669 6441

Laman Web : <http://www.umt.edu.my>

FAKULTI SAINS DAN TEKNOLOGI

JABATAN SAINS FIZIK

PENGAKUAN DAN PENGESAHAN LAPORAN PROJEK PENYELIDIKAN I DAN II

Adalah ini diakui dan disahkan bahawa laporan penyelidikan bertajuk:

PREPARATION AND FOURIER TRANSFORM INFRARED SPECTROSCOPY STUDIES OF CHITOSAN BASED OLEIC ACID DOPED POLYMER GEL ELECTROLYTES oleh SITI AISHAH ABDULLAH, no matrik UK10114 telah diperiksa dan semua pembetulan yang disarankan telah dilakukan. Laporan ini dikemukakan kepada Jabatan Sains Fizik sebagai memenuhi sebahagian daripada keperluan Ijazah Sarjana Muda Sains Gunaan (Fizik Elektronik dan Instrumentasi), Fakulti Sains dan Teknologi, Universiti Malaysia Terengganu.

Disahkan oleh:

Penyelia Utama

Nama : Dr. Mohd Ikmar Nizam Mohamad Isa
Cop Rasmi : DR. MOHD IKMAR NIZAM BIN MOHAMAD ISA

Pensyarah

Jabatan Sains Fizik
Fakulti Sains dan Teknologi
Universiti Malaysia Terengganu
21030 Kuala Terengganu

Tarikh: 27 April 2007

Penyelia Kedua (jika ada)

Nama :

Cop Rasmi :

Tarikh:

Ketua Jabatan Sains Fizik

Nama : Prof. Madya. Dr. Senin Hassan
Cop Rasmi : PROF. MADYA DR. SENIN HASSAN

Ketua Jabatan
Jabatan Sains Fizik
Fakulti Sains dan Teknologi
Universiti Malaysia Terengganu
21030 Kuala Terengganu

Tarikh: 29 April 2007

ACKNOWLEDGEMENT

First of all, I would like to thanks to God Almighty because of His blessing, I can finish my final year project and thesis that have taken hours of my time.

Thousand grateful I would like to give to Dr. Mohd Ikmar Nizam bin Mohamad Isa my final year project supervisor for his precious guidelines and advices. This thesis would not been realized without your never ending encouragement.

Besides that, I would like to thank my beloved father and mother; Abdullah bin. Jaudin and Alimah bt. Hj. Md Sani for their moral, and financial support.

Not forgetting, my final year project members, especially to Azlina bt. Hassan, Nurul A'iin bt. Morsid and Suhana bt. Ramli for keep supporting and giving me so much information in order to complete my thesis preparation.

Last but not least, I would like to convey my special thanks to Mohd Raffe bin Khalid the one who has made me feel special for love and continuous support.

TABLE OF CONTENTS

SUBJECT	PAGE
Acknowledgments	iii
Table of Contents	iv
List of Figures	vi
List of Tables	viii
List of Abbreviations / Symbols	ix
Abstract	xi
Abstrak	xii
CHAPTER 1 INTRODUCTION	1
CHAPTER 2 LITERATURE REVIEW	
2.1 Electrolyte	5
2.2 Polymer	6
2.3 Chitosan	6
2.4 Oleic acid	7
2.5 Fourier Transform Infrared Spectroscopy (FTIR)	9
2.6 Electrochemical Impedance Spectroscopy (EIS)	10
CHAPTER 3 METHODOLOGY	
3.1 Introduction	11
3.2 Material and chemical requirement	11
3.3 Sample preparation	12
3.4 Characterization using Fourier Transform Infrared Spectroscopy (FTIR)	15
3.5 Characterization using Electrochemical Impedance Spectroscopy (EIS)	18

CHAPTER 4 RESULTS & DISCUSSION

4.1	Introduction	20
4.2	FTIR for pure chitosan acetate (CA)	20
4.3	FTIR for pure oleic acid (OA)	22
4.4	FTIR of CA-OA	24
4.5	EIS of CA-OA	30

CHAPTER 5 CONCLUSION AND SUGGESTION

FOR FURTHER STUDY

34

REFERENCES

35

VITAE

38

LIST OF FIGURES

FIGURES	PAGE
1.1 Structure of chitin and chitosan	2
1.2 Structure of oleic acid	2
3.1 Materials and chemical used in this study	11
3.2 Solution was stirred using hotplate and stirrer	13
3.3 Solutions was poured into plastic petri dishes	13
3.4 Film in the petri dishes left to dry at room temperature for the films to form.	14
3.5 Film in the desiccators for continuous drying	14
3.6 Schematic illustration of FTIR system	15
3.7 FTIR Spectrum 100 interfaced to a computer	17
3.8 ATR inside FTIR Spectrum 100	17
3.9 HIOKI 3531-01 LCR Hi-Tester interfaced to a computer	18
3.10 Sample holder with two stainless-steel electrodes	19
4.1 Structure of CA	21
4.2 FTIR of CA	21
4.3 Structure of OA	23
4.4 FTIR of OA	23
4.5 FTIR for (I) pure CA, (II) OA, CA and (III) 10 wt.% OA, (IV) 20 wt.% OA, (V) 30 wt.% OA, (VI) 40 wt.% OA, (VII) 50 wt.% OA	25
4.6 FTIR for (I) OA, CA and (II) 10 wt.% OA, (III) 20 wt.% OA, (IV) 30 wt.% OA, (V) 40 wt.% OA, (VI) 50 wt.% OA	26
4.7 FTIR spectrum obtained (I) pure OA, CA and (II) 10 wt.% OA, (III) 20 wt.% OA, (IV) 30 wt.% OA, (V) 40 wt.% OA, (VI) 50 wt.% OA	27
4.8 FTIR of (I) OA, CA and (II) 10 wt.% OA, (III) 20 wt.% OA, (IV) 30 wt.% OA, (V) 40 wt.% OA, (VI) 50 wt.% OA	28
4.9 FTIR spectra for the shift out of plane in (I) OA, CA and (II) 10 wt.% OA,	

(III) 20 wt.% OA, (IV) 30 wt.% OA, (V) 40 wt.% OA, (VI) 50 wt.% OA	29
4.10 Impedance plot of pure CA	30
4.11 Impedance plot of CA-10 wt.% OA	30
4.12 Impedance plot of CA-20 wt.% OA	31
4.13 Impedance plot of CA-30 wt.% OA	31
4.14 Impedance plot of CA-40 wt.% OA	32
4.15. Impedance plot of CA-50 wt.% OA	32
4.16. The protonation of oleic acid	33

LIST OF TABLES

TABLES	PAGE
Table 3.1: Amount of chitosan and oleic acid used in this studies	12
Table 4.1: Lists of electrical conductivity in room temperature	33

LIST OF ABBREVIATIONS / SYMBOL

ABBREVIATIONS / SYMBOL

OA	Oleic acid
CA	Chitosan acetate
DMF	Dimethyl formamide
DBP	Dybutyl phthalate
EC	Ethylene carbonate
PC	Propylene carbonate
FA	Fatty acids
PUFAs	Polyunsaturated fatty acids
GPE's	Gel polymer electrolytes
T _g 's	Glass transition temperatures
NMR	Nuclear magnetic resonance
IR	Infrared
FTIR	Fourier Transform Infrared
EIS	Electrochemical impedance spectroscopy
ATR	Attenuated total reflection
XPS	X-Ray photoelectron spectroscopy
XRD	X-Ray diffraction
σ	Conductivity
R _b	Bulk resistance
t	Thickness of the film
A	Area of the sample
wt.%	Weight percent
S cm ⁻¹	Siemens per centimeter
Z _r	Real impedance
Z _i	Imaginary impedance
E	Energy gap

<i>v</i>	Velocity
<i>m</i>	Mass
ml	milliliter
g	gram
g / mol	Gram per mole
Hz	Hertz
K	Kelvin
°C	Degree Celsius
pH	Acidity or alkalinity of a solution

ABSTRACT

In this study, chitosan acetate-oleic acid films have been prepared by the solution cast technique where chitosan as a polymer host, and oleic acid as a salt. 1.0 g of chitosan powder was dissolved in 100 ml of 1.0% acetic acid. These film polymer electrolytes with different amounts of salt were investigated as possible ionic conducting polymers. Interaction between chitosan and the salt can be proven by infrared and Electrochemical Impedance Spectroscopy methods. The assymmetric and the symmetric CH₂ stretch of pure oleic acid can be observed at 2919 and 2852 cm⁻¹. When chitosan solution was added to oleic acid, the asymmetric and symmetric CH₂ become more intense. These indicate chitosan-salt interactions. The highest conductivity at room temperature is 1.02 x 10⁻⁹ Siemens per centimeter for the film containing 10 wt.% of oleic acid.

ABSTRAK

Dalam kajian ini, filem chitosan asetik – asid oleik telah disediakan dengan menggunakan teknik ‘solution casting’ dimana chitosan telah digunakan sebagai polimer perumah dan asid oleik sebagai garam. Satu gram serbuk chitosan telah dilarutkan di dalam 100 ml 1% asid asetik. Polimer elektrolit dengan jumlah garam yang ditentukan telah dikaji sebagai suatu polimer pengkonduktor ionik. Interaksi di antara chitosan dan garam dapat dibuktikan dengan kaedah infrared dan spektroskopi impedans elekrokimia. Puncak assimetrik dan simetrik CH₂ bagi asid oleik dapat dilihat pada nombor gelombang 2919 dan 2852 cm⁻¹. Apabila chitosan ditambah ke dalam asid oleik, mod getaran assimetrik dan simetrik CH₂ menjadi semakin menumpu. Ini menunjukkan interaksi diantara chitosan dan garam. Kekonduksian tertinggi pada suhu bilik ialah 1.02×10^{-9} Siemens per sentimeter bagi filem yang mengandungi 10 wt.%.