UNIVERSITI PUTRA MALAYSIA

EFFECT OF PRESSURE AND THICKNESS ON THE ELASTIC AND DIELECTRIC PROPERTIES OF CHITOSAN

NURUL HAZWANI MOHD HASHIM

FS 2012 49

EFFECTS OF PRESSURE AND THICKNESS ON ELASTIC AND DIELECTRIC PROPERTIES OF CHITOSAN

NURUL HAZWANI MOHD HASHIM

MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA

2012

EFFECT OF PRESSURE AND THICKNESS ON THE ELASTIC AND DIELECTRIC PROPERTIES OF CHITOSAN

By

NURUL HAZWANI MOHD HASHIM

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

January 2012

DEDICATION

I dedicate this thesis to my family especially my beloved father and mother for their unconditional love and support.

Abstract of thesis presented to Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

EFFECT OF PRESSURE AND THICKNESS ON THE ELASTIC AND DIELECTRIC PROPERTIES OF CHITOSAN

By

NURUL HAZWANI MOHD HASHIM

January 2012

Chairman: Halimah Mohamed Kamari, PhD

Faculty: Science

This dissertation describes the effect of pressure on the elastic properties of chitosan and also the effect of thickness and pressure on electrical properties of chitosan. The powdered chitosan is being pressed into pellets and the elastic and electrical properties were studied. The experimental investigation was divided into two categories. The minor experimental work, which provides supportive evidence to elastic and electrical properties, consists of work on x-ray diffraction and FTIR. The main experiments consist of work on ultrasonic and electrical measurements. The electrical measurements were measured at low frequencies from 10^{-2} to 10^{4} Hz while ultrasonic properties were determined with MATEC 8000 at 5MHz resonating frequency and at room temperature.

The semicrystalline structure of the chitosan sample was evident by the XRD spectrum. Ultrasonic measurement is used as a non-destructive testing (NDT) technique in analyzing the physical of liquid and solid material. NDT is important because the properties of the sample can be tested without destroying or changing the physical properties of the sample. The elastic moduli, longitudinal modulus (L), shear modulus (G), Young's modulus (E), bulk modulus (K) and Poisson's ratio

increase with increasing of pressure from 2.0 to 6.0 tonne. The large difference between *L* and *G* due to the volume effects shows that the materials formed are easier to bend than to be elongated. Electrical measurement of chitosan shows that sample with 4 mm thickness gives the highest value of $\varepsilon'(\omega)$ ~ 430 compared to 2 mm sample $\varepsilon'(\omega)$ ~ 140. And for chitosan pellets with different pressure, it can be observed that sample with 2.0 tonne pressed pressure gives the highest value of $\varepsilon'(\omega)$ ~ 429 compared to 6.0 tonne sample $\varepsilon'(\omega)$ ~ 229. The dielectric graphs were then normalized into a master curve and fit using the universal law to obtain the properties and mechanism that took part, with the parameters involved such as p, n and α. The entire fitted graph showed that chitosan consisted of quasi-dc and also dipolar mechanism. In complex impedance, the grain boundary effect that appears as a semicircle at low frequency is bigger than the high frequency bulk semicircle. There is no overlapping of peaks in the modulus and impedance plot which suggested that this sample is a long-range and localized relaxation.The activation energies obtained from the master plot, impedance and modulus spectroscopy studies on chitosan pellets with different pressure and thickness reveals the defect relaxation nature.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN TEKANAN DAN KETEBALAN KE ATAS SIFAT ELASTIK DAN DIELEKTRIK KITOSAN

Oleh

NURUL HAZWANI MOHD HASHIM

Januari 2012

Pengerusi: Halimah Mohamed Kamari, PhD

Fakulti: Sains

Disertasi ini menerangkan tentang kesan tekanan pada sifat-sifat kenyal kitosan dan juga kesan ketebalan dan tekanan pada sifat-sifat elektrik kitosan. Serbuk kitosan ditekan menjadi bentuk pelet dan sifat-sifat elektrik dan kekenyalannya dikaji. Penyelidikan eksperimen terbahagi kepada dua kategori. Eksperimen minor yang memberi maklumat sokongan kepada ciri kenyal dan elektrik, terdiri daripada pembelauan sinar-x dan FTIR . Eksperimen utama pula terdiri daripada pengukuran ultrasonik dan elektrik. Ciri elektrik diukur pada frekuensi rendah daripada 10^{-2} to 10^{4} Hz dan ciri ultrasonik ditentukan dengan MATEC 8000 bergetar pada frekuensi 5MHz dan pada suhu bilik.

Struktur separa kristal bagi sampel kitosan dapat dibuktikan menggunakan spektrum XRD. Pengukuran ultrasonik digunakan sebagai satu teknik ujian tidak musnah (NDT) dalam menganalisis sifat fizikal bahan samada cecair atau pepejal. NDT adalah penting kerana sifat-sifat sampel boleh diuji tanpa merosakkan atau mengubah sifat-sifat fizikal sampel itu. Modulus elastik seperti modulus membujur (L), modulus ricih (G), modulus Young (E), modulus pukal (K) dan nisbah Poisson meningkat dengan tekanan dari 2.0 tan ke 6.0 tan. Perbezaan besar antara L dan G

adalah disebabkan oleh kesan isipadu dan ini menunjukkan bahan yang terbentuk lebih mudah untuk dibengkokkan daripada dipanjangkan.

Pengukuran elektrik kitosan menunjukkan sampel yang mempunyai ketebalan sebanyak 4 mm mempunyai nilai ketelusan nyata yang paling tinggi iaitu $\varepsilon'(\omega)$ ~430 berbanding dengan ketebalan sampel 2.0 mm $\varepsilon'(\omega)$ ~140. Bagi pellet kitosan yang berlainan tekanan, dapat dilihat sampel yang ditekan sebanyak 2.0 tan memberikan nilai ketelusan nyata paling tinggi iaitu $\varepsilon'(\omega)$ ~ 429 berbanding dengan sampel 6.0 tan $\varepsilon'(\omega)$~229. Graf-graf dielektrik kemudian dinormalkan ke dalam satu lengkung induk dan disuai padan menggunakan hukum universal untuk mengenal pasti sifat dan mekanisma yang terlibat, dengan parameter seperti p, n dan α. Graf keseluruhan menunjukkan yang kitosan terdiri daripada kuasi-dc dan dipolar.

Dalam kompleks impedans, kesan sempadan butiran yang muncul sebagai separa bulatan adalah lebih besar daripada separa bulatan butiran berfrekuensi tinggi. Tiada pertindihan bagi puncak dalam plot modulus dan impedans. Ini menunjukkan bahawa sampel ini adalah satu pengaktifan jarak jauh dan tenaga relaksasi. Tenaga pengaktifan yang diperolehi daripada plot normal, spektoskopi impedans dan modulus pada pelet kitosan dengan berbeza tebal dan tekanan menunjukkan sifat kecacatan puncak relaksasi.

ACKNOWLEDGEMENTS

First and foremost I am grateful to Allah for giving me strength and patience to complete this research. I would like to express my utmost gratitude to Dr Halimah Mohamed Kamari for her continuous monitoring, supporting and encouragement throughout this entire period of research. Working with her has provided me with a vast understanding on the dielectric science and theoretical experiences from which I will continue to draw benefit in the future. Without her this research will never be completed.

I would like to record my appreciation to Prof. Dr. Kaida Khalid and Assoc. Prof. Dr. Mohammad Zaki Ab. Rahman for their constant supervison, help and guidance during the period of research. I would also like to express my deepest appreciation to Assoc. Prof Dr. Wan Mohd Daud Wan Yusoff for his continuous guidance and encouragement.

Finally, the most appreciation I would like to express is to my family, especially to my father, Mohd Hashim Mydin and my mother, Che Mariyam Pa'chik for their support, encouragement and prayer. I would like to thank my brothers and sister, Syimir Irfan, Harish Iman and Syazni Arina for their understanding and patience. Lastly to my grandfather, grandmother and aunt, Pa'chik Omar, Rahilla Wuzee Ally and Marliana Pa'chik thank you for always being there for me.

I certify that a Thesis Examination Committee has met on 19th January 2012 to conduct the final examination of Nurul Hazwani Mohd Hashim on her thesis entitled **"Effect of Pressure and Thickness on the Elastic and Dielectric Properties of Chitosan"** in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U. (A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Examination Committee were as follows:

Jumiah Hassan, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Wan Mahmood Mat Yunus, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Zaidan Abdul Wahab, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Mohamad Mat Salleh, PhD
Professor
Institute of Microengineering and Nanoelectronics (IMEN)
Universiti Kebangsaan Malaysia
Malaysia
(External Examiner)

SEOW HENG FONG, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisor Committee are as follows:

Halimah Mohamed Kamari, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Kaida Khalid, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

Mohammad Zaki Ab. Rahman
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

BUJANG BIN KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

NURUL HAZWANI MOHD HASHIM
Date: 19 January 2012

TABLE OF CONTENTS