



**EFFECT OF ROSELLE (*Hibiscus Sabdariffa*) ON  
SENSING PROPERTIES OF CHITOSAN FILM  
SENSORS ON FERTILIZER CONCENTRATION  
DETECTION.**

By

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Doctor of Philosophy

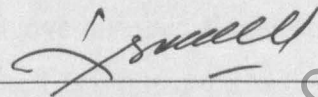
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## APPROVAL AND DECLARATION SHEET

This thesis titled Effect of Roselle (*Hibiscus Sabdariffa*) On Sensing Properties of Chitosan Film Sensors On Fertilizer Concentration Detection prepared and submitted by Roshida Binti Mustaffa (Matrix no: 1140410584) and has been found satisfactory in term of scope, quality and presentation as fulfillment of the requirements for award of the degree of Doctor of Philosophy (Material Engineering) in University Malaysia Perlis (UniMAP).

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## LIST OF ABBREVIATION

AFM	Atomic force microscope
AN	Ammonium Nitrate
CF	Chitosan Film
CFS	Chitosan Film Sensor
CRBFS	Chitosan-Roselle Blend Film Sensor
CS	Chitosan Solution
DSC	Differential scanning calorimeter
DTG	Derivatives Thermogravimetry
EDTA	Ethylenediaminetetraacetic acid
FTIR	Fourier Transform Infrared Spectroscopy
MS	Magnesium Sulphate
RE	Roselle Extract
SPA	Semiconductor Parametric Analyser
SEM	Scanning Electron Microscope
TGA	Thermogravimetric analyser
UF	Urea Formaldehyde
UV-Vis	UV-Vis Spectrometer
XRD	X-ray Powder Diffraction

## LIST OF SYMBOLS

$\alpha$	Acceleration ( $\text{ms}^{-2}$ )
$\sigma$	Conductivity ( $\text{Sm}^{-1}$ )
$^{\circ}$	Degree
$\lambda$	Lambda
$\mu$	Micro
$X_c$	Relative Crystallinity
$\Omega$	Resistance (ohm)
$\rho$	Resistivity (ohm)
$N$	Sample size
$\theta$	Theta

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# KESAN ROSELLE TERHADAP SIFAT CHITOSAN FILEM SENSOR PADA PENGESANAN KEPEKATAN BAJA.

## ABSTRAK

Tujuan projek ini adalah untuk menganalisis ciri-ciri penderiaan sensor kitosan filem (CFS) dan campuran kitosan-roselle sensor filem (CRBFS) dalam pelbagai kepekatan baja. Projek ini telah membentangkan fabrikasi dan pencirian filem kitosan serta campuran kitosan dan roselle. Kepekatan kitosan sebanyak 1.75% w/v telah dipilih sebagai kepekatan yang terbaik untuk filem kitosan. Ia telah terbukti dari ujian elektrik bahawa kedua-dua CFS dan CRBFS dapat beroperasi pada suhu bilik dan memenuhi semua ciri-ciri penderiaan yang diperlukan termasuk memberi tindak balas yang baik, pemulihan, kepekaan, kebolehlungan, kestabilan dan pemilihan apabila terdedah kepada udara basah, udara kering dan kepekatan baja yang berbeza bermula dari 10ppm hingga 50ppm. Semua sensor filem telah menunjukkan tindak balas yang baik dalam kedua-dua medium samaada air dan tanah. Manakala bagi campuran larutan antara kitosan dan roselle, kepekatan kitosan sebanyak 1.50% telah dipilih untuk dicampur dengan ekstrak roselle berdasarkan sifat elektrik dan penderiaan terbaik yang diperolehi semasa ujikaji. CRBFS memberi nilai tindak balas terbaik berbanding CFS dengan nilai 80% kepada 90%. UV-VIS spektrum kitosan menunjukkan 1.75% w/v daripada kepekatan kitosan yang paling tinggi intensiti untuk filem kitosan dan 1.50% w/v daripada kepekatan kitosan untuk filem campuran kitosan. CRBFS menunjukkan prestasi terbaik dengan jurang jalur optik dengan nilai 2.63eV dan pengaliran elektrik dengan nilai  $3.17 \times 10^{-4} \text{ Sm}^{-1}$  manakala CFS menunjukkan prestasi dengan jurang jalur optik dengan nilai 2.87eV dan pengaliran elektrik dengan nilai  $3.02 \times 10^{-5} \text{ Sm}^{-1}$ . Keputusan AFM dari filem kitosan pula menunjukkan dengan peningkatan dalam kepekatan kitosan juga membawa kepada peningkatan saiz bijian. Kitosan-roselle campuran juga menunjukkan peningkatan pada saiz butiran apabila meningkat kuantiti ekstrak roselle yang digunakan. Dengan peningkatan dalam saiz butiran, maka kekasaran pada permukaan meningkat. Imej-imej AFM juga menunjukkan bahawa jika permukaan kasar maka permukaan filem mempunyai kawasan interaksi yang lebih besar dan membuktikan bahawa filem-filem lebih sensitif semasa ujian elektrik dengan tindakan yang cepat dan stabil. Perubahan struktur mikro dan permukaan morfologi adalah berkaitan dengan prestasi elektrik, peningkatan saiz butiran dan kekasaran permukaan membawa kepada pengurangan dalam bilangan sempadan bijian. Imej-imej SEM untuk CFS dan CRBFS juga menunjukkan bahawa kitosan dan campuran kitosan filem dengan kepekatan yang berbeza juga mempunyai struktur morfologi yang berbeza. Imej-imej CFS dan CRBFS menunjukkan permukaan yang baik dan padat. Dari ujian haba (TGA, DTG dan DSC), menunjukkan CFS dengan kepekatan 1.75% w/v mempunyai suhu lebur yang lebih tinggi dibandingkan dengan kepekatan kitosan yang lain. Ini bermakna kitosan filem dengan kepekatan 1.75% w/v memberikan rintangan haba yang lebih tinggi daripada filem kitosan dengan kepekatan yang lain. Ujian haba untuk filem campuran kitosan juga menunjukkan bahawa 1.50% w/v dengan 4% w/v (96:4) ekstrak roselle menunjukkan suhu lebur yang lebih tinggi berbanding dengan campuran yang lain. Hasil ujian dari FTIR menunjukkan bahawa peratusan intensiti tidak wujud di CFS tetapi dengan CRBFS ianya wujud dan ujian elektrik yang dilakukan telah bertambah baik dengan penambahan ekstrak roselle. Pemerhatian XRD juga membuktikan bahawa CRBFS adalah lebih tinggi dan stabil berbanding dengan CFS dalam peratusan penghabluran relatif. Penghabluran ini telah dipertingkatkan daripada interaksi intra dan antara molekul, yang dijelaskan oleh ikatan hidrogen dalam kitosan dan kitosan campuran. Dengan kesemua ujian spektroskopi,