STUDY ON IONIC CONDUCTION OF CARBOXYMETHYL CELLULOSE-KAPPA CARRAGEENAN BLEND FOR SOLID BIOPOLYMER ELECTROLYTES

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citation which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Disertasi ini membentangkan penyiasatan terperinci mengenai struktur dan pengaliran ionik biopolimer semulajadi untuk mengatasi pelupusan sisa polimer yang dihadapi sekarang. Sistem elektrolit biopolimer (BEs) terdiri daripada elektrolit biopolimer karbosimetil sellulose-kappa karagenan (CMC-KC) sebagai perumah dan ammonium nitrat (NH_4NO_3) sebagai penderma proton telah berjaya disediakan oleh teknik tebaran larutan. Pencirian BEs dijalankan menggunakan spektroskopi Fourier Transform Infra merah (FTIR), Difraksi X-ray (XRD), Spektroskopi Impedan Elektrik (EIS), dan Pengukuran Bilangan Transmisi (TNM). Analisis FTIR menunjukkan bahawa interaksi berlaku dalam campuran polimer CMC-KC berikutan peralihan dan kemunculan puncak di nombor gelombang dalam julat 900-2700 cm^{-1}. Di samping itu, terdapat perubahan di puncak dengan pengenalan NH_4NO_3 dalam campuran CMC-KC pada nombor gelombang 828 cm^{-1}, 1047 cm^{-1}, 1322 cm^{-1}, dan 3225 cm^{-1} yang sesuai dengan lenturan simetri (NO_3^-), jalur getaran (NO_3^-), jalur getar (NH_4^+), dan jalur NH_4NO_3 asal. Analisis XRD bagi campuran CMC-KC mendedahkan bahawa peningkatan sistem BEs dalam fasa amorphous dalam sampel B3 dengan nisbah 80:20 KC, dan selepas NH_4NO_3 ditambah dalam campuran CMC-KC BEs, sampel S7 yang terkandung 30 wt.% NH_4NO_3 menunjukkan peningkatan amorphousness yang disokong oleh tahap kehabluran (X_c) yang rendah dan juga dikaitkan dengan hasil konduktiviti ionik. Kekonduksian ionik diperoleh pada nilai optimum 3.19×10^{-7} Scm^{-1} pada suhu bilik apabila perumah polimer diadun dengan nisbah 80:20 KC. Konduktiviti terus meningkat kepada 2.00×10^{-4} Scm^{-1} dengan penambahan 30 wt. % NH_4NO_3. Analisis suhu kekonduksian semua elektrolit dibantu dengan termal dan didapati mengikuti aturan Arrhenius. Kajian dielektrik mengesahkan bahawa elektrolit mematuhi tingkah laku bukan-debye. Kajian pengangkutan membuktikan bahawa kekonduksian ion dikawal oleh pekali resapan, D, dan mobiliti ionik, μ, dikira menggunakan dekonvolusi ATR-IR dengan menggunakan fungsi Gaussian-Lorentz diselaraskan kepada perisian Origin Lab 8.0. Pengukuran nombor pemindahan ionik mengesahkan bahawa kation spesies ion dengan konduksi tertinggi (S7) berada pada 0.85. Secara keseluruhan, dipercayai bahawa sistem BE sekarang ini berpotensi sebagai calon baharu elektrolit dalam fabrikasi peranti elektrokimia.

ABSTRACT

This thesis presents a detailed investigation of structural and ionic conduction properties of environmentally friendly natural biopolymers electrolyte to overcome polymeric waste disposal faced nowadays. In this research, biopolymer electrolytes (BEs) system comprising carboxymethyl cellulose-kappa carrageenan (CMC-KC) blend biopolymer electrolyte as the host and ammonium nitrate (NH_4NO_3) as the proton provider was successfully prepared by solution casting technique. The characterizations of the BEs was carried out using Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), Electrical Impedance Spectroscopy (EIS), and transference number measurement (TNM). The FTIR analysis showed that interaction took place in the CMC-KC polymer blend due to the shifting and appearance of peaks in wavenumbers in the range of 900–2700 cm^{-1}. In addition, there were changes in the peaks with the addition of NH_4NO_3 in the CMC-KC blend at wavenumbers 828 cm^{-1}, 1047 cm^{-1}, 1322 cm^{-1}, and 3225 cm^{-1} which corresponded to the symmetric bending (NO_3^-), vibrational band (NO_3^-), vibrational band (NH_4^+), and N-H band of pure NH_4NO_3 respectively. XRD analysis of the CMC-KC blend revealed that the BEs system has increased in amorphous phase in sample B3 with a ratio of 80:20 of KC, and after NH_4NO_3 was added in the CMC-KC blend of BEs, the sample S7 that contained 30 wt.% of NH_4NO_3 showed increased amorphousity supported by the low degree of crystallinity (X_c) and also correlated to the ionic conductivity result. The ionic conductivity was obtained at an optimum value of 3.19×10^{-7} Scm^{-1} at room temperature when the polymer host was blended with 80:20 ratio of KC. Conductivity was further enhanced to 2.00×10^{-4} Scm^{-1} with the addition of 30 wt.% of NH_4NO_3. The conductivity-temperature analysis of all electrolytes was thermally assisted and was found to follow the Arrhenius rule. Dielectric and Modulus studies confirmed that the BEs obeyed non-debye behaviour. Transport study proved that the ionic conductivity was controlled by the diffusion coefficient, D, and ionic mobility, μ calculated using ATR-IR deconvolution by employing the Gaussian-Lorentz function adjusted to the Origin Lab 8.0 software. The ionic transference number measurement confirmed that the ion species cation with the highest conduction (S7) was at 0.86. Overall, it is believed that this present BEs system has potential as a new electrolyte candidate in the fabrication of electrochemical devices.

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LIST OF SYMBOLS

ml	Mililitre
℃	Degree Celcius
g	Grams
°	Degree
σ	Ionic Conductivity
%	Percentage
π	Pi
θ	Theta
A	Area
cm	Centimeter
cm^{-1}	Per Centimeter
cm^{2}	Square Centimeter
f	Frequency
Hz	Hertz
MHz	Mega Hertz
Zi	Imaginary Parts of Modulus
Zr	Real Parts of Modulus
~	Approximately
a.u	Arbitrary unit
η	Number of ions
D	Diffusion coefficient
μ	Ionic mobility

LIST OF ABBREVIATIONS

BEs	Biopolymer electrolytes
CMC	Carboxymethyl cellulose
CA	Cellulose acetate
EIS	Electrochemical impedance spectrospcopy
EES	Electrical energy storage
FTIR	Fourier transform infrared spectroscopy
FWHM	Full width at half maximum
HEC	Hydroxyethyl cellulose
HPC	Hydroxypropyl cellulose
KC	Kappa carrageenan
LiBs	Li-ion batteries
LST	Linear Systems Theory
NH_4NO_3	Ammonium nitrate
NH_4Br	Ammonium bromide
NH_4Tf	Ammonium trifluoro methane sulfonate
PEs	Polymer electrolytes
PVC	Polyvinyl chloride
PEO	Polyethylene oxide
PVA	Polyvinyl alcohol
PVP	Poly(vinyl) pyrrolidone
RFID	Radio-frequency identification
TNM	Transference number measurement
XRD	X-ray diffractometer

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