STRUCTURAL, THERMAL AND ELECTRICAL CHARACTERIZATION OF CARBOXYMETHYL CELLULOSE – AMMONIUM CHLORIDE – BASED SOLID BIOPOLYMER ELECTROLYTES FOR USE IN RECHARGABLE PROTONIC CELL

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Two solid biopolymer electrolyte (SBE) systems of carboxymethyl cellulose doped ammonium chloride (CMC-AC) and plasticized with propylene carbonate (CMC-AC-PC) have been successfully prepared by solution casting technique. Both SBE systems were translucent, free-standing and flexible with no phase separation. In this research, two different IR methods; Gaussian 09W software as computational and Fourier Transform Infrared (FTIR) as experimental, were used to identify the complexation between polymer, salt and plasticizer. Gaussian 09W analysis reveals the complexation occurred between CMC and AC at 1412cm^{-1} and 1585cm^{-1}. No significant complexation is observed with the addition of PC, hence, PC has created new pathway and acted as lubricant in the SBEs. X-Ray Diffraction (XRD) analysis showed the amorphous pattern for both systems. With the addition of PC, XRD analysis showed that PC had increased the amorphousness of the SBEs. Thermal analysis by Differential Scanning Calorimetry (DSC) showed decrease in the value of glass transition temperature, T_g with addition of salt / plasticizer concentration

helps in increasing the ionic conductivity by the ease movement of the polymer chains. The electrical properties of SBEs were investigated using Electrical Impedance Spectroscopy (EIS). The highest conducting CMC-AC SBE was achieved at (1.43 ± 0.02) x 10^{-3} S/cm with addition of 16 wt.% AC and improved to (1.01 ± 0.03) x 10^{-2} S/cm when plasticized with 8 wt.% of PC. The temperature-conductivity of SBEs were observed to obey the Arrhenius rule (R^2 ~ 1) and it is a thermally activated process. The ionic transport properties were determined via FTIR deconvolution method. It shows that the ionic conductivity results for both SBE systems were predominantly controlled by the number density (η), mobility (μ) and diffusion coefficient (D) of ions. By performing the Transference Number Measurement (TNM), the charge transport in these SBE systems is predominantly ions and the conducting species were identified as cation or protons (H^+) which confirmed the FTIR analysis. The conduction mechanisms for both SBE systems can be represented by the Quantum Mechanical Tunnelling (QMT) model. The rechargeable protonic cell (RPC) was assembled using both systems with the configuration Zn + $ZnSO_4.7H_2O$ // the highest conducting SBE // MnO_2 and showed promising performance at room temperature. This work implies that there is potential and possible practical application of the present SBE as a new invention of bio-based electrolytes system in the fabrication of electrochemical devices.

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PENCIRIAN STRUKTUR, TERMAL DAN ELEKTRIKAL BERASASKAN KARBOSIMETIL SELULOSA BESERTA AMONIUM KLORIDA DALAM ELEKTROLIT BIOPOLYMER PEPEJAL DAN APLIKASI TERHADAP SEL PROTON YANG BOLEH DICAS SEMULA

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Di dalam kajian ini, dua sistem biopolimer elektrolit pepejal (SBE) yang mengandungi karbosil metilselulosa didop dengan amonium klorida (CMC-AC) dan ditambah bahan pemplastik, propilina karbonat (CMC-AC-PC) telah berjaya dihasilkan melalui teknik penuangan larutan. Kedua-dua sistem biopolimer elektrolit pepejal adalah bersifat lutsinar, berbentuk bebas dan mudah lentur dan tiada pengasingan fasa. Dalam kajian ini terdapat dua kaedah pengukuran inframerah iaitu analisis menggunakan perisian *Gaussian* 09W sebagai simulasi pengkomputeran dan analisis menggunakan spektroskopi Inframerah Transformasi Fourier (FTIR) sebagai pengukuran eksperimental. Ia digunakan untuk mengenalpasti kompleksasi antara polimer, bahan pendopan and bahan pemplastik. Analisis *Gaussian* 09W menunjukkan kompleksasi antara CMC dan AC telah berlaku di 1412cm^{-1} dan 1585cm^{-1}. Tiada kompleksasi ketara diperhatikan dengan penambahan PC, justeru, PC telah memberi laluan baru dan bertindak sebagai “pelincir” di dalam sistem SBE. Analisis pembelauan Sinar-X (XRD) menunjukkan kedua-dua sistem adalah amorfus.

Analisis XRD menunjukkan keamorfusan SBE meningkat dengan penambahan PC. Analisis terma melalui Pengimbasan Perbezaan Kalorimetri (DSC) menunjukkan suhu peralihan kaca, T_g menurun dengan penambahan kepekatan bahan pendopan / bahan peliat dalam membantu meningkatkan kekonduksian ionik disebabkan pergerakan mudah rantaian polimer. Ciri-ciri elektrikal SBE disiasat menggunakan Spektroskopi Impedans Elektrik (EIS). Sistem CMC-AC memperoleh kekonduksian tertinggi pada (1.43 ± 0.02) x 10^{-3} S/cm bagi sampel 16 wt. % dan meningkat dengan penambahan bahan pemplastik, PC kepada (1.01 ± 0.03) x 10^{-2} S/cm untuk sampel 8 wt. %. Analisis suhu-kekonduksian SBE diperhatikan mematuhi hukum Arrhenius (R^2 ~ 1) di mana ia terbantu terma. Pencirian pengangkutan ionik ditentukan melalui kaedah pengenyahkonvolutan FTIR. Ia menunjukkan kekonduksian ionik bagi kedua-dua sistem SBE secara dominan dikawal oleh kebolehgerakan (μ) dan pekali peresapan (D) berbanding kepadatan nombor (η) ion. Daripada pegukuran nombor transferens (TNM), pengangkutan cas dalam sistem SBE adalah kebanyakannya ion dan pembawa cas bergerak adalah kation atau dikenali sebagai proton (H^+) yang menyokong analisis FTIR. Mekanisma kekonduksian untuk kedua-dua sistem SBE adalah model penerowongon mekanik kuantum (QMT). SBE dengan kekonduksian tertinggi bagi kedua-dua sistem telah digunakan dalam fabrikasi sel proton yang boleh dicas semula (RPC) menggunakan konfigurasi Zn + $ZnSO_4.7H_2O$ // SBE dengan kekonduksian tertinggi // MnO_2 dan ia telah menunjukkan prestasi yang baik pada suhu bilik. Berdasarkan prestasi dan potensi bateri proton cas semula di dalam kajian ini, ia sesuai diaplikasikan dalam penggunaan peranti elektrokimia.