STUDIES ON IONIC TRANSPORT AND IMMITTANCE RESPONSE OF CARBOXYMETHYL CELLULOSE/POLYVINYL ALCOHOL-BASED SOLID BIOPOLYMER ELECTROLYTES AND ITS APPLICATION

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We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations 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

Elektrolit polimer (PE) telah menarik evolusi hebat kerana aplikasi luas di dalam bidang peranti penyimpan tenaga. Banyak kajian telah diberi tumpuan berdasarkan penggunaan polimer berasaskan sumber petroleum yang memberikan kelemahan termasuk keberkesanan kos, kekurangan sumber petroleum dan masalah alam sekitar. Oleh itu, penyelidikan terkini ini dibangunkan biopolimer yang terdiri daripada elektrolit campuran polimer iaitu karboksimetil selulosa (CMC)–polivinil alkohol (PVA) sebagai perumah yang dihasilkan melalui teknik tebaran larutan. Penggabungan dopan ionik (NH_4NO_3) diikuti oleh proses campuran plastik iaitu etilena karbonat (EC) ke dalam CMC–PVA sebagai elektrolit biopolimer pepejal (SBEs) telah disiasat untuk peningkatan sifat struktur, optikal dan termal melalui Fourier transform inframerah (FTIR) spekstroskopi, pembelauan sinar-X (XRD), mikroskopi imbasan electron (SEM), analisis termogravimetrik (TGA) dan kalorimetri pengimbasan berbeza (DSC). Peningkatan ini penting kerana dapat mempengaruhi kekonduksian ionik dan pengangkutan dalam system SBEs yang boleh diukur menggunakan spektroskopi impedans elektrik (IS). Sampel SBEs yang paling tinggi telah difabrikasikan di dalam kapasitor dua lapis elektrikal (EDLC) yang mana prestasi mereka dinilai melalui voltametri kitaran (CV), pengukuran caj-discaj galvanostatik (GCD) dan analisis impedans elektrokimia (EIS). Kekompleksan pada kumpulan berfungsi yang aktif seperti C–O–C, –OH and $–COO^-$ dipercayai mempengaruhi sifat kristal dimana SBEs menjadi lebih amorfos apabila ditambah dengan NH_4NO_3 dan EC. Analisis morfologi menunjukkan sampel yang dibangunkan tidak mempunyai fasa pengasingan kerana kekompleksan dalam sistem SBEs. Semua sampel SBEs dikatakan mempunyai kestabilan termal sehingga suhu 300 °C dan kekonduksian ionik telah meningkat kepada 1.70×10^{-3} S/cm dengan penambahan 30 wt. % NH_4NO_3 dan terus meningkat ke nilai tertinggi 3.92×10^{-3} S/cm apabila ditambah dengan 6 wt. % EC. Berdasarkan pendekatan dekonvolusi-IR, pengangkutan ionik menunjukkan bahawa kekonduksian ionik telah dikawal oleh bilangan ion (η), kelajuan pergerakkan ion (μ) dan pekali resapan ion (D). Kekondusian tertinggi dari sampel NH_4NO_3 dan EC telah menunjukkan kestabilan pada 1.73 V dan 1.89 V, melalui kestabilan elektrokimia (potensi tingkap). Sampel SBEs dengan campuran plastik menunjukkan kestabilan kitaran yang lebih baik berbanding sampel SBE tanpa campuran plastik pada ketumpatan arus yang lebih tinggi, 0.339 mA/cm^2. Akibatnya, sistem dengan campuran plastik menghasilkan had kapasiti, ketumpatan tenaga dan kuasa yang lebih tinggi. Oleh itu, kajian terkini ini menunjukkan kebolehan CMC–PVA sebagai peranti elektrolit dengan memberikan ciri-ciri elektrokimia yang menguntungkan apabila difabrikasikan di dalam EDLC.

ABSTRACT

Polymer electrolytes (PEs) have been attracting attention owing to their wide application in areas of energy storage devices. Extensive research has been focusing on the application of petroleum-based polymers which give drawbacks including high costs, petroleum resources depletion and environmental problems. Thus, this present research has been carried out on biopolymers comprising of carboxymethyl cellulose (CMC)–polyvinyl alcohol (PVA) polymer blend as host which is prepared via the solution casting technique. The incorporation of ionic dopant (NH_4NO_3) followed by plasticizer, namely ethylene carbonate (EC) into the CMC–PVA also known as solid biopolymer electrolytes (SBEs) was investigated for the enhancement of the structural, optical and thermal properties via Fourier transform infrared (FTIR) spectroscopy, x-ray diffraction (XRD) spectroscopy, scanning electron microscopy (SEM), thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). This enhancement is important because it could influence the ionic and transport conduction properties of the SBEs that is measured by electrical impedance spectroscopy (IS). The highest conducting SBEs samples were fabricated in an electrical double layer capacitor (EDLC) where its performance was assessed via cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and electrochemical impedance spectroscopy (EIS). The complexation at the active functional group of C–O–C, –OH and $–COO^-$ is believed to influence the crystalline nature where the SBEs became more amorphous upon the addition of the NH_4NO_3 and EC. Morphology analysis showed that the developed samples have no phase segregation that is also due to the occurrence of complexation in the SBEs system. All SBEs samples were found to be thermally stable up to 300 °C and the ionic conductivity had increased to 1.70×10^{-3} S/cm with the addition of 30 wt. % NH_4NO_3 and further increased to 3.92×10^{-3} S/cm when added with 6 wt. % EC. Based on IR-deconvolution approaches, ionic transport elucidated that number of ions (η), ions mobility (μ) and diffusion coefficient (D) governed the ionic conductivity. The highest conducting samples both from NH_4NO_3 and EC were found to be stable up to 1.73 V and 1.89 V, respectively based on their electrochemical stability (potential windows). The plasticized SBEs demonstrated better cycling stabilities than un-plasticized SBEs at higher current density, 0.339 mA/cm^2. As a result, the plasticized system exhibited higher specific capacitance, energy and power density. Therefore, the present research revealed the possibility of CMC–PVA as an electrolyte system by demonstrating favorable electrochemical properties in an EDLC practical application.

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η	Number of ions
μ	Mobility of ions
D	Diffusion coefficient
C_{sp}	Specific capacitance
I_R	Internal resistance
T_g	Glass transition temperature
H^+	Proton
ε_r	Dielectric constant
ε_i	Dielectric loss
wt. %	Weight percentage
λ	Wavelength
2θ	Bragg angle
χ_c	Degree of crystalline in percentage
A_c	Area of crystalline region
A_T	Total area under the peak representing the area of crystalline region and amorphous area region
Z^*	Complex impedance
Z_r	Real impedance
Z_i	Imaginary impedance
σ	Ionic conductivity
t	Thickness
R_b	Bulk resistance
A	Area of contact surface
C_0	Vacuum capacitance
ε_0	Permittivity of free space
ω	Angular frequency
f	Frequency
M_r	Real modulus
M_i	Imaginary modulus
t_{H^+}	Proton transference number
R	Resistance

I_{ss}	Steady state current
I_o	Initial current
η %	Coulombic efficiency
E_d	Energy density
P_d	Power density
t_d	Discharging time
t_c	Charging time
V_d	Voltage drop
i	Current
R_d	Diffusion resistance
R_s	Ohmic resistance
R_{ct}	Charge transfer resistance
T_d	Maximum decomposition temperature
ΔH	Enthalpy change
p	Deviation of the vertical axis
k^{-1}	Capacitance value of CPE
E_a	Activation energy
R^2	Regression value
σ_o	Pre-exponential factor
k_b	Boltzmann constant
M	Number of moles
N_A	Avogadro's number
E	Electric charge
σ_ω	Total conductivity
A	Temperature dependent parameter
s	Power law exponent
σ_{dc}	Frequency independent dc conductivity
σ_{ac}	Frequency independent ac conductivity
W_m	Maximum barrier height
D	Dipole moment

LIST OF ABBREVIATIONS

CMC	Carboxymethyl cellulose
PVA	Polyvinyl alcohol
PEs	Polymer electrolytes
SBEs	Solid biopolymer electrolytes
EDLC	Electric double layer capacitor
FTIR	Fourier transform infrared spectroscopy
XRD	X-ray Diffraction
SEM	Scanning electron microscopy
TGA	Thermal gravimetric analysis
DSC	Differential scanning calorimetry
EIS	Electrical impedance spectroscopy
TNM	Transference number measurement
LSV	Linear sweep voltammetry
CV	Cyclic voltammetry
GCD	Galvanostatic charge-discharge
EIS	Electrochemical impedance spectroscopy
EC	Ethylene carbonate
NH_4NO_3	Ammonium nitrate
PEO	Polyethylene oxide
SPE	Solid polymer electrolyte
GPE	Gel polymer electrolyte
CPE	Composite polymer electrolyte
Al_2O_3	Aluminium oxide
SiO_2	Silicon dioxide
TiO_2	Titanium dioxide
LiPSS	Lithium polystyrene sulfonate
LiI	Lithium iodide
$LiAlO_2$	Lithium aluminate
PEG	Polyethylene glycol
LAGP	Lithium aluminium germanium phosphate
$LiClO_4$	Lithium perchlorate

LiTFSI	Lithium bis (trifluoromethylsulphonyl) imide
LLZO	Lithium Lanthanum zirconate
PMMA	Polymethyl methacrylate
LiTf	Lithium trifluoromethanesulfonate
GG	Guar gum
PVdF	Polyvinylidene fluoride
$BMImBF_4$	1-butyl-3-methylimidazolium tetrafluoroborate
HNT	Halloysite nanotube
SN	Succinonitrile
HFP	Hexafluoro propylene
LC	Liquid crystal
PVC	Polyvinyl chloride
PAN	Polyacrolnitrile
CH_3COOLi	Lithium acetate
$LiSO_4$	Lithoum suphate
AMPS	2-acrylamido-2-methylpropanesulfonic acid
KC	κ-carrageenan
Gly	Glycerol
NH_4Br	Ammonium bromide
CS	Chitosan
PVP	Polyvinyl pyrrolidone
$AgNO_3$	Silver nitrate
$(Mg(CF_3SO_3)_2$	Magnesium trifluoromethanesulfonate
KIO_3	Potassium iodate
$NaHCO_3$	Sodium hydrogen carbonate
$MnSO_4$	Manganese sulphate
LiTrif	Lithium triflate
NH_4I	Ammonium iodide
NH_4SCN	Ammonium thiocyanate
NH_4CO_3	Ammonium carbonate
NH_4Tf	Ammonium triflate
NH_4HCO_2	Ammonium formate
PC	Propylene carbonate
DMC	Dimethyl carbonate
DEC	Diethyl carbonate
MC	Methyl cellulose

PEGB	Poly (ethylene glycol) borate ester
$LiBF_4$	Lithium tetrafluoroborate
LC	Lignocellulose
PEI	Polyetherimide
LiOAc	Lithium acetate
NaI	Sodium iodide
KI	Potassium iodide
DMSO	Dimethyl sulfoxide
$Mg(ClO_4)_2$	Magnesium perchlorate
$LiPF_6$	Lithium hexafluorophosphate
$ZnCl_2$	Zinc chloride
IR	Infrared
ZnSe	Zinc selenide
TA	Thermal analyzer
NMP	N-methyl-2-pyrrolidone
ESR	Equivalent series resistance
QMT	Quantum-Mechanical Tunnelling
SPH	Small Polaron Hopping
OLPT	Overlapping-Large Polaron Tunnelling
CBH	Correlated Barrier Hopping
MSA	Methanesulfonic acid

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