

UNIVERSITI TEKNOLOGI MARA

**NANOCOMPOSITE POLYMER GEL
ELECTROLYTE BASED ON
LiBOB-CELLULOSE ACETATE FOR
LITHIUM-OXYGEN BATTERY**

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of the requirements for the degree of
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CONFIRMATION BY PANEL OF EXAMINERS

I certify that a panel of examiners has met on 9th June 2016 to conduct the final examination of Siti Zafirah Binti Zainal Abidin on his Doctor of Philosophy thesis entitled “Nanocomposite Polymer Gel Electrolyte Based on LiBOB–Cellulose Acetate for Lithium–Oxygen Battery” in accordance with Universiti Teknologi MARA Act 1976 (Akta 173). The Panel of Examiners recommends that the student be awarded the relevant degree. The panel of Examiners was as follows:

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

This thesis focuses on the preparation and characterization of liquid electrolytes (LEs), polymer gel electrolytes (PGEs) and nanocomposite polymer gel electrolytes (NCPGEs). In this work, lithium bis(oxalato) borate (LiBOB) salt was incorporated as the source of charge carriers. LEs were first prepared by dissolving LiBOB in the solvent, γ -butyrolactone using various molar concentrations of LiBOB. The optimum conductivity (0.8 M LiBOB) of the liquid electrolyte was then gelled with different concentrations of cellulose acetate (CA) to form PGEs. The ionic conductivity of all electrolyte samples were calculated using the bulk resistance value obtained from the complex impedance plot in the frequency range between 100 Hz to 1 MHz. The ionic conductivity pattern of PGEs slightly increases at low cellulose acetate concentration and starts declining with increasing cellulose acetate content. The conductivity reaches a maximum value of 7.05 mS cm^{-1} for the electrolyte containing 2.4 wt.% CA at room temperature. This phenomenon is best clarified by the Breathing Polymeric Chain model. Although PGE has a high conductivity, its physical properties and dimensional stability need to be improved for practical application. Therefore, the highest conductivity of PGE was dispersed with different weight ratios of nanosized silica to obtain NCPGEs. The highest conductivity of 6.49 mS cm^{-1} is obtained with the addition of 2.5 wt.% SiO_2 . Ionic conductivity for all systems was also studied as a function of temperature from 303 K up to 363 K. The plot of $\log \sigma$ versus $1000/T$ for each sample obeys Arrhenius rule implying the ionic conductivity to be thermally assisted. The conduction mechanism in liquid electrolyte, polymer gel electrolyte and nanocomposite polymer gel electrolyte can be best explained using the small polaron hopping (SPH) model since the power law exponent s increases with the increase in temperature. Transference number characterization supports the ionic conductivity results. The voltammogram of highest conducting NCPGE with an anodic decomposition limit of the electrolyte was stable up to 5.5 V vs. Li. The addition of nanosized silica into PGE enhances the physical properties which is supported by the results of viscosity and Environmental Scanning Electron Microscope (ESEM). Fourier Transform Infrared (FTIR) spectroscopy was performed to study the molecular interaction between components in each electrolyte system. The complexations between the materials used in electrolyte systems are divulged based on shifting of the bands, changes in intensity of the bands, changes in shape and existence of some new peaks in FTIR spectra. The highest conducting PGE and NCPGE were chosen as an electrolyte to fabricate Lithium–Oxygen battery. Lithium–Oxygen battery containing nanocomposite polymer gel electrolytes exhibits the most stable performance with higher discharge time and discharge capacity. The formation of discharge products (i.e. Li_2O_2 and Li_2O) are also reduced since the nanosized silica particles enforcing their formation in only nanosize during discharge appear on the carbon air-electrode.

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