UNIVERSITI TEKNOLOGI MARA

# PREPARATION AND CHARACTERIZATION OF CHITOSAN GRAFTED PMMA VIA GAMMA RADIATION FOR ELECTROLYTE IN SUPERCAPACITOR

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Thesis submitted in fulfillment
of the requirements for the degree of
**Doctor of Philosophy**

**Faculty of Applied Sciences**

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# ABSTRACT

Solid polymer electrolytes may generally be defined as membranes that possess transport properties comparable with that of common liquid ionic solutions. In the present study, Chitosan (Ch), methyl methacrylate (MMA) were used to produce a grafted polymer Ch-g-PMMA using gamma irradiation grafting method. The grafted polymer Ch-g-PMMA, lithium triflouromethanesulfate ($LiCF_3SO_3$ or LiTf), ethylene carbonate (EC) and silicon dioxide ($SiO_2$) were then used in the preparation of solid polymer electrolytes (SPE), gel polymer electrolyte (GPE) and composite polymer electrolyte (CPE). All samples were prepared by solution cast technique. Physical and electrical properties of SPE, GPE and CPE were investigated to find the suitable grafted polymer electrolytes composition for fabrication in lithium polymer supercapacitor. The conductivity of all samples was calculated from the complex impedance plot in the frequency range 100 Hz to 1 MHz. The SPE film containing 50 wt% $LiCF_3SO_3$ in Ch-g-PMMA exhibits the highest conductivity of $1.42 \times 10^{-4}$ $Scm^{-1}$ at room temperature. The conductivity increased up to $2.96 \times 10^{-4}$ $Scm^{-1}$ when 30 wt% EC was added to Ch-g-PMMA with 50% $LiCF_3SO_3$. A further enhancement in the conductivity was obtained after ceramic filler particles ($SiO_2$) was incorporated to form CPE. The highest room temperature conductivity of $4.21 \times 10^{-4}$ $Scm^{-1}$ was achieved when 6 wt% $SiO_2$ was added to Ch-g-PMMA with 50 wt% $LiCF_3SO_3$ and 30 wt% EC. Attenuated total reflectance-Fourier transformed infrared (ATR-FTIR) spectroscopy justified that PMMA was successfully grafted onto the chitosan backbone. Differential scanning calorimetry (DSC) studies show that the plastisized sample presents a lower value of glass transition ($T_g$) compared to the plasticizer-free sample which was due to lubricating effect. The temperature dependence conductivity shows that the conductivity of SPE, GPE and CPE was thermally activated implying to obey the Arrhenius-rule. The electrochemical stability window for Ch-g-PMMA-LiTf-EC-$SiO_2$ system is up to 3.2 V. Electrical double layer capacitor (EDLC) with configuration C/(94[70 [50Ch-g-PMMA-50 $LiCF_3SO_3$]-30EC]-6 $SiO_2$)/C have exhibited 2.5 V working voltage and can perform for 500 cycles at current of 5 mA. Thus, it can be inferred that the samples could be a promising electrolyte candidate in any electrochemical devices particularly in the EDLC application.

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