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EFFECT OF ELECTRON BEAM IRRADIATION ON PMMA/ENR 50/$LiCF_3SO_3$ ELECTROLYTE

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Candidate'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 reference work, this thesis has not been submitted to any other academic institution or non-academic institution for any other degree or qualification.

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ABSTRACT

Thin, transparent and flexible freestanding films of poly (methyl methacrylate) (PMMA)/ 50% epoxidised natural rubber (ENR 50) blends were obtained in this study. However, the films obtained were not homogenous in which phase separations were observed on the surface of the films. The in-homogeneity of the blends were confirmed from their optical micrographs by the presence of black patches of highly viscous of ENR 50 that unable to enter into PMMA phase. The in-homogeneity of the blends were further confirmed by the presence of two T_gs in their differential scanning calorimetry (DSC) thermograms. Therefore, the films were irradiated with electron beam irradiation at 0, 10, 20 and 30 kGy of irradiation doses in order to improve the homogeneity of the films. Physically, the homogeneity of the films were slightly improved after irradiation. From the optical micrograph, the homogeneity of the blend was improved when it was irradiated up to 20 kGy of irradiation dose in which the presence of black patches of highly viscous phase of ENR 50 was almost diminishing. This can be further confirmed from its DSC thermogram in which the distance of the two glass transition temperature, T_gs were almost merging. From the Fourier Transform Infra Red (FTIR) analysis, this system exhibited the lowest intensity of C=O, O-CH_3 and C-O-C bands indicating an increased in the interaction of the polymers, hence giving a more stable film which has been confirmed from their thermo gravimetric (TG) analysis. However, above 20 kGy of irradiation dose, the black patches of highly viscous phase of ENR 50 were observed again. This was probably due to the reformation of interchain cross-linking that increased the viscosity of the polymer hence limits its mobility to enter into PMMA phase. This 20 kGy irradiated system exhibited the highest ionic conductivity of 1.79 x 10^{-7} S/cm at room temperature and the lowest activation energy, E_a when 0.2 g of lithium triflate ($LiCF_3SO_3$) salt was added into the system. From its FTIR analysis, it was exhibited the highest intensity of $\nu(SO_3)$ indicating the presence of the highest number of free lithium ion due to the formation of a less viscous phase of ENR 50. From its optical micrograph, it showed that the $LiCF_3SO_3$ salt was more soluble compared to the other irradiated system. However, no freestanding film can be obtained when more than 0.3 g of $LiCF_3SO_3$ salt was added into the system due to the formation of salt congestion. All the irradiated electrolyte systems were found to obey the Arrhenius rule in which the transportation of ions were via ion hopping.

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CHAPTER 1

INTRODUCTION

Liquid-based electrolytes are commonly used in many electrochemical devices such as toys, camera and etc. However, the liquid based electrolyte batteries exhibit problems such as leakage of toxic liquid and sometimes may cause explosion (Latif, 2005). Therefore, it is harmful to the environment and users. In addition, the liquid-based electrolyte batteries have short life time and need to be recharged for several hours (Murata *et al.*, 2000). Therefore, many researches had focused on a new solid based electrolyte system that exhibit several advantages over liquid electrolyte such as (Mohamed & Arof, 2004):

i. It is more stable than the liquid electrolyte.
ii. It has long shelf-lives and can be operated at a wide range of temperatures.
iii. It is easy to prepare in thin film form and safer to use.

There are several types of solid electrolyte that are:

i. Glass such as Ag^{+}-ion conducting on silver-borate glasses composite $(AgI)(Ag_2O{\cdot}B_2O_3)$ (Foltyn *et al.*, 2008).
ii. Crystalline compound such as 12-phosphotungstic acid (PWA) doped in silicon dioxide (SiO_2)/(polyethylene oxides (PEO)/polypropylene oxide(PPO)/polytetramethylene oxide (PTMO) (Honma *et al.*, 2001).
iii. Polymer electrolyte such as poly(vinyl chloride)(PVC)/poly(methyl methacrylate)(PMMA)/lithium perchlorate ($LiClO_4$) (Choi & Park, 2001).