

UNIVERSITI TEKNOLOGI MARA

**STRUCTURAL, THERMAL AND
ELECTRICAL PROPERTIES OF
PMMA/ENR 50/LIBF₄
ELECTROLYTES CONTAINING
CARBOXYLIC ACIDS MODIFIED
SiO₂ FILLERS**

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ABSTRACT

Study on PMMA/ENR 50 blends as electrolyte is still being carried out because this blend has produced flexible and free standing film of PMMA. Since the blend is not homogeneous, several modifications have been done to improve the phase separations of the blend such as adding plasticizers and fillers. It was found that the addition of filler such as SiO₂ not only has improved the homogeneity of the blend yet enhance the ionic conductivity of the polymer blend systems. Unfortunately, the filler tends to agglomerate due to the formation of silanol groups (Si-OH) that is prone to react with the surrounding moisture. Therefore, in this study carboxylic acids of various carbon chain lengths; i.e. butanoic acid (C₄), octanoic acid (C₈), dodecanoic acid (C₁₂) and hexadecanoic acid (C₁₆) has been used to reduce the number of silanol groups by replacing the -H atom from the silanol group with the R_nCOO⁻ of the acid via esterification technique. The presence of R_nCOO⁻ on modified SiO₂ (MoC_x-SiO₂) filler were confirmed by elemental analysis (CHNS) and Fourier Transform Infrared Spectroscopy (FTIR). It was found that MoC₁₂-SiO₂ filler exhibited the highest percentage of -H replacement in which 61% of H from Si-OH group has been reduced. From Emission Scanning Electron Microscopy (FESEM) analysis, MoC₁₂-SiO₂ filler showed a compact structure indicating the C₁₂ has filled up the porous structure of SiO₂ particles. This MoC₁₂-SiO₂ filler also gave the smoothest surface with less agglomerate when added into PMMA/ENR 50 blend (PEMoC₁₂S) systems with the two single glass transition temperature (T_gs) of the separated phase almost merging. The less MoC_x-SiO₂ filler agglomerates also increased the flexibility of polymer chains where the polymer chains were able to freely rotate. However, the film obtained was slightly brittle due to the formation of polymer networking via hydrogen bonding between the main functional group of C=O, OCH₃ and COC of the polymers and the acid modified filler. This interaction has been confirmed from its FTIR analyses in which the intensity of OH band was slightly increased in PELMoC_xS systems. However, this interaction was not excessive as the two T_gs obtained in PELMoC_xS systems was lower than PELS systems. Due to this, these PELMoC_xS systems also exhibited higher thermal degradation temperature, T_d than the PELS system. The highest ionic conductivity of 5.56 x 10⁻⁷ S/cm at room temperature was achieved in PELMoC₁₂S film due to the formation of a smoother surface for chain rotation and ion transport. Both polymer systems obeyed Arrhenius rule and the lowest activation energy, E_a also achieved in PELMoC₁₂S system.

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TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENT	vi
LIST OF TABLES	ix
LIST OF FIGURES	x
LIST OF SYMBOLS	xiv
LIST OF ABBREVIATIONS	xv
CHAPTER ONE: INTRODUCTION	1
1.1 Research Background	1
1.2 Problem Statements	3
1.3 Objectives of Study	3
1.4 Scope of Study	4
1.5 Significance of Study	5
CHAPTER TWO: LITERATURE REVIEW	6
2.1 Introduction of Polymer Electrolyte (PE)	6
2.2 Role of Fillers in Composite Polymer Electrolytes (CPE)	8
2.3 Modified Inorganic Filler	11
2.3.1 Silane Coupling Modified Inorganic Filler	11
2.3.2 Polymer Modified Inorganic Filler	12
2.3.3 Acids Modified Inorganic Filler	14
2.4 Poly(Methyl)Methacrylate (PMMA) as a Polymer Host	16
2.5 50 % Epoxidized Natural Rubber (ENR 50)	18
2.6 Selection of Doping Salt	19

2.7	Materials Characterization	20
2.8	Elemental Analysis	20
2.9	Fourier Transform Infrared Spectroscopy (FTIR)	22
2.10	Morphological Study of Filler and PEs	24
2.11	Thermal Analysis Study on PE Systems by Differential Scanning Calorimetry (DSC)	26
2.12	Stability Study on PE Systems by Thermal Gravimetric Analysis (TGA)	29
2.13	Ionic Conductivity Study of Polymer Electrolyte Systems	31
2.14	Ionic Conduction Mechanism	33
2.15	Dielectric Studies on Polymer Electrolytes	37
2.16	Modulus Studies on Polymer Electrolytes	39

CHAPTER THREE: RESEARCH METHODOLOGY 40

3.1	Introduction	40
3.2	Modification of SiO ₂ Filler Using Carboxylic Acids of Various Chain Lengths	40
3.3	Preparation of Stock Solution	41
3.4	Fabrication of PMMA/ENR 50/PMMA/ENR 50/MoC _x -SiO ₂ Films	41
3.5	Samples Characterization	42
3.5.1	Elemental Analysis	42
3.5.2	Fourier Transform Infrared Spectroscopy (FTIR) on the Fillers and Polymer Blend Films	42
3.5.3	Field Emission Scanning Electron Microscopy (FESEM)	42
3.5.4	Differential Scanning Calorimetry (DSC)	43
3.5.5	Thermogravimetry Analysis (TGA)	43
3.5.6	Impedance Spectroscopy	43

CHAPTER FOUR: RESULT AND DISCUSSION

MODIFICATION OF MOC_x-SIO₂ FILLER AND ITS EFFECT ON THE PROPERTIES OF PMMA/ENR 50 BLENDS		44
4.1	Introduction	44
4.2	Modification of MoC _x -SiO ₂ Filler	44