

**UNIVERSITI TEKNOLOGI MARA**

**PREPARATION AND  
CHARACTERIZATION OF CELLULOSE -  
BASED POLYMER ELECTROLYTES FOR  
HUMIDITY SENSORS APPLICATION**

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## ABSTRACT

Conventional polymer electrolytes use synthetic polymer. This work is an effort to substitute synthetic polymer by eco – friendly “ biopolymer” for developing proton conducting polymer. Methyl cellulose (MC) was used as host polymer and orthophosphoric ( $H_3PO_4$ ) acid was the ionic dopant. A further modification was done by dispersing  $SiO_2$  fillers to obtain polymer – ceramic composites. All films were prepared by “solution – casting technique”. The variation in films morphology has been examined using Scanning Electron Microscopy (SEM) while the interactions between MC -  $H_3PO_4$  and MC -  $H_3PO_4$  –  $SiO_2$  investigated using Fourier Transform Infrared Spectroscopy (FTIR). IR studies revealed that neither  $H_3PO_4$  nor  $SiO_2$  interact with MC because no bands were found to shift. Furthermore, the conductivity of methyl cellulose films containing 0 – 5 M  $H_3PO_4$  and 3M  $H_3PO_4$ + (1-10wt.%  $SiO_2$ ) have been determined using Impedance Spectroscopy (IS). The incorporation of  $H_3PO_4$  in MC was found to introduce pores in the MC films whose number increased with the concentration of  $H_3PO_4$ . The addition of  $SiO_2$  led to the formation of  $SiO_2$  – polymer network interfaces as well as partial blocking of pores as  $SiO_2$  (at large concentration) started to cluster around the periphery of pores. It has been proposed that MC -  $H_3PO_4$  form “solvent swollen polymer” and the conductivity increases as more  $H_3PO_4$  is incorporate in it. The conductivity for MC + x.  $H_3PO_4$  films increase in the range of 0 to 4 M. For  $x > 4$  M, the conductivity decreased and the structure collapsed into a glue like- material. The highest ionic conductivity i.e.  $2.30 \times 10^{-3} \text{ Scm}^{-1}$  was obtained when 4 M  $H_3PO_4$  was introduced into MC films. The conductivity enhancement in  $SiO_2$  – composites showed that there are two threshold peaks upon addition of  $SiO_2$ . These results have been explained as “Percolation threshold model”.

Another aspect of the present work is fabrication and characterization of a “Humidity Sensor” using our MC – based polymer electrolyte composites. The humidity sensor was prepared by depositing “sputtered finger electrode sometimes referred as digital electrode” of gold on the electrolyte film on a glass substrated. The best sensor resistance (or impedance,  $Z_r$ ) changed with relative humidity. The sensor was stable upto R.H ~ 70 % and beyond this it degraded and MC films needs to be further improved.

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## TABLE OF CONTENTS

	<b>Page</b>
<b>DECLARATION</b>	<b>ii</b>
<b>ABSTRACT</b>	<b>iii</b>
<b>ACKNOWLEDGEMENT</b>	<b>iv</b>
<b>TABLE OF CONTENTS</b>	<b>v</b>
<b>LIST OF TABLES</b>	<b>ix</b>
<b>LIST OF FIGURES</b>	<b>x</b>
<b>LIST OF ABBREVIATIONS</b>	<b>xiv</b>
<b>LIST OF PUBLICATIONS</b>	<b>xv</b>
<b>CHAPTER 1: INTRODUCTION</b>	
1.1 Background	1
1.2 Humidity sensor and need of proton conducting polymer	2
1.3 Proton conducting polymer electrolyte as possible humidity sensing material	3
1.4 Problem identification	5
1.5 Objectives of the research	5
1.5 Scope of the thesis	6
1.6 Research aims and rationale	7
<b>CHAPTER 2: LITERATURE REVIEW</b>	
2.1 Introduction	8
2.2 Polymer electrolyte: General	9
2.3 Proton Conducting Electrolyte: General Description	12
2.3.1 <i>Methods to modify polymer electrolyte composites</i>	14
2.3.2 <i>Biopolymer methyl cellulose (MC): a possible polymer for developing novel proton conducting polymer electrolyte composites</i>	15

# CHAPTER 1

## INTRODUCTION

### 1.1 BACKGROUND

“Solid State Electronics” is a well known branch of science and technology which is responsible for rapid development of Integrated Circuits (IC’s) leading to miniaturised electronic devices, computers etc. The basic materials used in Solid State Electronics are semiconductors which are “electronic conductors”. A new branch of science and technology started in 1967 after the discovery of “good Solid State Ionic Conductors or Solid Electrolyte” viz.  $\text{RbAgI}_5$  and  $\beta$  – Alumina (Owens and Argue, 1967; Kummer, 1967; Chandra, 1981). This branch of technology is called “SOLID STATE IONICS” because it uses “ionic conductors” as basic device materials. Since the first discovery in 1967, a large number of solid electrolytes have been discovered including polycrystalline, glassy, gel, polymer, composites etc. The polymer electrolytes and polymer – ceramic composites are preferred materials because of many advantage like low cost, ease of fabrication in thin film form, mouldability etc. Most of the ionic conductor are have *mobile ions with small ionic radii* like  $\text{Ag}^+$ ,  $\text{Cu}^+$ ,  $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{H}^+$ ,  $\text{F}^-$ ,  $\text{O}^{2-}$  etc. These have been used in developing solid state ionic devices like batteries, fuel cell, sensors, electrochromic display etc. The present work is concerned with developing proton conducting polymer and its application as gas sensor (viz. Humidity Sensor). Hence, in the following sections, we give highlights of polymer electrolytes and humidity sensor before actually describing the scope of the thesis.