CHAPTER 1

INTRODUCTION

1.1 Research background

Various efforts have been devoted to develop solid polymer electrolytes (SPE) due to its potential to replace liquid electrolytes (LE) that are known to give problems in devices. The SPE membrane will act as separator and electrolyte and will save cost in the fabrication of electrochemical devices such as electrical double layer capacitors (EDLC), polymer exchange membrane fuel cells (PEMFC), batteries and solar cells. Examples of such studies include that of Latham et al. (2002) who studied the supercapacitor fabricated from poly (urethane) electrolytes and composite electrodes. The highest capacitance value obtained is 35 F g⁻¹. Chitosan-based membrane fuel cell has been studied by Wan et al. (2008) and a current density of 65 mA cm⁻² was achieved at 50 °C. Lakshmi and Chandra (2002) have fabricated solid-state proton batteries using phosphotungstic and phosphomolybdic acid composite electrolytes and obtained an open circuit voltage of 1.5 V. Singh et al. (2008) studied solid electrolytes based on 1-methyl 3-propyl imidazolium iodide (PMII) and polyethylene oxide (PEO) for dye-sensitized solar cell application. Some polymers used as SPEs are chitosan [Buraidah et al., 2009; Yahya and Arof, 2004; Du et al., 2011; Choudhury et al., 2011], polyethylene oxide (PEO) [Ali et al., 1998], and poly (vinyl alcohol) (PVA) [Rajendran et al., 2003].

1.2 Problem statement

As briefed in the research background, SPEs have been developed to overcome drawbacks of LE such as leak prone, corrosive, and can have hazardous effects to the environment. However SPEs also have weaknesses such as low conductivity, low mechanical strength, poor contact and limitation of working voltage. Due to these, a lot of efforts have been carried out to upgrade thermal and electrochemical stability, mechanical strength, film forming properties, electrode-electrolyte contact when SPEs are used in electrochemical devices and most importantly it must be safe to people and the environment.

1.3 Scope and objective of the research

Based on the issues mentioned in the problem statement, the main purpose of this dissertation is to develop a solid polymer electrolyte with high conductivity using methyl cellulose (MC), which is a biodegradable polymer. In this dissertation, MC is suggested as an alternative polymer host. MC based polymer electrolyte was prepared by solution casting technique. Two types of MC based polymer electrolytes were prepared. These are unplasticized MC-NH₄NO₃ and plasticized MC-NH₄NO₃ with poly (ethylene glycol), PEG. The present work focus on the conductivity of MC based polymer electrolyte samples and the factors that influence the increase or decrease in conductivity such as the nature of samples, the interaction between materials in samples and thermal stability of samples. Experiments were carried out using some techniques. These are:

• Electrochemical Impedance Spectroscopy (EIS)

EIS was used to determine the impedance of samples at room and elevated temperatures. Conductivity will be calculated using the appropriate equation. The effect of temperature on the conductivity of the prepared samples will be investigated in the temperature range from 298 K to 373 K. Once the polymer-salt (MC doped with NH_4NO_3 salt) composition has been optimized, PEG will be added. To understand conductivity variation with NH_4NO_3 and PEG at room temperature, XRD will be carried out.

• X- Ray Diffraction (XRD)

The study on the nature of the films (whether crystalline, amorphous or both) was performed using XRD technique. The degree of crystallinity was evaluated using the criterion given by Hodge *et al.* (1996). The relation between the degree of crystallinity or crystalline fraction with conductivity variation with NH_4NO_3 and PEG will be explained. For support to these efforts, FTIR have to be carried out.

• Fourier Transform Infrared (FTIR)

The interaction between the MC-NH₄NO₃, MC-PEG, NH₄NO₃-PEG and MC-NH₄NO₃-PEG will be investigated using FTIR technique. This technique can also be used to support recrystallization of salt observed from XRD.

• Thermalgravimetric analysis (TGA)

TGA was used to determine the thermal stability of the prepared samples and understand the effect of NH_4NO_3 and PEG on the decomposition and degradation temperature of MC.

• Linear sweep voltammetry (LSV)

Electrochemical stability of prepared samples will be determined using LSV technique to get the information about limitation voltage of the samples before using them as membrane in any devices.

Development of the SPE using MC forms the primary objective of the work. The second objective of this dissertation is to develop an electrical double layer capacitor (EDLC) using activated carbon electrode prepared by the dip-coating method and using the highest conducting sample from plasticized system as membrane or separator.

1.4 Thesis organization

This thesis has eight chapters. Each chapter describes an aspect of the present study.

Chapter 1 is a short introduction which summarizes the research work. The scope and objectives of the thesis are emphasized in this chapter.

Chapter 2 contains literature review. This chapter gives an inexhaustive overview of polymer electrolyte, plasticized polymer electrolytes, basic properties of polymer electrolytes, types of polymer electrolytes, proton conducting polymer electrolytes and their examples. In this chapter, methyl cellulose (MC), ammonium nitrate (NH₄NO₃) and poly (ethylene glycol) (PEG) with their general properties are introduced as materials in this work. The fundamental information about electrical double layer capacitor are also explained.

Chapter 3 described the experimental methods used in this work. It explains the preparation of MC based polymer electrolyte using solution cast technique and the preparation to make electrode slurry and EDLC. The characterizations carried out to study details of the prepared components and devices are also described.

Chapter 4, 5 and 6 are important chapters in this dissertation because details and explanations about the results can be obtained from these chapters. Chapter 4 gives results on unplasticized MC-NH₄NO₃ polymer electrolyte system. Chapter 5 presents results on plasticized MC-NH₄NO₃ polymer electrolyte system and results on EDLC and their performance are displayed in Chapter 6.

Chapter 7 will discuss the overall results obtained in this work and finally Chapter 8 will conclude the dissertation with some suggestions for further studies.