ELECTROCHEMICAL PROPERTIES OF NANO-COMPOSITE POLYMER BLEND GEL ELECTROLYTES FOR BATTERY APPLICATIONS

SYNOPSIS OF THE THESIS SUBMITTED

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Introduction

Solid State Ionics (SSI) is an interdisciplinary area of Science and Technology that deals with the study of ionic solid having/exhibiting a wide range of ionic conductivity from 10^{-13} – 10^{-1} S cm^{-1} [1][2]. The class of Solid Material having ionic conductivity of the order of 10^{-5} – 10^{-1} S cm^{-1} comparable to those liquid electrolytes are referred to as "Fast Ion Conductor" or "Superionic Solid". These materials (show tremendous promises) have a wide variety of applications in electrochemical devices such as batteries, supercapacitors, fuel cell sensors, electrochromic display devices, etc [3]. A Superionic Solid or Solid Electrolytes have the following properties:

- High ionic conductivity of the order of 10^{-5} – 10^{-1} S/cm.
- Ions as principle charge carriers i.e., $t_{ion} \approx 1$.
- Negligible electronic conductivity (Small electronic transference number ($t_e < 10^{-4}$).
- Low Activation Energy (0.1 ev – 1 ev).

Based on the microstructure and physical properties, the ion-conducting material is classified as (i) Framework crystalline / polycrystalline solid electrolyte (ii) Glassy / Amorphous Solid Electrolytes (iii) Composite Solid Electrolytes (iv) Polymer-based electrolyte [4][5]. Among these all, polymer-based electrolytes have attracted the interest of scientists due to high ionic conductivity near to that of liquid electrolytes ease of fabrication in form of thin film, good electrode-electrolyte contacts, free from leakage as compared to traditional liquid electrolytes. It has superior properties as compared to other solid electrolytes [6]. Polymer solid electrolytes are formed by adding alkali metal salt MX (M = Li, Na, Ag, Cu, etc.) and (X = I, F, Cl, etc.) having low lattice energy and bulky anion [7] in host polar polymers like polyethylene oxide (PEO), polypropylene oxide, etc. The first polymer electrolyte was reported by Fenton et. al. in 1973 by complexing alkali metal salts in a high molecular weight polar polymer polyethylene oxide (PEO) [8] and then Wright et. al. reported the electrical properties of this polymer salt complexes [9]. Later, Armand et. al. in 1979 in their work, demonstrated the battery application by using PEO and lithium salt complex solid polymer electrolyte (i.e., PEO – Li^+ system) [5].

Polymer electrolytes should possess the following properties to be used in the development of solid-state devices:

- High ionic conductivity $\sigma \geq 10^{-4}$ S cm^{-1} at room temperature close to liquid electrolytes.
- High ionic transference number $t_{ion} \sim 1$.

- Good mechanical, thermal, and chemical stability. High chemical stability is required to avoid chemical reaction at electrode-electrolyte interfaces.
- Wider electrochemical stability window as high as 4 to 5 V.
- Low activation energies for ion conduction.
- Low glass transition temperature ($\boldsymbol{T_g}$) is required for high ionic conductivity.

Based on the preparation method and physical properties, structural properties, the polymer electrolytes are classified into the following categories: (1) Solvent-free polymer salt complexes or dry solid polymer electrolytes (2) Plasticized polymer electrolytes (3) Rubbery polymer electrolytes (4) Gel polymer electrolytes (5) Composite polymer electrolytes.

The conventional solid polymer electrolytes (polymer-salt complexes) possess very low ionic conductivity because ion transportation through PE is governed by polymer chain segmental motion as well as local relaxation which is not possible in the partially crystalline PE. For that, a high degree of amorphicity in polymer electrolyte is required. Alternatively, the gel polymer electrolyte system has been found as a potential candidate due to the possessor of high ionic conductivity of the order of 10^{-4} to 10^{-3} S cm^{-1}. Apart from it, other few interesting points are no leakage like a liquid electrolyte, easy to prepare, and shaped it in the form of thin-film, no degradation, high compatibility with electrodes, high energy density etc. Gel polymer electrolytes (GPEs) are usually obtained by incorporating a large amount of organic liquid solvents/plasticizers into a polymer matrix. GPE is neither solid nor liquid [10]. The organic solvent/plasticizer remains trapped within the polymer matrix and gel is formed or polymer network swelled with organic plasticizers hence it possesses both the cohesive properties of solid and diffusive properties of the liquid. The ionic conductivity of these GPEs is comparable to liquid electrolytes. The idea of the plasticizing polymer was proposed by Feuillade and Perche in 1975 [11]. The addition of plasticizers can result in greater ion dissociation which increases the number of charge carriers for ionic transport [12] and enhances the flexibility of the polymer chain hence mobility of ion. The mobility of ions in the PE determines the conductivity σ and the performance of the PE. A low molecular weight plasticizer, such as ethylene carbonate (EC), propylene carbonate (PC), dimethyl carbonate (DMC), etc. improves the electrical conductivity of PE by enhancing polymer chains flexibility and also improve the stability of the electrode/electrolyte interface [13]. But with the addition of plasticizers into the polymer matrix, the mechanical strength becomes poor which is not suitable with electrodes [14]. The researchers have adopted a few methods to improve ionic conductivity and mechanical strength such as blending, adding nanoparticles, and crosslinking, etc. [13]. A mixture of two polymers with or without any chemical bonding is known as polymer blending and a pictorial model of the polymer blend is shown in Fig.1.

Nicotera et al. have demonstrated a notable enhancement of lithium-ion conduction in PMMA-PVDF based blend polymer electrolytes [15]. A gel polymer electrolyte comprising of PMMA and PVDF polymers was found to be a superior electrolyte for the improvement of a lithium battery by Rajendran et al. [16]. Improvement in ionic conductivity after the blending of PMMA – PVC based polymer blending with LiTFSI salt is also reported [17].

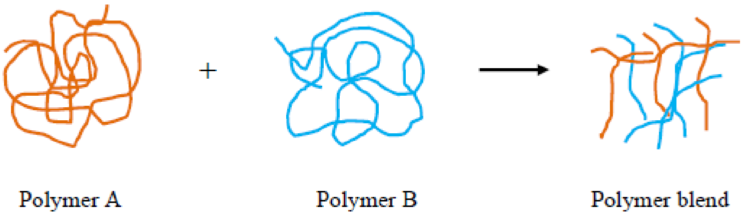


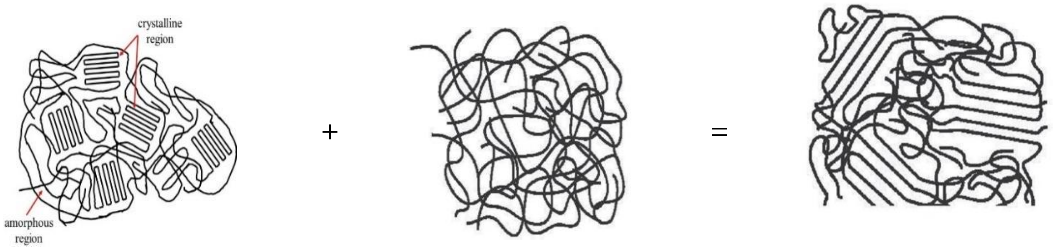
Figure 1 Pictorial model of the polymer blend

Another pragmatic and simplest approach is to a dispersion of nano-fillers (inert insulating ceramic filler) such as SiO_2, TiO_2, ZrO_2, $BaTiO_3$, Al_2O_3, ZnO into a polymer matrix to improve its structural and electrochemical properties[18]. The addition of a small amount of nano-filler enhances the electrical properties of the polymer electrolytes by increasing the degree of amorphicity. Apart from it, it also provides better mechanical integrity, thermal stability, interfacial stability in contact with polymer electrolytes with various electrodes. The improvement in ionic conductivity and amorphous phase on the addition of Al_2O_3, SiO_2 nano-filler in PEO-PMMA-$LiClO_4$ system has been reported by Choudhary et al.[18]. Xiong et al. have shown improvement in the mechanical properties and reduction in the crystallinity when silica nanoparticles are incorporated in starch-based polymer electrolytes [19]. Khanmirzaei et al. reported an enhancement in ionic conductivity in biopolymer doped with TiO_2 nanoparticles [20].

A large number of polymer electrolytes system have been reported using various polymer host such as poly (ethylene oxide) PEO, poly (acrylonitrile) (PAN), poly (methyl methacrylate) (PMMA), poly (vinylidene fluoride) (PVDF), poly (vinylidene fluoride–co–hexafluoropropylene) (PVDF–co–HFP), etc. with high ionic conductivity ~ 10^{-4} – 10^{-3} S cm^{-1} at ambient temperature [21][22][23]. However, still it needs to improve the electrochemical properties of gel polymer electrolytes to be used in electrochemical batteries to increase the discharge capacity, energy density, power density, etc., A polymer electrolytes play a vital role in a battery as a separator. The electrical properties of polymer electrolyte affected by various parameters such as simultaneous anions and cations motion, ion-pair formation, amorphous nature of the film, less viscous medium for the motion of ions, and high dielectric constant. Hence our motivation is to improve the electrochemical properties of gel polymer electrolyte. The main objective of the present thesis is "**Electrochemical properties of nano-composite polymer blend gel electrolytes for battery applications**".

Current work of the Thesis

The present thesis has been focused on the preparation and characterization of Li^+ ion conducting gel polymer electrolytes. The present gel polymer electrolyte is comprised of poly (vinylidene fluoride hexafluoropropylene) PVDF-HFP and poly (methyl methacrylate) PMMA as host polymer. PVDF-HFP has received a lot of attention due to its excellent properties such as a low degree of crystallinity and high dielectric constant $\varepsilon \approx 8.4$ that help in higher dissociation of lithium salt. The PVDF-HFP semicrystalline polymer possesses both amorphous and crystalline phases in which the amorphous HFP part can trap large amounts of liquid electrolytes whereas the crystalline VDF part assists in the improvement of the mechanical stability for the formation of a free-standing film [24]. Along with this PMMA is blended with it owing to its amorphous nature which aids in increasing the amorphicity. Along with this PMMA is used as a blender with PVDF–HFP polymer due to its advantageous properties such as good compatible nature with other polymers, high chemical resistance, electrical properties, and highly amorphous in nature which aid for increasing the amorphicity [25]. A schematic diagram of the structure for the blended polymer electrolyte is shown in Fig. 2.



Semicrystalline polymer

Amorphous polymer

Polymer with the more amorphous region

Figure 2 Schematic diagram of the structure for the blended polymer electrolyte

As a conducting species, $LiClO_4$ salt is used. Propylene carbonate (PC) and diethylene carbonate (DEC) is taken as plasticizers or organic solvent. PC has a high dielectric constant ($\varepsilon \approx 64.6$) but has high viscosity ($\eta \approx 2.53$ mPa s), whereas DEC has a low dielectric constant ($\varepsilon \approx 2.82$) and low viscosity ($\eta \approx 0.748$ mPa s). So, a mixture of PC: DEC (1:1) plasticizers having high dielectric constant and lower viscosity is used to achieve high ionic conductivity. For the preparation of nanocomposite gel polymer electrolyte films, Al_2O_3 nanoparticles are used as filler owing to its high surface to volume ratio and high dielectric constant. The films were prepared by solvent casting technique. The prepared gel polymer electrolyte films were characterized by using a various technique such as structural, thermal, mechanical, electrical, electrochemical techniques. The following series were prepared.

Series (a)

PVDF-HFP: PMMA + PC: DEC + X wt.% $LiClO_4$ where X = 2, 4, 5, 7.5, 10

Series (b)

PVDF-HFP: PMMA + $LiClO_4$ + X wt.% PC: DEC where X = 20, 30, 40, 50, 60

Series (c)

PVDF-HFP: PMMA+ $LiClO_4$ + PC: DEC +X wt.% Al_2O_3 where X = 0.5, 1, 2, 3, 4

The aim of the present study may fold

(1) Preparation of all series of gel polymer electrolytes using solution cast technique.
(2) To study the effect of variation of different compositions on the structural, thermal, and mechanical properties using XRD, SEM, FTIR, AFM, DSC, and Tensile strength respectively.
(3) Investigation of the electrochemical properties of the prepared GPE membrane by adopting Electrochemical impedance Spectroscopy and Cyclic voltammetry.
(4) Fabrication of battery using optimized GPE membranes from each series and measurement of the discharge characteristics of the fabricated battery.

The entire work of the thesis is organized in **seven** chapters as summarized below.

Chapter 1 present history and general overview of the classification of Solid-State Ionics materials or fast ionic conductor (FICs). A detail information regarding polymer electrolytes and its classifications such as dry solid polymer electrolytes, plasticized polymer electrolytes, rubbery polymer electrolytes, gel polymer electrolytes, composite polymer electrolytes are briefly discussed in this chapter. The essential properties of host polymers, salts, plasticizers, and nano-fillers to fulfill the requirement of gel polymer electrolytes to be used in electrochemical devices are explained in detail. The possible applications of the polymer electrolyte in various electrochemical devices such as Electrochemical Capacitor or Supercapacitor, Fuel cell, Dye-sensitized solar cell (DSSC), Electrochromic display devices (ECDs), Sensors, Batteries have been presented also.

In **Chapter 2**, several theories have been explained to understand the ion conduction mechanism in the polymer electrolytes. Various theoretical models have been proposed to explain ion conduction mechanism in disordered materials which are applicable to polymer electrolytes systems are discussed such as Free Volume Model, Configurational Entropy Model, Static Bond Percolation Model, Dynamic Bond Percolation Model (DBP), Space Charge Model, Amorphous Phase Model, Effective Medium Theory. For explaining the temperature dependence of ionic conductivity, a number of empirical relations such as the Arrhenius equation, Vogel-Tamman-Fulcher (VTF) equation, Williams-Landel-Ferry (WLF) equation, etc have been reviewed. The applicability of Jonscher's power law to Frequency-dependent conductivity is also discussed.

Chapter 3 gives details about used materials in the GPE system, techniques to fabricate the present gel polymer electrolyte system, and characterization techniques. To prepare the gel polymer electrolyte membrane, a typical solvent casting technique has been adopted and the preparation method is discussed in detail and also represented in the form of a chart. The physical characterization techniques such as X-ray diffraction (XRD), Fourier Transform infra-red spectroscopy (FTIR), Scanning electron microscope (SEM), Differential scanning calorimetry (DSC), Atomic force microscope (AFM), Tensile strength are explained to discover structural, morphological, thermal and mechanical properties. Electrochemical impedance spectroscopy, Linear sweep voltammetry, and Transport number measurement techniques are presented for the electrical and electrochemical analysis of gel polymer electrolytes.

Chapter 4 emphasizes the characterization results obtained from the different characterization techniques. The variation of $LiClO_4$ salt, PC: DEC plasticizers, and Al_2O_3

nano-fillers in the present GPE system on the structural, morphological, thermal, and mechanical properties have been analyzed. Transport number measurement and cyclic voltammetry and electrochemical stability window of all series are reported in this chapter.

Chapter 5 reports the electrical properties in which ionic conductivity, temperature-dependent ionic conductivity, AC conductivity, dielectric properties, and modulus properties have been discussed for all prepared GPE series. The temperature dependence of ionic conductivity may provide valuable information for ionic conduction behavior. The temperature dependence of the ionic conductivity of the prepared GPE membrane has been found to follow the Arrhenius-type thermally activated process for the membranes. AC conductivity gives an understanding of electrical relaxation phenomena. This is commonly used to characterize the ion transport phenomenon occurring in ion-conducting material in the frequency domain. The applicability of Jonscher's power law to AC conductivity has been observed. The composition and temperature-dependent dielectric properties such as dielectric constant (ε'), tangent loss ($\tan \delta$) as a function of frequency have been elaborated. In dielectric analysis electrode-electrolyte effect is so prominent which suppresses the high-frequency bulk region. to overcome this problem, the modulus analysis is carried out to study conductivity relaxation phenomena. In addition to these, to gain an insight into the temperature and composition dependence of the ion dynamics, the scaling behavior of the conductivity and modulus spectra has been undertaken. The scaling (time-temperature superposition principle/thermorheological simplicity) is a tool that helps us to examine whether the conduction mechanism involving ion dynamics is temperature-dependent or not.

Chapter 6 discusses the basic working principle of battery and fabrication of primary battery using optimized gel polymer electrolytes membranes from all series as a separator sandwiched between the suitable anode and cathode materials. The fabricated batteries have been characterized by measuring open-circuit voltage (OCV) and discharge characteristics at no load and constant load respectively. Various battery parameters such as current density, discharge capacity, power density, and energy density from the discharge profile have been reported.

Chapter 7 summarizes all the characterization results reported in the thesis and provides overall conclusions of the presented research work.

The proposed content of the Thesis

1 Introduction

1.1 Introduction on Solid State Ionics

1.2 Classification of Ion-Conducting Materials

  1.2.1 Framework crystalline / polycrystalline solid electrolyte

  1.2.2 Glassy /Amorphous Solid Electrolytes

  1.2.3 Composite Solid Electrolyte

1.3 Polymer Electrolytes

  1.3.1 Polymer based electrolytes: An Overview

  1.3.2 Classification of Polymer Electrolytes:

    1.3.2.1 Solvent-free polymer salt complexes or dry solid polymer electrolytes

    1.3.2.2 Plasticized Polymer Electrolytes

    1.3.2.3 Rubbery Polymer Electrolytes OR Polymer-in-Salt Systems

    1.3.2.4 Gel Polymer Electrolytes

    1.3.2.5 Composite Polymer Electrolytes

1.4 Applications of Polymer electrolytes

1.5 Motivation and objective of the present work

References

2 Theoretical Details

2.1 Introduction

2.2 Theoretical models for Temperature Dependence of Ionic Conductivity

  2.2.1 Arrhenius Theory / Arrhenius model for ion transport

  2.2.2 VTF Theory / Vogel – Tammann – Fulcher Theory

  2.2.3 WLF (Williams – Landel – Ferry) Theory

2.3 Theoretical Models for DC Conductivity

  2.3.1 Free Volume Model

  2.3.2 Configurational Entropy Model

  2.3.3 Static Bond Percolation Model

  2.3.4 Dynamic Bond Percolation Model (DBP)

  2.3.5 Space Charge Model

  2.3.6 Amorphous Phase Model

3 Experimental details

4 Characterization Results

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