SYHNTHESIS AND CHARACTERIZATION OF GEL POLYMER ELECTROLYTE BASED IONIC LIQUID FOR BATTERY APPLICATION

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CHEMICAL ENGINEERING
UNIVERSITI TEKNOLOGI PETRONAS
SEPTEMBER 2015

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by
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Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

SEPTEMBER 2015

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Perak

CERTIFICATION OF APPROVAL

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Approved by,

____

(Dr Nurul Ekmi bt Rabat)

UNIVERSITI TENOLOGI PETRONAS
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September 2015

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contain herein have not been undertaken or done by unspecified sources or person.

MOHAMED ASYRAF BIN MOHAMED LIAS

ABSTRACT

In order to keep the rapid growth of development about the portable electronic device, improving the performance of gel polymer electrolyte (GPE) has become the goal of this research. This research has improved the performance of liquid electrolyte battery through replacing it with GPE. GPE was synthesized by the combination of polymer, ionic solution (1-ethyl-3-methylimidazolium trifluoro-methanesulfonate (EMITf) and salt solution (magnesium triflate (Mg (Tf) $_2$).GPE was tested with different ratio between electrolyte and polymer to study the best ratio with good stability, conductivity and crystalline structure. Then three different polymers compared were Poly (vinylidene fluoride hexafluoropropylene) (PVdF-HFP), Polyvinyl achohol (PVA) and Poly polyvinyl alcohol-co- Poly (vinylidene fluoride hexafluoropropylene) (PVA-PVdf-HFP) to investigate the highest conductivity among the polymers. Fourier Transform infrared (FTIR), Thermogravimetric analysis (TGA) and scanning electron microscopic (SEM) and conductivity test were used to investigate GPE conductivity and thermal properties. PVA-PVdf-HFP had shown a good result in ratio of GPE 5:1.75 electrolyte to polymer. In addition, PVA-PVdf-HFP based GPE shows semi crystalline microstructure and amorphous region picture. Meanwhile, the Thermogravimetric Analysis (TGA) test had shown below than 20 percent loss in weight after blend up to 260 ^{o}C. Lastly, the DC conductivity measurement had shown a measurement 3.125 x 10^{-4}Scm^{-1}. Based on the research, PVA-PVdf-HFP had shown the best conductivity and properties to act as the host polymer in GPE.

ACKNOWLEDGEMENT

I would like to express my deepest appreciation to all those who provided me the possibility to complete this report. A special gratitude I give to my final year project supervisor, Dr. Nurul Ekmi bt Rabat, whose contribution in stimulating suggestions and encouragement, helped me to coordinate my project especially in writing this report throughout this semester.

Furthermore I would also like to acknowledge with much appreciation the crucial role of the staff, who gave the permission to use all required equipment and the necessary materials to complete my final year project. A special thanks goes to my friends, who gave suggestion and opinion about my final year project. I have to appreciate the guidance given by other supervisor as well as the panels especially in our project presentation that has improved our presentation skills thanks to their comment and advice.

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LIST OF ABBREVIATIONS

CPE	Composite polymer electrolyte
DSC	Differential Scanning Calorimetry
EC	Ethylene carbonate
EMITF	1-ethyl-3-methylimidazolium trifluoro-methanesulfonate
FTIR	Fourier Transform Infrared
GPE	Gel Polymer electrolyte
g equiv.$^{-1}$	Unit of Conductivity
Hz	Hertz
k	Constant
Li+	Lithium ion
Li	Lithium
LiBF4	Lithium Fluoroborate
$LiCLO_2$	Lithium monochloride dioxide
Mg2+	Magnesium ion
$Mg (Tf)_2$	Magnesium triflate
PC	Polycarboxylate
PEO	Polyethylene oxide
PPO	Poly propylene oxide
PS	Polystyrene
PMMA	Polymethyl methacrylate
PVdF-HFP	Poly (vinylidene fluoride-hexafluoropropylene)
PVA	Poly vinyl alcohol
SEM	Scanning Electron Microscope
SPE	Solid Polymer Electrolyte
Scm^{-1}	Unit of conductivity
T	Temperature
TiO_2	Titanium dioxide
TGA	Thermogravimetric Analysis
V	Volt
XRD	X-Ray Diffraction
ZrO_2	Zirconium dioxide
^{O}C	Degree Celcius

ε	Dielectric constant
(-C-F)	C-F bond
-co-	composite

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

During 20th century most polymer have been used as structural material or as electric insulators. However in the past two years polymers have been used in much advance application such as electron or ion conductor. When the polymer is combined with appropriate and suitable salts solution, material with conductivity was produced which can be utilized to use as an electrolyte electricity device(Hessenbrunch, 2015). The research of polymer electrolyte has been intensively growth for the past few years.

Polymer electrolyte can be classified into four different categories including dry solid polymer electrolyte (SPE), plasticized polymer salt complex, polymer gel electrolyte (GPE) and composite polymer electrolyte (CPE).Conventional polymer salt complex or dry SPE is a mixture ionic salts and polar polymer host such as polyethylene oxide (PEO) and poly propylene oxide (PPO). The casting film is synthesized using solution cast method or sol gel method. Second category which is plasticized polymer salt complex is synthesized by adding liquid plasticizer into dry SPE's and make the mixture possesses liquid and solid like behavior. Meanwhile CPE is technically electrolyte in a composite solid state in electrolyte system.

Fourth category is gel polymer electrolyte (GPE) which is the main concern in this research. GPE is basically synthesized by adding polymer into the combination of ionic liquid solution and salt. The properties of GPE can be classified based on the structure of the polymer network. GPEs are widely used in fuel cells, supercapacitor, electro chromic display device, sensors, and high-low solid state battery (Sekhon, 2003).

Gels are three dimensional (3D) structure of polymer and possess both cohesive properties of solid and the diffusive transport properties of liquid. This kind of property increases the demand of gel application in replacing electrolyte due to its unique stability and conductivity properties.

In the initial stage of this research, gel polymer electrolyte was synthesized with three different polymers to study the effect of polymer on GPE conductivity and stability. Then, a study about the ratio of salt solution and polymer to establish the best ratio for GPE conductivity.

1.2 Problem Statement

Conventional battery has been using the liquid electrolyte as the medium to transfer ion. The usage of liquid electrolyte had been widely used because of the highest conductivity value which meets the characteristic of electricity material requirement. Even though the liquid electrolyte had highest conductivity, it also had a problem in their stability, design temperature and handling procedure (Sekhon, 2003).

In term of stability, liquid electrolyte which used in battery had difficulty in deposition of uneven chemical at the electrode and electrolyte interface after few cycles of operation. This situation can damage the battery and decrease its efficiency of conductivity. Meanwhile the design temperature of liquid electrolyte refer to its operation which can only occur within 0 ^{o}C to 100 ^{o}C. The operation limit made the liquid electrolyte not suitable for the wide electricity application. Besides, liquid electrolyte lead to risk of leakage during operation.

1.3 Objective of the Study

As far as this project is concern, there were several objectives that are needed to be achieved. The objectives are as follows:

1. To investigate the optimum ratio of the ionic liquid to polymer based on conductivity.

2. To study the effect of different polymers which are polyvinyl alcohol (PVA), poly (vinylidene fluoride hexafluoropropylene) (PVdF-HFP) and poly polyvinyl alcohol-co- poly (vinylidene fluoride hexafluoropropylene) (PVA-PVdf-HFP) on conductivity of the gel polymer electrolyte(GPE)

3. To study the properties of different polymer based gel GPE by testing it with Fourier Transform Infrared (FTIR) Spectroscopy, Scanning Electron Microscope (SEM), Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA) and DC conductivity measurement.

1.4 Scope of Study

Gel polymer electrolyte (GPE) was prepared from various polymers. The imitation of this study only covered three different type of polymer which are polyvinyl alcohol (PVA), poly (vinylidene fluoride hexafluoropropylene) (PVdF-HFP) and poly polyvinyl alcohol-co- poly (vinylidene fluoride hexafluoropropylene) (PVA-PVdf-HFP). Ionic liquid 1-ethyl-3-methylimidazolium trifluoro-methanesulfonate (EMITf) was used to mix with magnesium triflate ($Mg\ (Tf)_2$) solution as the electrolytes of GPE. The main technique to produce GPE is the solution casting technique.

This study been conduct on ambient condition and particular temperature during dilute and stir process. Characterization of the chosen GPE was studied using Fourier Transform Infrared (FTIR), Scanning Electron Microscope (SEM), Differential Scanning Calorimetry (DSC), and Thermo gravimetric Analysis (TGA) while its conductivity was tested using AC impedance.

CHAPTER 2

LITERATURE REVIEW

2.1 Polymer

Typically conventional polymer such as plastic, rubbers, and others offer significant resistance to electrical conduction and are either dielectrics or insulator. However, Polymer has been used as the host of the gel polymer electrolyte (GPE) which is the main important part to change the liquid electrolyte to gel phase. Polymer provides mechanical stability to the electrolyte that give effect on the conductivity behavior of GPE as well as the interaction of polymer with salt and solvent (Sekhon, 2003). Unfortunately, several electrolyte will decrease its conductivity as polymer is added while the proton electrolyte will increase its conductivity as the polymer is added.

2.1.1 Poly (vinylidene fluoride) (PVdF) based Polymer

PVdF is a semicrystalline polymer and the electrolytes based on PVdF are expected to have high anodic stabilities due to strong electron withdrawing functional groups (-C-F). It also has high permittivity, relatively low dissipation factor and high dielectric constant (ε=8.4) which assist in high ionization of lithium salts, providing a high concentration of charge carriers. Therefore, it is mainly the PVdF based polymer that gains a wide popularity both as homo- and co-polymer electrolyte for use in lithium-polymer batteries. Hence, the present study was primarily aimed at the development and characterization of a PVdF based gel polymer electrolyte containing EC: PC as plasticizer and the imide lithium salt. The effect of added lithium salt, viz., and imide has been investigated mainly to understand the role of imide salt towards the enhancement of conductivity values in comparison with lithium ionic solution.

Besides, PVdf found to be most suitable due to its outstanding mechanical properties, high chemical resistance, good thermal stability as well as high piezoelectric and pyroelectric coefficients. PVdf is often used by modification with other polymer to overcome the limitation high crystallinity that give poor stability to the loaded liquid electrolyte resulting in low ionic conductivity (Jefrey, 2015). Choe et al. (Cheo *et al.*, 1995) reported that the PVdF based electrolytes plasticized with a solution of LiN $(SO_2CF_3)_2$ in PC had a conductivity of $1.74x10^{-3}$ Scm-1 at 30ºC and has an oxidative stable potential limits between 3.9 and 4.3V vs Li+/Li.

Nicotera et al. (Nicotera *et al.*, 2006) measured the ionic conductivity and the lithium salt diffusion coefficient of PMMA/PVdF based blend electrolytes with EC/PC as plasticizers and lithium perchlorate as salt, which revealed maximum lithium mobility for the composition PMMA 60%-PVdF 40%. Raman spectroscopic study confirmed the change of interaction between the lithium cations and the plasticizer molecules for different PMMA/PVdF ratios. Wang et al. (Wang *et al.*, 2007) prepared the nanocomposite polymer electrolytes comprising of poly (vinylidene fluoride) (PVdF) as a host polymer, lithium perchlorate ($LiClO_4$) as salt and TiO_2 used as a filler by solvent-casting technique. The prepared films were characterized by XRD, DSC and SEM. The conductivity value was found to be of the order of 10^{-3} Scm^{-1} for the sample with 10% TiO_2.

2.1.2 Poly (vinyl alcohol) (PVA) based Polymer

Polyvinyl alcohol have been intensely investigated, because of its interesting properties and applications. PVA is a prominent polymer because of its good solvent holding capability and wide temperature window (Shukla, 2000). Awadhia *et.al*. (Awadhia *et al*, 2007) have reported PVA based proton conducting gel electrolyte with high ionic conductivity. Moreover, because of the nontoxic nature of PVA, they are particularly suitable for biomedical application like artificial tissue.

Polyvinyl alcohol (PVA) also has been used as the polymer host to form the gel electrolyte. PVA is a very attractive material for the gel based electrolyte preparation. PVA has carbon chain back bone with hydroxyl groups attached (Bhargav, 2009). The O-H bonds in the PVA assist the polymer complex formation. PVA also has some good advantages such as high mechanical strength, excellent ionic conductivity, non-toxic, biocompatible, biodegradable and easy to prepare (Rajendran, 2004). PVA has been used to fabricate environmental friendly electrochemical devices such as lithium battery, electrochemical capacitor and supercapacitor.

Aziz et al, 2013 has reported that Gel polymer electrolytes (GPEs) consisting of polyvinyl alcohol (PVA) and double iodide salts have been prepared using ethylene carbonate (EC) and propylene carbonate (PC). Potassium iodide (KI) and tetramethylammonium iodide (TMAI) were used as mixed salts for making the electrolyte ion conducting. The highest room temperature (298K) conductivity of 12.91 $mS \cdot cm^{-1}$ is obtained for the GPE containing the double salts in the ratio of 70 wt.% KI:30 wt.% TMAI, whereas the single salt electrolytes, 100 wt.% KI:0 wt.% TMAI and 0 wt.% KI:100 wt.% TMAI have the conductivities of 12.48 and 3.94 $mS \cdot cm^{-1}$, respectively. Utilizing the highest conducting binary salts electrolyte as the electrolyte medium in dye-sensitized solar cells (DSSCs) produced the highest efficiency (η) of 3.45%.

Besides, Subramanian (2006) has reported that a polymer blend electrolyte based on polyvinyl alcohol (PVA) and polyacrylonitrile (PAN) was prepared by a simple solvent casting technique in different compositions. The ionic conductivity of polymer blend electrolytes was investigated by varying the PAN content in the PVA matrix. The ionic conductivity of polymer blend electrolyte increased with the increase of PAN content. A maximum ionic conductivity of 3.76×10^{-3} S/cm was obtained in electrolyte solution. The effect of ionic conductivity of polymer blend electrolyte was measured by varying the temperature ranging from 298 to 353 K. Linear sweep voltammetry and DC polarization studies were carried out to find out the stability and lithium transference number of the polymer blend electrolyte. Finally, a prototype cell was assembled with graphite as anode, LiMn2O4 as cathode, and polymer blend electrolyte as the electrolyte as well as separator, which showed good compatibility and electrochemical stability up to 4.7 V.

2.1.3 Poly (vinylidene fluoride hexafluoropropylene) (PVdF-HFP)

In recent years, the studies on PVdF-co-HFP based systems are electrochemically stable and indispensable for the electrode properties. The PVdF-co-HFP based electrolyte system shows high electrochemical stability in the range 4V. Fan et al. (Fan *et al*., 2002) studied the thermal, electrical and mechanical properties of EC/PC/$LiClO_4$ based PEO/P (VdF-co-HFP) blends. They concluded that the polymers have good compatibility and PVdF-HFP hinders the crystallinity of PEO. Saika and Kumar (Saika and Kumar, 2004) made systematic studies on the ionic conductivity and transport properties of polymer electrolytes comprising of copolymer PVdF-co-HFP/ PC/ DEC/ $LiClO_4$ and PVdF/ PC/ DEC/ $LiClO_4$ separately. The co-polymer complex showed higher ionic conductivity and transport number compared to PVdF system.

The higher conductivity of the polymer electrolyte based on copolymer was attributed to its higher amorphousity. Wang et.al (Wang *et al*, 2004) reported that the polymer electrolyte composed of poly (methyl methacrylate-co-acrylonitrile-co-lithium methacrylate) (PMAML) and poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-co-HFP) exhibited high ionic conductivity and good electrochemical stability. The ionic conductivity was about 2.6×10^{-3} Scm^{-1} at ambient temperature and the electrochemical window of the polymer electrolyte was about 4.6V. Charge – discharge test results revealed that lithium ion batteries with these gel polymer electrolytes have good electrochemical performance.

2.2 Gel

2.2.1 Gel Polymer Theory

Polymer gel is a chemically cross-linked network of polymer chains and exclude networks formed through interchain physical association. Chemically cross-linked polymer gels can be prepared via the following three methods. First is random crosslinking of existing polymer chains, such as chemical or irradiation vulcanization, then polymerizations of multifunctional monomer systems and end-linking reactions of well-defined precursor polymer chains. The different preparation methods result in different polymer gel structures and hence different properties. Polymerization from monomers involves complex reaction kinetics. End-linking methods can be regarded as special and simplified cases for the random cross-linking method. The mixing term of the gel system is treated by Flory-Huggins polymer solution theory, and the corresponding free energy per unit volume, f_{mix}, is

Equation 1:

$$(Fmix/kT) = (1 - ø) \ln (1 - ø) + X ø (1 - ø) \qquad (1)$$

k is the Boltzmann constant, T is the absolute temperature, ø is the polymer volume fraction of the swelling gel, and (1 – ø) is the solvent volume fraction. The parameter ø is the Flory polymer-solvent interaction parameter (Hueang *et al*, 2002). In the right side of equation 1, the first term represents the translational entropy of solvent molecules and the second term is the interaction between polymer chains and solvent molecules. It has been known that equation 1 underestimates the monomer correlation of polymer chains and hence overestimates the intrachain monomer repulsion in good solvent. The polymers are treated in Flory's theory as unconnected monomers, and the conformational entropy of polymer chains and their interactions with the environment are separated. However, it is their ability to change conformations with the environmental conditions that give polymers fascinating properties and numerous applications. Therefore, it is very desirable to have a polymer solution theory that can correlate the chain conformations with the thermodynamic properties of the system.

For gel made from solution, there are two parameters characterizing the preparation conditions: the polymer volume fraction $ø_0$ and the polymer/solvent interaction parameter X_0. Most of the discussions in the literature focus on the effect of $ø_0$, but here we want to emphasize that the polymer/solvent interaction is also important for the relaxed state of gels made from solutions. The relaxed state of gels is defined as the state where the mesh chains do not exert elastic forces on the crosslinks, and it is generally accepted that the nascent gel beyond the gelation point is in the relaxed state. Gels made under different conditions have their unique relaxed states and hence have different molecular structures.

2.2.2 Gel Polymer Electrolyte

Gel polymer electrolyte (GPE) has become an intention or interest by many researchers for its future development. This is because, GPE has a very suitable characteristic in electricity application. Moreover, GPE frequently used as an excellent substitute to replace liquid electrolytes for battery and supercapacitor applications(Pandey and Hashmi, 2009). There are also several reports about investigation in solid-state rechargeable magnesium batteries using polymer gel electrolyte. In 2000, Kumar had do research by test the conductivity of gel polymer electrolyte based magnesium salt and the conductivity. The result GPE shows an excellent result(Kumar and Munichandraiah, 2000a).

2.3 Ionic solution

Ionic liquid plays an important role as conductivity element in the formation of GPE. Selecting the suitable ionic liquid will give different effects in electricity conductivity and properties of the GPE. In recent year, a room temperature ionic liquid has been proposed as non-volatile polar media in variety of electrochemical(Pandey and Hashmi, 2009). The room temperature of ionic liquid contains many suitable properties such as non-volatile, non-flammable, excellent thermal stability, and higher ionic conductivity.

There was a report in 2009 made by Pandey, the 1-ethyl-3-methylimizodilium triflouro-methanesulfonate (EMItf) was chosen as the main ionic liquid which was used as part of the material in the synthesis the GPE (Pandey and Hashmi, 2009). Based on the report, The electrical conductivity of the ionic liquid EMITf and $Mg(Tf)_2$/EMITf liquid electrolyte and gel polymer electrolyte films was evaluated by means of an AC impedance technique using a LCR Hi-Tester (HIOKI-3522-50, Japan) over the frequency range 100 kHz to 1Hz with a signal level of 10mV. The conductivity of the ionic liquid and the liquid electrolyte was evaluated using a special two-electrode conductivity cell. Conductivity measurements of gel polymer films were performed by sandwiching the films between two stainless-steel foils. The electrochemical stability of the electrolyte films was evaluated by means of linear sweep voltammetry using stainless steel as the working electrode and an Mg disc as both the counter and the reference electrode.

2.4 Salt solution

Salt solution is a one of the materials to be used in the synthesis of GPE. In this research the salt solution role in GPE is solution that contains ion which is acquired capacity to conduct electricity. Magnesium based solution had be choose due to its high stability and abundant in resource which meet the requirement of economic value(Kumar and Munichandraiah, 2000a). The stability of the magnesium can be defined as the magnesium metal can be easily handled safely under oxygen condition and humid atmosphere(Kumar and Munichandraiah, 2000b).

Besides that, magnesium is also is an attractive anode of battery since it has high specific energy because it has low electrochemical equivalent as low as 12.5 g equiv.$^{-1}$(Pandey and Hashmi, 2009). This several characteristics of the magnesium lead the chosen of magnesium based solution as the salt solution in gel polymer electrolyte is a good decision. Most of the characteristics give the good effects in term electricity and safety aspect which are basic principle and interest in creating or designing material for future development.

2.5 Magnesium batteries

One of the main challenges that current rechargeable battery technologies face is their inability to maintain energy and power densities sufficient to meet those demanded by their applications. In fact, the gap between the energy storage needs and what state of the art systems are capable of providing is increasing. This ever increasing gap has been a persistent force that drove many of the innovations made over the last 40 years (Linden, 2004). For example, lithium batteries using lithium metal anodes have attracted attention as a candidate to fill up the said gap. However, this system suffers from the basic property of lithium to form needle-like lithium crystals, known as dendrites, when it is plated.

These grow with subsequent plating/stripping cycles, resulting in an internal short circuit and fire hazards (Gofer, 2013). While effective countermeasures are still being discussed, the birth of the first commercial Li-ion battery in the early 1990s was catalyzed by the need to overcome these challenges. This resulted in a decline in further technical progresses and commercialization of what was referred to as the "ultimate lithium metal anode". If we wish to move forward towards achieving an ultimate energy density goal, technologies beyond Li-ion batteries would be needed. Fortunately, in recent years, such desire has led to an increased interest in other chemistries that employ metals poised to provide higher energy densities without compromising the safety of the battery.

For example metals such as magnesium and aluminum were proposed. Magnesium metal has been attracting an increased attention as it possesses higher volumetric capacities than lithium metal, i.e., 3832 mAh cm^{-3} vs 2061 mAh cm^{-3} for lithium. It may also provide an opportunity for battery cost reductions due to its natural abundance in the earth crust fifth most abundant element (Linden, 2011). More importantly, despite the fact that magnesium metal is not competitive with lithium metal on both specific capacity (2205 mAh g^{-1} vs 3862 mAh g^{-1} for lithium), the electrochemical processes related to its reversible plating/stripping have demonstrated the absence of dendrites formation which has thus far alleviated safety concerns related to employing it as a negative electrode in batteries.

CHAPTER 3

METHODOLOGY

3.1 Research Outline

Gel Polymer Electrolyte (GPE) are composed of a polymer, an organic solvent and an electrolyte salt. The polymer can be used as a matrix that keeps the electrolyte solution in i.e. that prevents it from escaping or leaking. The solvent is used to dissolve the polymer and the salt as it dissociates the salt components and frees the conducting ions. The salt, which is a magnesium-based one, provides the free ions necessary for conduction after it has been broken into parts by the polar solvent. Figure 3.1 shows the research outline on study the best ratio of polymer to electrolyte.

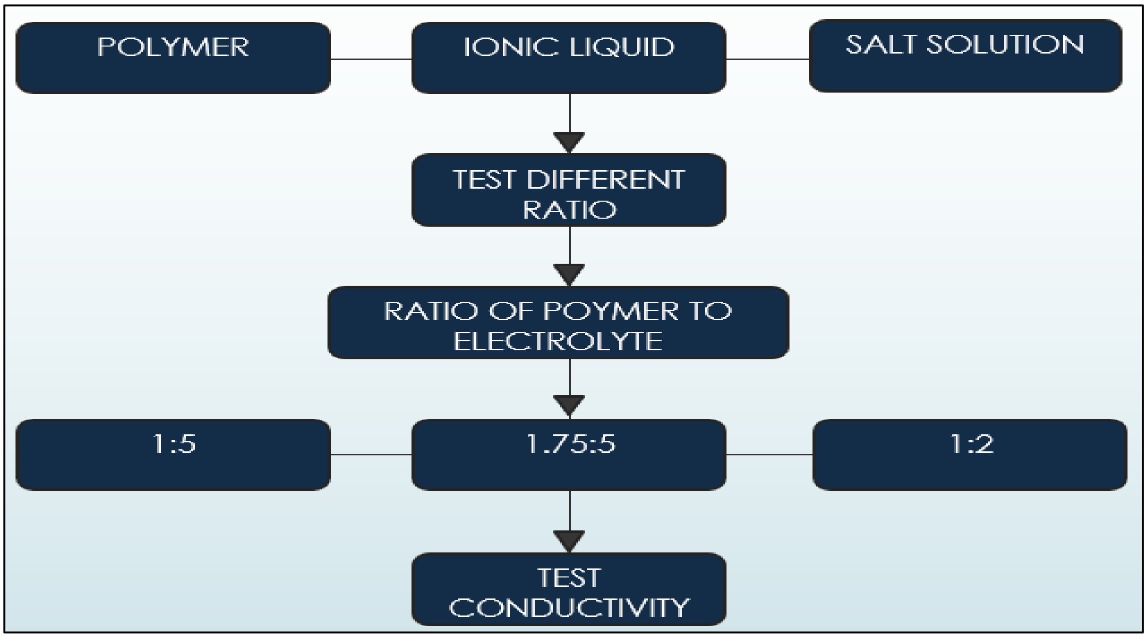

Figure 3.1 Research Outline of Ratio Testing

Figure 3.2 shows the research outline on study effect of different polymer to the conductivity of GPE electrolyte.

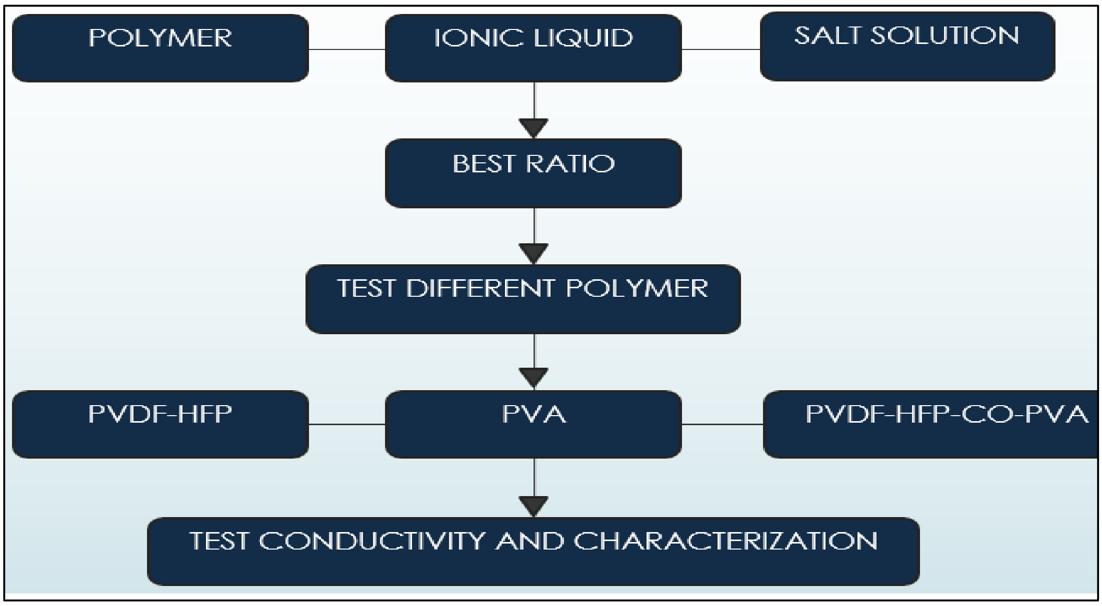

Figure 3.1 Research Outline of Different Polymer Testing

3.2 Material

Table 3.1 shows the material and chemical that were used in synthesized gel polymer electrolyte (GPE). As mentioned in previous section, there are three different polymer were used as the host polymer for GPE. One type of ionic liquid and salt were used as the electrolyte solution for GPE. In addition normal concentration of acetone solution was used as solvent.

Table 3.1 List of Material

Chemical	Function
Polyvinyl alcohol (PVA), Poly (vinylidene fluoride hexafluoropropylene) (PVdF-HFP) and Poly polyvinyl alcohol-co-Poly (vinylidene fluoride hexafluoropropylene) (PVA-PVdf-HFP)	Polymer
1-ethyl-3-methylimidazolium trifluoro-methanesulfonate (EMITf)	Ionic Liquid
Magnesium triflate	Salt solution
Acetone solution	Solvent

3.3 Preparation Technique

3.3.1 Preparation of the Gel Polymer Electrolyte with Different Ratio

1. 0.3 g of magnesium triflate was prepared in a beaker.
2. 3 ml of 1-ethyl-3-methylimidazolium trifluoro-methanesulfonate (EMITf) ionic liquid was prepared in the beaker.
3. Magnesium salt was dissolved into the ionic liquid solution and stir it for 20 minutes at 60 °C.
4. 0.6 g of polymer was prepared and dissolved in 3 ml of acetone solution with magnetic stirrer for 20 minutes at 60 °C.
5. Magnesium salt and ionic liquid with the polymer mix with magnetic stirrer for 1-2 hour at 40 °C.
6. Step 1-5 is repeated with different ratio of polymer: ionic solution and salt for 1:5, 1.75:5, and 1:2.

3.3.2 Preparation of the Gel Polymer Electrolyte with Different Polymer

1. 0.3 g of magnesium triflate was prepared in a beaker.
2. 3 ml of 1-ethyl-3-methylimidazolium trifluoro-methanesulfonate (EMITf) ionic liquid was prepared in the beaker.
3. Magnesium salt was dissolved into the ionic liquid solution and stir it for 20 minutes at 60 °C.
4. 0.6 g of polymer was prepared and dissolved in 3 ml of acetone solution with magnetic stirrer for 20 minutes at 60 °C.
5. Magnesium salt and ionic liquid with the polymer mix with best ratio by magnetic stirrer for 1-2 hour at 40 °C.
6. The conductivity of each specimen was tested using AC impedence.
7. Step 1 to 6 is repeated with Polyvinyl alcohol (PVA), Poly (vinylidene fluoride hexafluoropropylene) (PVdF-HFP) and Poly polyvinyl alcohol-co-Poly (vinylidene fluoride hexafluoropropylene) (PVA-PVdf-HFP).

3.4 Testing

There are five testing in order to study the conductivity and thermal properties of gel polymer electrolyte (GPE).

1. Fourier Transform Infrared (FTIR) Spectroscopy

   Fourier Transform Infrared (FTIR) spectrum was recorded on PerkinElmer Spectrum Version 10.4.4 between 4000 and 450 cm^{-1}.

2. Scanning Electron Microscope (SEM)

   SEM (model: Pro X, brand: Phenom) was used to evaluate the surface morphology of the sample material and microstructure of GPE. The parameter used are 10000 x magnification and 10 miro meter size picture.

3. Differential Scanning Calorimetry (DSC)

   Differential Scanning Calorimetry testing was done using equipment DSC Q2000 V24.11 Build 124. The parameter used are -50°C as starting temperature, 250°C as end temperature and 20°C per minute as heating rate.

4. Thermogravimetric Analysis (TGA)

   To measure the change of mass of the resulted hydrogel associated with the change of temperature. The measurement done by TGA model: PYRIS 1, brand: Perkin Elmer The parameter used are 30 °C as starting temperature , 800 °C as end temperature and 100°C per minute as heating rate.

5. DC conductivity measurement

   To measure the conductivity of the resulted GPE. The GPE will be mounted between steel electrodes inside a sample holder. Conductivity is measured by using Ohm’s law. The conductivity is calculated using equation (2).

$$Conductivity(Scm^{-1} = \frac{Thickness(cm)}{Area(cm^{2})\ x\ Bulk\ Reesistence\ (\Omega)} \qquad (2)$$

3.5 Solution Cast Technique

Figure 3.3 shows the procedure of cast solution technique. This is the crucial part in order to synthesis the gel polymer electrolyte.

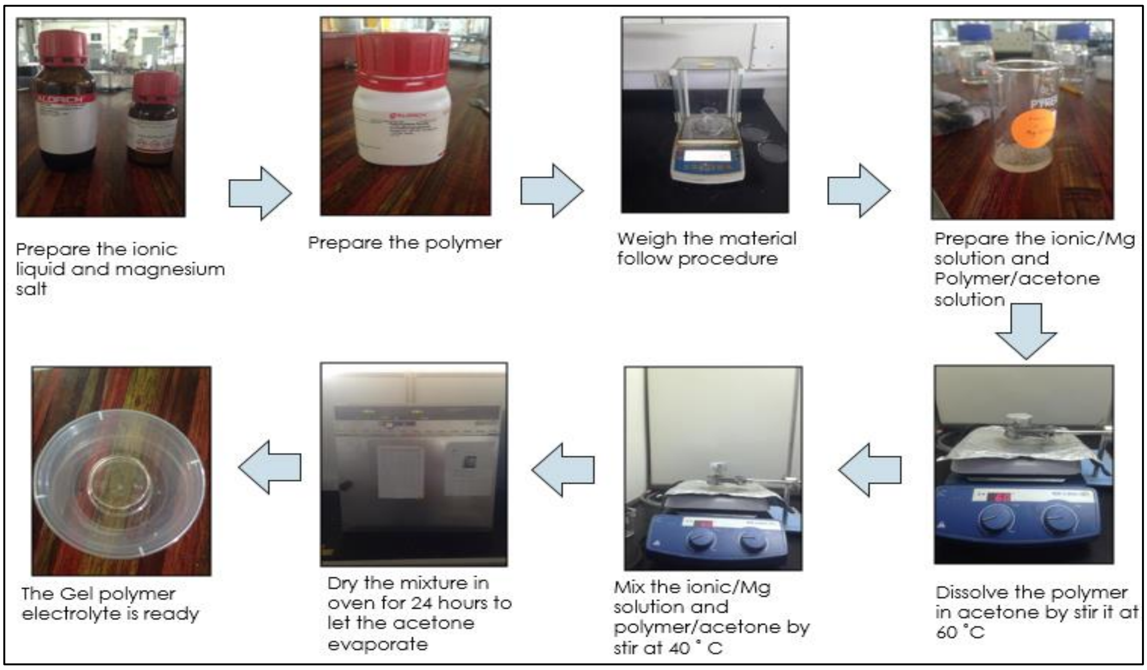

Figure 2.3 Cast Solution Procedure

3.6 Gant Chart and Key Milestone

Table 3.2 shows the gantt chart and activity for final year project I. The activities was covered from week 1 until week 14 for semester 1.

Table 3.2 Gantt Chart FYP I

No	Week/activity	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Title		🟦												
2	Preliminary Research Work and Literature Review		🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦
3	Submission of extended proposal (first draft)						🟦								
4	Submission of extended proposal (final draft)							🟥							
5	Proposal Defense								🟥	🟥					
6	Detailed Literature Review								🟦	🟦	🟦	🟦	🟦	🟦	
7	Preparation of Interim Report								🟦	🟦	🟦	🟦	🟦	🟦	
8	Submission of the draft of Interim Report													🟥	
9	Submission of the draft of Interim Report														🟥

🟦 **Gant chart**

🟥 **Key milestone**

Table 3.3 shows the gant chart and activity for final year project II. The activities was covered from week 1 until week 14 for semester 2.

Table 3.3 Gantt Chart FYP II

No	**Week/activity**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
1	Lab Experiment and Sample Preparation	🟦	🟦	🟦	🟦	🟦	🟦								
2	Submission of Progress Report							🟥							
3	Testing and Recording Result							🟦	🟦	🟦					
4	Pre-SEDEX									🟥	🟥				
5	Submission of Final Report (Draft)										🟥				
6	Submission of Technical Paper (Soft)											🟦			
7	Finalization of Project, Mock VIVA											🟦	🟦		
8	VIVA													🟥	
9	Submission of Dissertation (HARD)														🟥

🟦 **Gant chart**

🟥 **Key milestone**

CHAPTER 4

RESULT AND DISSCUSSION

4.1 Conductivity

The conductivity of the samples is calculated using the formula in equation below. Using the resistance recorded, the conductivity of the gel polymer electrolyte is calculated. The conductivity is calculated using equation (3)

$$Conductivity(Scm^{-1} = \frac{Thickness(cm)}{Area(cm^2)\ x\ Bulk\ Reesistence\ (\Omega)} \qquad (3)$$

4.1.1 Sample of GPE with different ratio

Figure 4.1 illustrates the picture of gel polymer electrolyte (GPE) with different ratio. There are three ratio of polymer to electrolyte GPE (a) 1:5, (b) 1.75:5, (c) 1:2

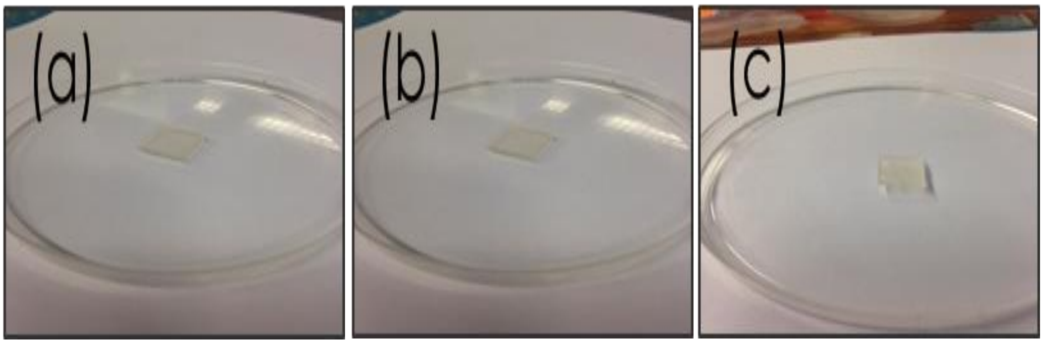

Figure 4.1 Picture of Sample Different Ratio GPE

4.1.2 Conductivity on different ratio of GPE

Table 4.1 illustrated the thickness, area, bulk resistance and conductivity for three different ratio of GPE. The thickness and area is same since it is constant for every sample of GPE.

Table 4.1 Conductivity of Different ratio of GPE

Ratio (Polymer: Electrolyte)	**Thickness of GPE (cm)**	**Area (cm2)**	**Bulk Resistance (Ω)**	**Conductivity (Scm-1)**
PVdf-HFP GPE (1:2)	0.1	1	500	0.0002
PVdf-HFP GPE (1.75:5)	0.1	1	380	0.000263158
PVdf-HFP GPE (1:5)	0.1	1	430	0.000232558

Figure 4.2 illustrated the graph for ratio of (a) 1:5, (b) 1.75:5 and (c) 1:2 of polymer to electrolyte. For this graph, GPE with polymer of PVdf-HFP is used.

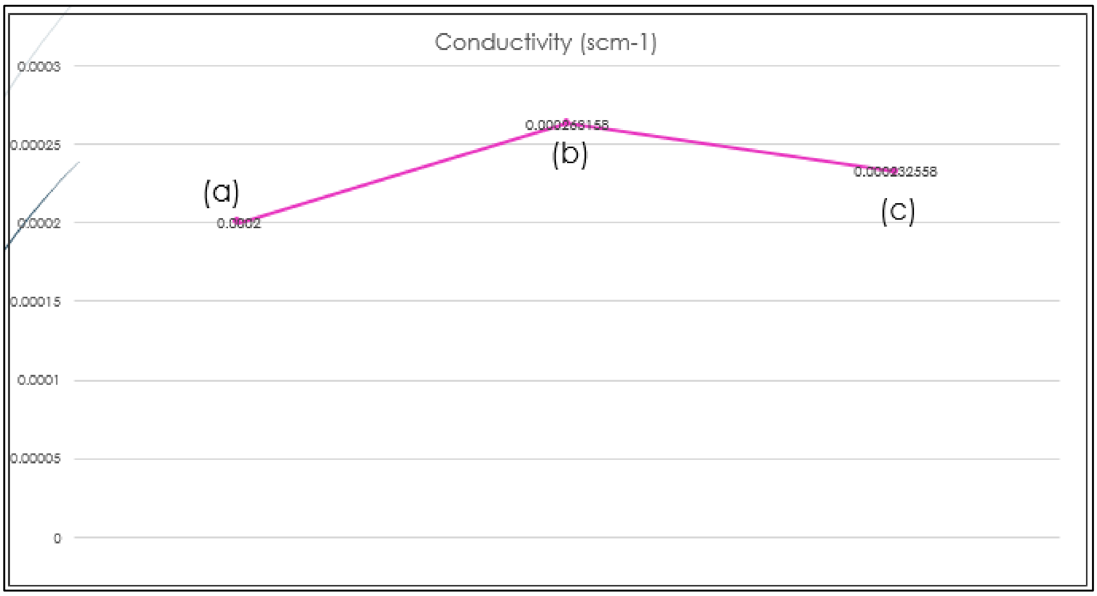

Figure 4.2 Conductivity Graph of Different Ratio GPE

Based on the graph GPE with ratio (1:2) shows in figure 4.2(a) had the lowest conductivity value which is 0.0002 Scm^{-1}. This happen due to excessive amount of polymer which lower the entrapment and movement of the electrolyte. Besides, the high amount of polymer also lead to the rigidness of the structure of the gel polymer electrolyte. While the GPE with ratio (1.75:5) shows in figure 4.2(b) ratio had the optimum value of conductivity which is 0.00026 $Scm^{-}1$. This situation was reasoned by the optimum amount of host polymer which suits the space for entrapment of electrolyte. The optimum amount of host polymer resulted a good space for movement of ion within the gel polymer electrolyte.

Besides, GPE with ratio (1:5) shows in figure 4.2(c) had the second lowest value of conductivity which is 0.00023 Scm^{-1}. This situation was happen due to lack amount of polymer which lead to the excessive loss of ionic liquid during mixing. The host polymer is too low until the polymer cannot create enough strength to hold all the electrolyte.

4.1.3 Sample of GPE with different polymer

Figure 4.3 illustrates the picture of gel polymer electrolyte (GPE) with different polymer. There are three different polymer based GPE (a) PVdf-HFP based GPE, (b) PVA based GPE, (c) PVdf-HFP-co-PVA GPE.

Figure 4.3 Picture of GPE with Different Polymer

4.1.4 Conductivity testing on different polymer:

Table 4.2 illustrated the thickness, area, bulk resistance and conductivity for three different polymer of GPE. The thickness and area is same since it is constant for every sample of GPE.

Table 2.2 Conductivity of Different Polymer of GPE

Type of Polymer	**Thickness of GPE (cm)**	**Area (cm^2)**	**Bulk Resistance (Ω)**	**Conductivity (Scm^{-1})**
PVA GPE	0.1	1	480	0.000208333
PVdf-HFP GPE ionic only	0.1	1	485	0.000206186
PVdf-HFP GPE	0.1	1	380	0.000263158
PVA-co-PVdf-HFP GPE	0.1	1	320	0.0003125

Figure 4.4 illustrates the graph conductivity of (GPE) with different polymer. There are three different polymer based GPE (a) PVdf-HFP based GPE, (b) PVdf-HFP with ionic only, (c) PVA based GPE, (d) PVdf-HFP-co-PVA GPE.

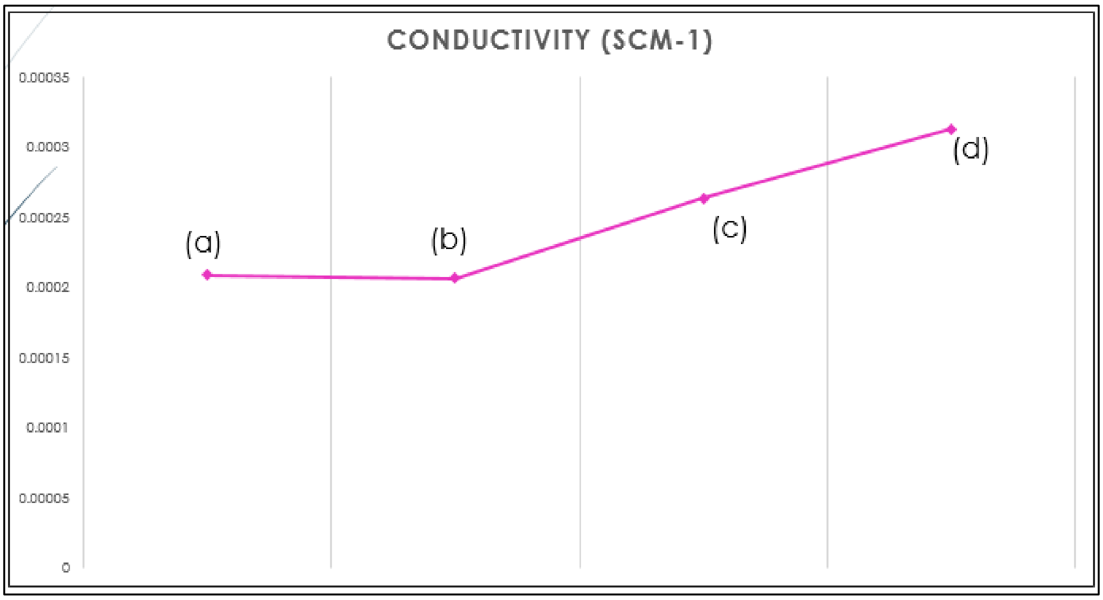

Figure 4.4 Graph of Conductivity on Different Polymer of GPE

Based on the graph, GPE of PVdf-HFP without magnesium show in figure 4.4(b) had the lowest conductivity value which is 0.000208 Scm^{-1}. While the GPE of PVA-co-PVdf-HFP show in figure 4.4(d) had the optimum value of conductivity which is 0.0003125 Scm^{-1} and the GPE of PVA show in figure 4.4(a) based had the second lowest value of conductivity which is 0.000206 Scm^{-1}. Lastly, GPE with PVdf-HFP based GPE had the second highest value of conductivity at 0.00026 Scm^{-1}

GPE of PVA based had the lowest value because of PVA is not have enough characteristic in holding the electrolyte and creating enough pore size for entrapment of electrolyte inside the host polymer. Some of electrolyte had been escape since the PVA cannot allocate all the electrolyte inside it network.

While GPE of PVA-co-PVdf-HFP based seem to have the optimum value of polymer to maintain the amount of electrolyte in the GPE and to let the ion movement during conductivity. In addition the the capability of the PVdf-HFP in allocate all the electrolyte together and the conductivity of the PVA had given the GPE the highest value of conductivity.

4.2 Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopic studies were conducted to investigate the ion and ionic liquid interaction with the host polymer in the gel electrolyte system at the microscopic level and possible conformational changes in the host polymer due to Mg salt/ionic liquid entrapment. Figure 4.5 illustrates the FTIR result for (a) GPE with EMITF only, (b) GPE with EMITf/Mg, (c) PVdf-HFP Film, (d) EMITF ionic liquid.

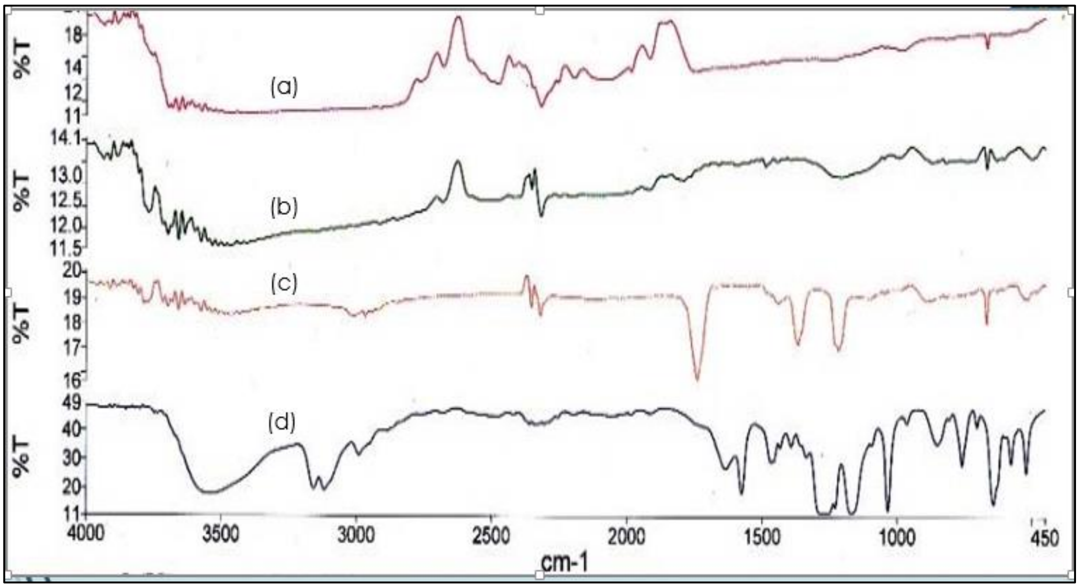

Figure 4.5 FTIR result for four component

Based on the graph there are conformational changes in the semi-crystalline host polymer PVdF-HFP due to the addition of ionic liquid or ionic liquid/Mg-salt solution have been monitored. Table 4.3 illustrate the wavelength rading for PVdf-HFP reading as shown in Figure 4.5(b).

Table 4.3 PVdf-HFP GPE Wavelength Reading

Group	Wavelength Reference (cm^{-1})	Wavelength Sample (cm^{-1})
C = O	1670- 1820 (weak)	1914
C - O	1000 - 1300	1202
$-CF_2$	400 - 700	665
C-H	1350 - 1490	1486
C – O - C	850 - 1200	867
$- CH_3$	2400 - 3000	2686

The vibrational bands observed at 2686 cm^{-1} are ascribed to $–CH_3$ asymmetric and symmetric stretching vibrations of PVdF-HFP respectively. The weak absorbance at 1914 cm^{-1} represents the C=O stretching vibration mode of PVdF-HFP polymer. The existence of C-O band has been confirmed by the strong absorbance band at around 1202 cm^{-1}. The band at 867 cm^{-1} is assigned to C-O-C symmetric stretching mode of vibration. The peak at 1486 cm^{-1} is ascribed to CH bending vibration. The C-H wagging mode of vibration has been confirmed by the presence of a band at 1486 cm^{-1}.The vibrational peaks at 665 cm^{-1} are assigned to bending and wagging vibrations of $–CF_2$ of PVdF-HFP polymer respectively.

A comparison indicates that the band at 976 cm^{-1} as shown in figue 4.5(a) disappears due to the addition of ionic liquid or ionic liquid/Mg-salt solution. Further, the band at 878cm^{-1} (assigned to the amorphous phase), which appears as a symmetrical peak, becomes appreciably broad and appears to be the sum of two peaks. These observations indicate the occurrence of substantial conformational changes in the crystalline texture of the host polymer PVdF-HFP due to interaction with the ionic liquid/Mg-salt in the gel polymer electrolyte.

The possible effects on the ionic liquid and Mg-salt solution in the ionic liquid due to entrapment in polymer host have also been examined. Bands associated with the triflate anion (CF_3SO_3) are shown in figure 4.5(b) are observed at 640 and 1157cm^{-1}. The bands are indicated by open circles in the spectra.

4.3 Scanning Electron Microscope (SEM)

Figure 4.6 illustrates the picture of SEM result for PVA based GPE, while figure 4.7 illustrates the picture of SEM result for PVdf-HFP based GPE and figure 4.8 illustrates picture of SEM result for PVA-co-PVdf-HFP based GPE.

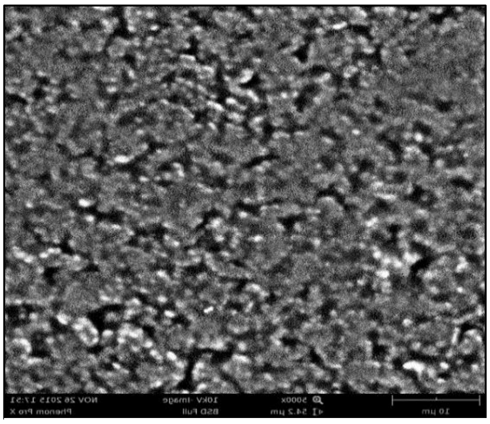

Figure 4.6 PVA based GPE

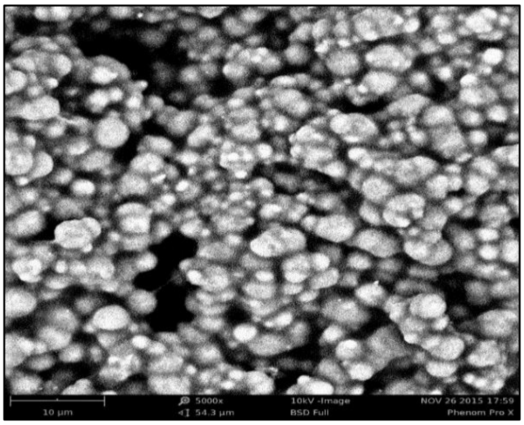

Figure 4.7 PVdf-HFP based GPE

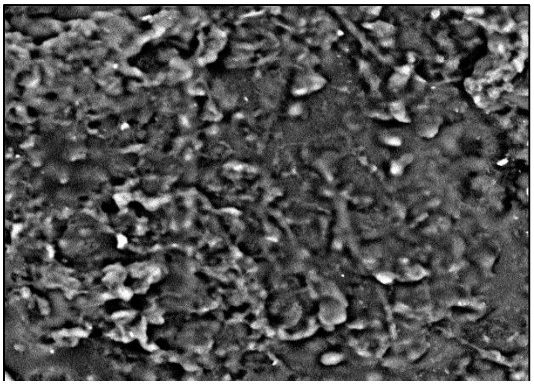

Figure 4.8 PVA-co-PVdf-HFP based GPE

The SEM testing is test under the condition of 25 ^{o}C and magnification of 10000x. The gel polymer electrolyte based PVdf-HFP-co-PVA based GPE shown in figure 4.8 clearly shows smooth and uniform surface morphology of the PVdf-HFP-co-PVA GPE. This smooth morphology confirms the complete amorphous nature of PVdf-HFP-co-PVA polymer and complete dissolution of the Magnesium salt. Large uniformly dispersed pores in the polymer microstructure lead to the retention of ionic liquid or liquid electrolyte and thereby better connectivity through the polymer that gives rise to high ionic conductivity.

The gel polymer electrolyte based PVdf-HFP shown in figure 4.7 also clearly shows smooth and uniform surface morphology of the PVdf-HFP GPE. This smooth morphology confirms the complete amorphous nature of PVdf-HFP-co-PVA polymer and complete dissolution of the Magnesium salt. Moderate size uniformly dispersed pores in the polymer microstructure lead to the moderate retention of ionic liquid or liquid electrolyte and thereby resulting moderate conductivity.

The gel polymer electrolyte based PVA shown in figure 4.6 also clearly shows smooth and uniform surface morphology of the PVA GPE. This smooth morphology confirms the complete amorphous nature of PVdf-HFP-co-PVA polymer and complete dissolution of the Magnesium salt. Small size uniformly dispersed pores in the polymer microstructure lead to the less retention of ionic liquid or liquid electrolyte and thereby resulting low conductivity.

4.4` Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) is a widely used thermal analysis technique in various research areas. The thermal response of a material, heat effects associated with phase transitions and chemical reactions as a function of temperature are analyzed in DSC. The difference in heat needed to increase the sample temperature, compared with a reference, is measured as a function of temperature. Figure 4.9 illustrates the DSC graph for gel polymer electrolyte based PVA-co-PVdf-HFP, figure 4.10 ilustrates the DSC graph for GPE based PVA and figure 4.11 illusrates the DSC graph for GPE based PVdf-HFP.

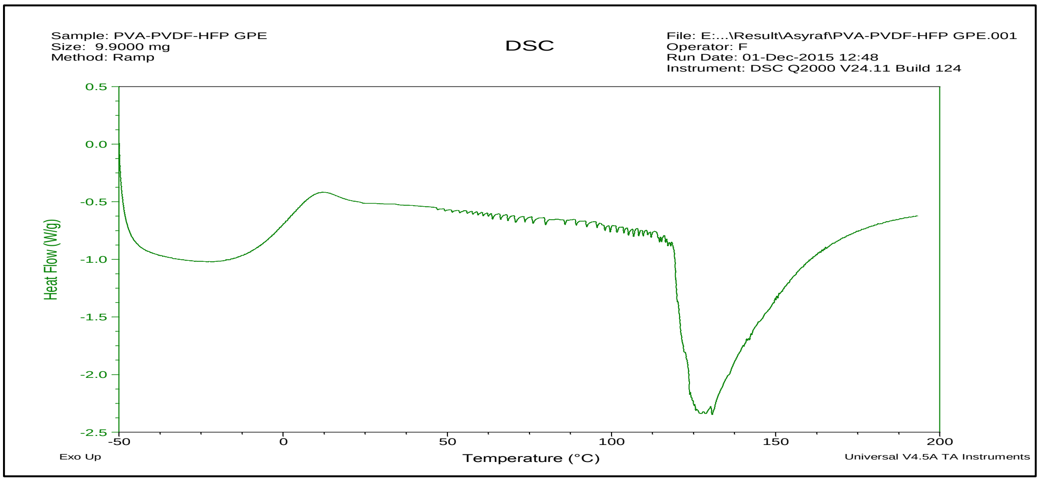

Figure 39 PVA-co-PVdf-HFP (DSC)

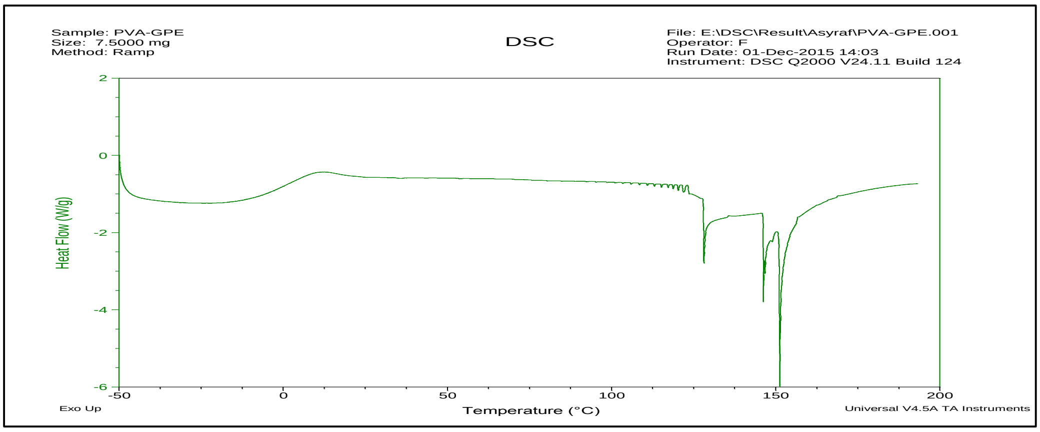

Figure 4.10 PVA based GPE (DSC)

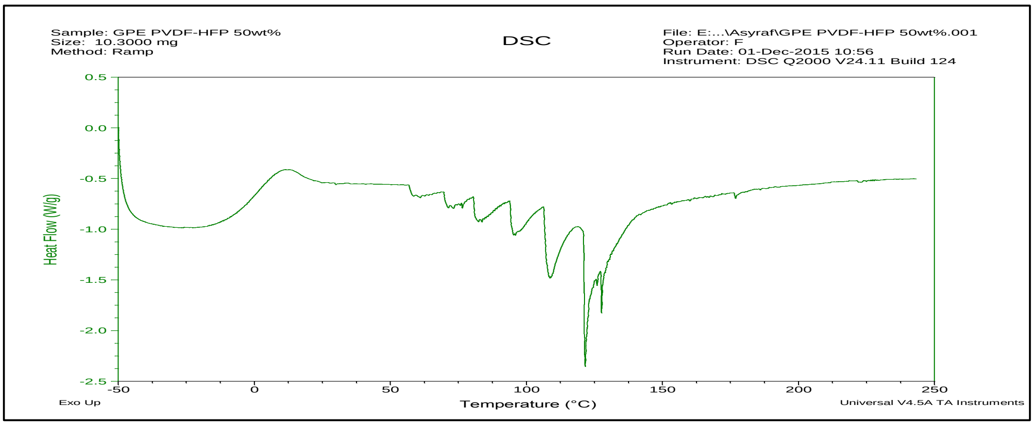

Figure 4.11 PVdf-HFP GPE (DSC)

DSC curves of the PVdf-HFP-co-PVA based GPE are presented in figure 4.9. The endothermic peak at 135 ◦C corresponds to the melting of the GPE. The endothermic peak around −40 ◦C observed in PVdf-HFP-co-PVA Film gel polymer electrolyte film is due to melting of the ionic liquid, EMITf. The electrolyte film remains stable in the gel phase over a substantially wide temperature range from −40 to 135 ◦C, which is advantageous for their potential applications in electrochemical devices and show good stability compared to others.

DSC curves of the PVA based GPE are presented in figure 4.10. The endothermic peak at 150 ◦C corresponds to the melting of the GPE. The endothermic peak around −40 ◦C observed in PVA Film gel polymer electrolyte film is due to melting of the ionic liquid, EMITf. The electrolyte film remains stable in the gel phase over a substantially wide temperature range from −40 to 150 ◦C, which is advantageous for their potential applications in electrochemical devices and show highest stability compared to others.

DSC curves of the PVdf-HFP based GPE are presented in figure 4.11. The endothermic peak at 128 ◦C corresponds to the melting of the GPE. The endothermic peak around −30 ◦C observed in PVdf-HFP 50wt% Film gel polymer electrolyte film is due to melting of the ionic liquid, EMITf. The electrolyte film remains stable in the gel phase over a substantially wide temperature range from −30 to 128 ◦C, which is advantageous for their potential applications in electrochemical devices and show moderate stability compared to others.

4.5 Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) function is to measure the change of mass of the resulted by gel polymer electrolyte associated with the change of temperature. Figure 4.12 illustrates the TGA graph for PVA-co-PVdf-HFP based GPE, Figure 4.13 illustrates the TGA graph for PVA based GPE and figure 4.14 illustrates the THA graph for PVdf-HFP based GPE.

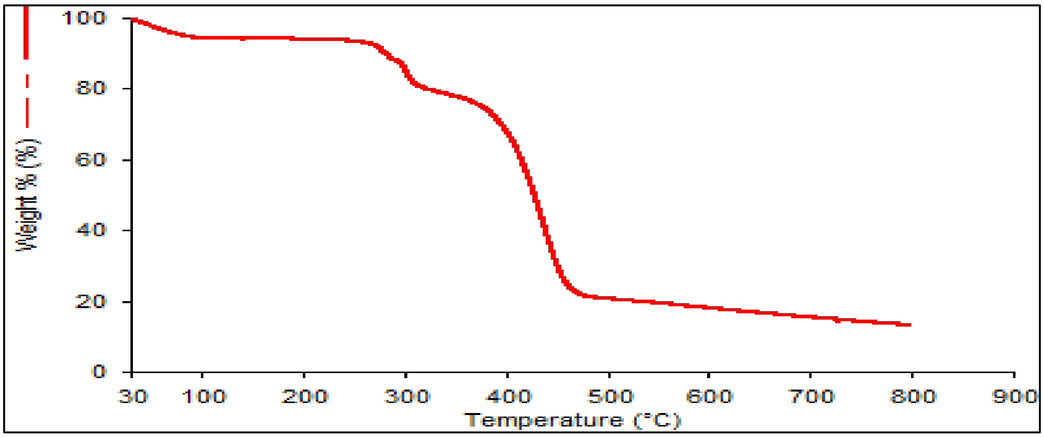

Figure 4.12 PVA-co-PVdf-HFP based GPE (TGA)

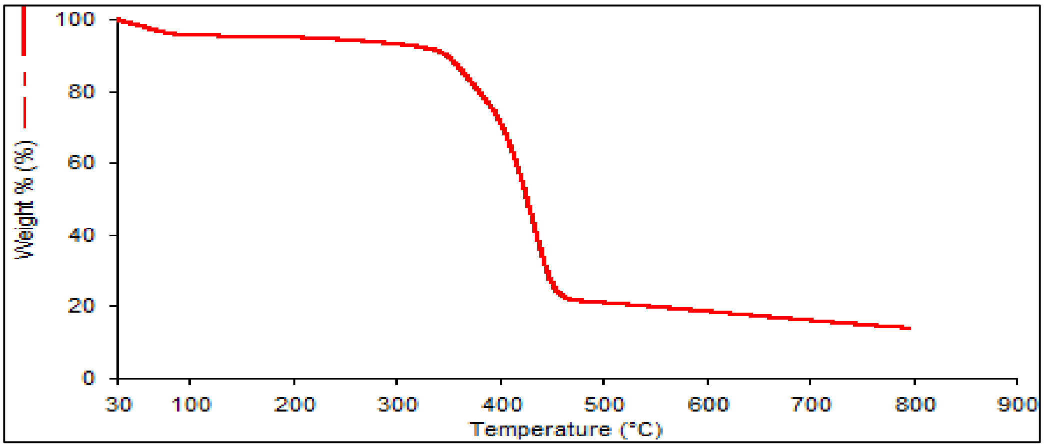

Figure 4.13 PVA based GPE (TGA)

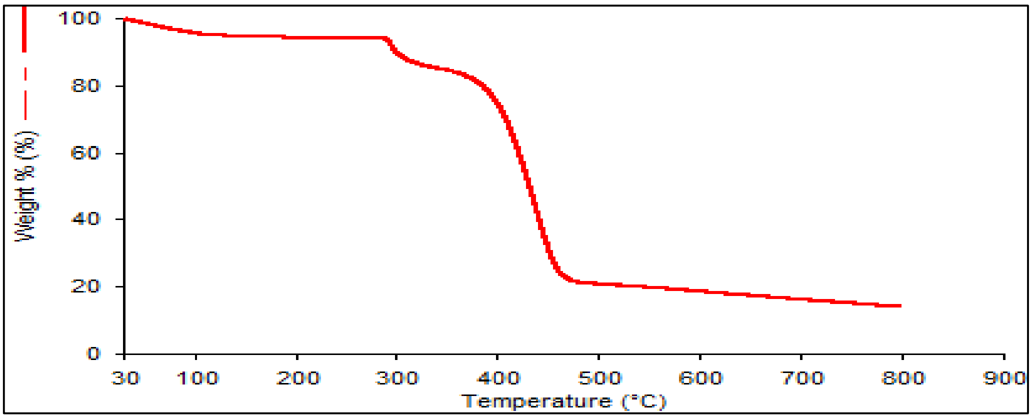

Figure 4.14 PVdf-HFP based GPE (TGA)

Almost no loss in weight is observed for the PVdf-HFP-co-PVA GPE as shown in figure 4.12 up to 260 ◦C. This indicates that no component is volatile in the blend film. The PVdf-HFP-co-PVA GPE Film still meet the requirement of weight loss of 20 percent at 325 ◦C.This is most likely due to moisture absorption in the film given that the Mg-salts are quite sensitive to moisture. The non-volatility is of special importance for the potential application of the gel polymer films as electrolyte/separator components in Mg batteries. However the stability test by the TGA still have meet the minimum requirement for battery application.

Almost no loss in weight is observed in figure 4.13 for the PVA GPE Film up to 300 ◦C. This indicates that no component is volatile in the blend film. The PVA GPE Film still meet the requirement of weight loss of 20 percent at 420 ◦C. This shows that the Mg salt have interrupt the structure of GPE and slightly decrease the durability of the GPE.

Almost no loss in weight is observed in figure 4.14 for the PVdf-HFP GPE Film up to 280 ◦C. This indicates that no component is volatile in the blend film. The PVdf-HFP GPE Film still meet the requirement of weight loss of 20 percent at 380 ◦C .This is most likely due to moisture absorption in the film given that the Mg-salts are quite sensitive to moisture. The non-volatility is of special importance for the potential application of the gel polymer films as electrolyte/separator components in Mg batteries.

Based on three graph on TGA analysis, PVA-co-PVdf-HFP can be conclude as most suitable polymer to be used as polymer for GPE because of its thermal stability with enhance value of conductivity lead for good operating condition for battery usage.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In a nutshell, based on three different ratio which are 1:2, 1.75:5 and 1:5, ratio of 1.75:5(polymer to electrolyte) had shown the highest value of conductivity which is 2.6 x 10^{-4} Scm^{-1}.Futhermore, based on three different polymers based GPE, PVA-co-PVdf-HFP had shown the highest conductivity at 3.125 x 10^{-4} Scm^{-1}. This had conclude that ratio of 1.75:5 with PVA-co-PVdf-HFP gives the highest conductivity value among the other ratio and polymer based. While in study the properties of different polymer based GPE, PVA based GPE had shown highest value in term of thermal stability which represent by DSC and TGA analysis. However, PVA-co-PVdf-HFP still meet the minimum requirement in term of thermal stability to replace the electrolyte in battery application. Therefore, PVA-co-PVdf-HFP is the best host polymer to be used in Gel polymer electrolyte synthesis.

5.2 Recommendation

For future recommendation it is suggested to futher on the nanosized particle of magnesium oxide. Obviously, nanosized particle improved the mechanical as well as he electrochemical and transport properties of polymer gel electrolyte. There it will a great opportunity to perform a deep research on the nanosized particle of magnesium oxide since the nanotechnology is a demand for time being.

In addition, further research on optimizing the ratio of PVA-PVdf-HFP ultill optimum ratio of particular polymer based GPE is obtained. The optimum ratio must represent by the highest conductivity among other ratio.

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APPENDICES

Calculation of Molarity for Magnesium Triflate:

$$Molarity = \frac{mole}{volume}$$

Molarity for Magnesium Triflate is 0.3 M, therefore:

$$0.3\ M = \frac{mole}{3\ ml}\ , Mole = 0.9\ mol$$

$$Mole = \frac{Mass}{Molar\ Mass}\ , Molar\ Mass\ of\ 322.44\ g/mol$$

$$0.9\ mol = \frac{Mass}{322.44\ g/mol}\ , Mass = 0.3\ g$$

Calculation of weight percent polymer in acetone Solution:

$$Polymer\ wt\% = \frac{weight\ of\ Polymer}{Weight\ of\ total\ Solution}\ , wt\%\ of\ Polymer = 20wt\%$$

$$20\ wt\% = \frac{weight\ of\ Polymer}{3\ gram}$$

$$0.2 = \frac{weight\ of\ Polymer}{3\ gram}$$

$weight\ of\ Polymer = 0.6$ grams

Table of Data:

Table A1 Mass of Component

No.	Chemical	Mass (g) / Volume (ml)
1.	Magnesium Triflate	0.298 g
2.	1-ethyl-3-methylimidazolium trifluoro-	3 ml

	methanesulfonate (EMITf)	
3.	Poly (vinylidene fluoride hexafluoropropylene) (PVdF-HFP)	0.618 g
4.	Acetone Solution	3 ml

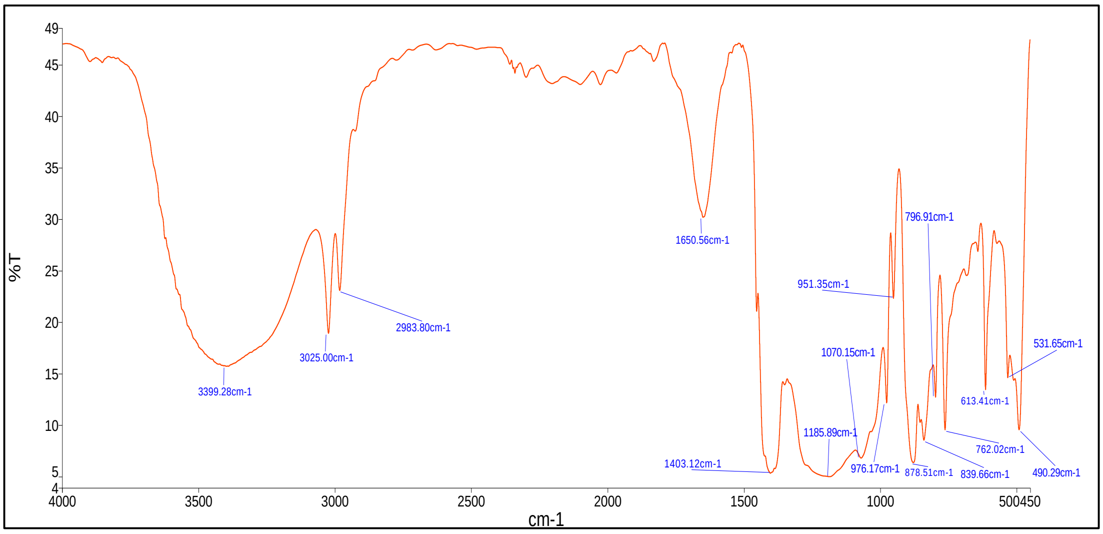


Figure A1 PVdf-HFP Film

Table A2 : FTIR Spectra reading on PVdf-HFP Film

Group	Wavelength Reference (cm^{-1})	Wavelength Sample (cm^{-1})
C = O	1670- 1820 (weak)	1650
C - O	1000 - 1300	1070, 1185
$-CF_2$	400 - 700	490, 531, 613
C-H	1350 - 1490	1403
C – O - C	850 - 1200	878 ,951, 976
CH_3	2400 - 3000	2983

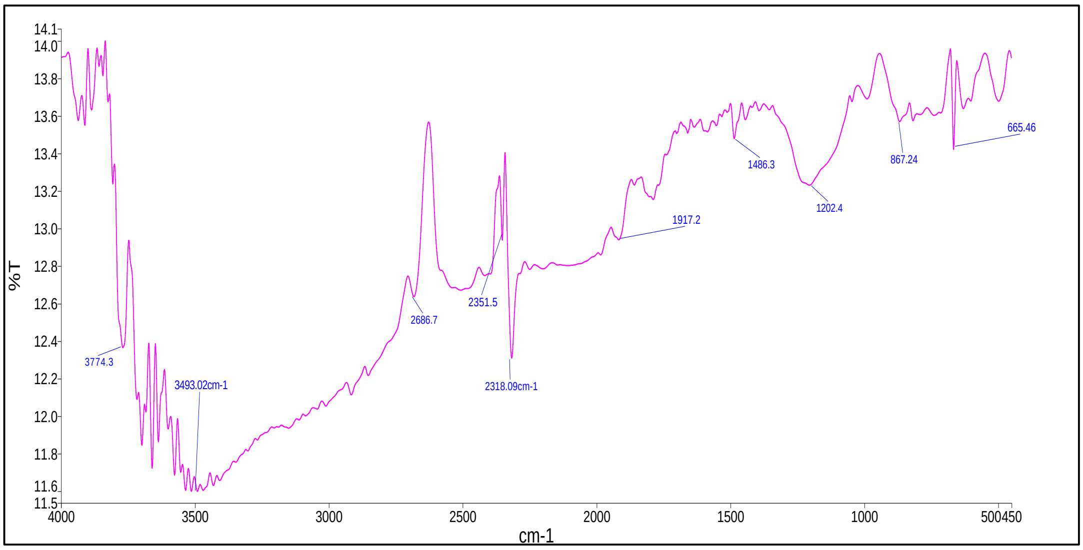


Figure A3 GPE with Mg/EMITF FTIR

Table A2 : FTIR Spectra reading on PVdf-HFP GPE Film

Group	Wavelength Reference (cm^{-1})	Wavelength Sample (cm^{-1})
C = O	1670- 1820 (weak)	1914
C - O	1000 - 1300	1202
$-CF_2$	400 - 700	665
C-H	1350 - 1490	1486
C – O - C	850 - 1200	867
CH_3	2400 - 3000	2686

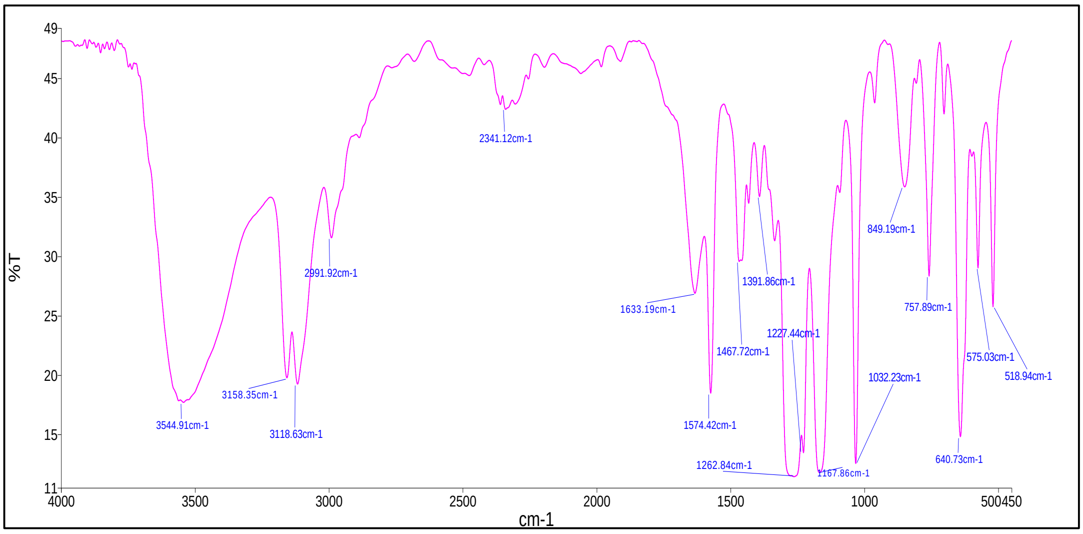


Figure A4 Ionic liquid, EMITF FTIR

Table A3 FTIR Spectra reading on Ionic liquid, EMITF

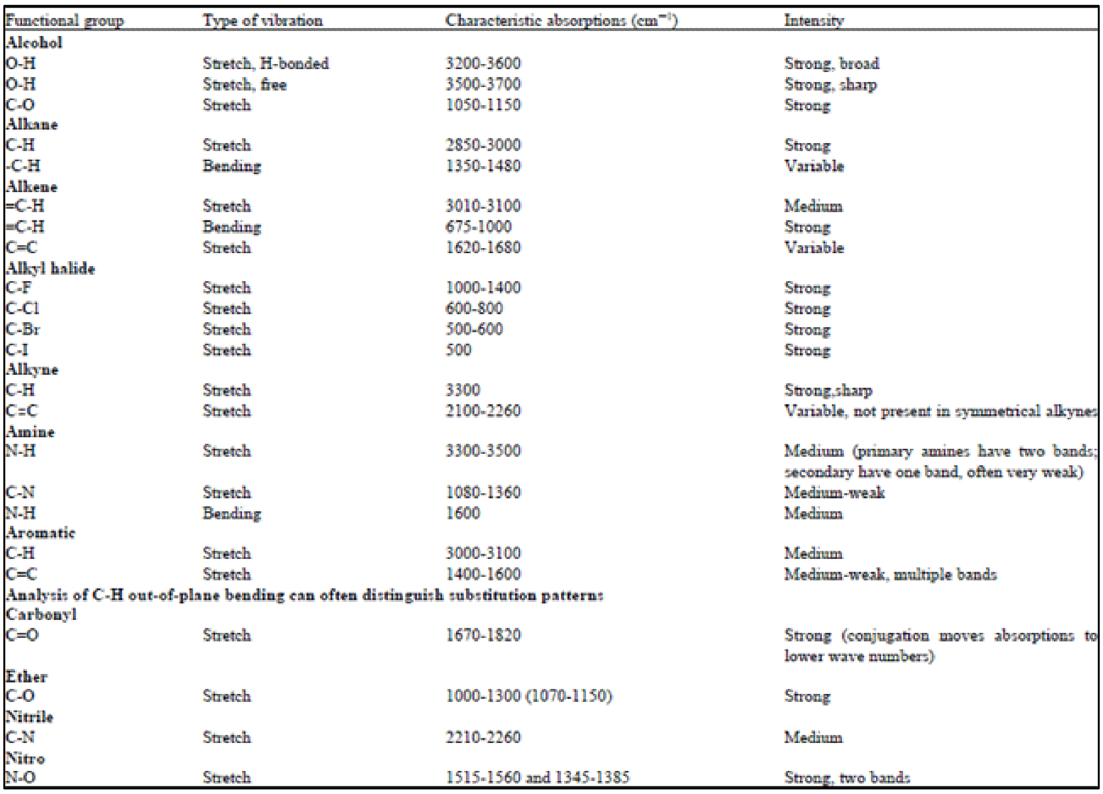
Group	Wavelength Reference (cm^{-1})	Wavelength Sample (cm^{-1})
C = O	1670- 1820 (weak)	1633
C - O	1000 - 1300	1032, 1157, 1227
$-CF_2$	400 - 700	518, 575, 640
C-H	1350 - 1490	1391, 1467
C – O - C	850 - 1200	1032, 1157
CH_3	2400 - 3000	2991

Table of wavelength

Component	Wavelength
PVdf-HFP	500 – 900 cm^{-1}
$(C_3SO_3)^-$	550 – 1200 cm^{-1}

(Pandey, 2009)

Wavelength Reference



Functional group	Type of vibration	Characteristic absorptions (cm^{-1})	Intensity
Alcohol			
O-H	Stretch, H-bonded	3200-3600	Strong, broad
O-H	Stretch, free	3500-3700	Strong, sharp
C-O	Stretch	1050-1150	Strong
Alkane			
C-H	Stretch	2850-3000	Strong
-C-H	Bending	1350-1480	Variable
Alkene			
=C-H	Stretch	3010-3100	Medium
=C-H	Bending	675-1000	Strong
C=C	Stretch	1620-1680	Variable
Alkyl halide			
C-F	Stretch	1000-1400	Strong
C-Cl	Stretch	600-800	Strong
C-Br	Stretch	500-600	Strong
C-I	Stretch	500	Strong
Alkyne			
C-H	Stretch	3300	Strong,sharp
C≡C	Stretch	2100-2260	Variable, not present in symmetrical alkynes
Amine			
N-H	Stretch	3300-3500	Medium (primary amines have two bands; secondary have one band, often very weak)
C-N	Stretch	1080-1360	Medium-weak
N-H	Bending	1600	Medium
Aromatic			
C-H	Stretch	3000-3100	Medium
C=C	Stretch	1400-1600	Medium-weak, multiple bands
Analysis of C-H out-of-plane bending can often distinguish substitution patterns			
Carbonyl			
C=O	Stretch	1670-1820	Strong (conjugation moves absorptions to lower wave numbers)
Ether			
C-O	Stretch	1000-1300 (1070-1150)	Strong
Nitrile			
C-N	Stretch	2210-2260	Medium
Nitro			
N-O	Stretch	1515-1560 and 1345-1385	Strong, two bands

Figure A5 Wavelength Table