

Electrochemical and Optical Studies on Aluminum-Ion Conducting Gel-Polymer Electrolytes

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Abstract

In the last three decades, Li-ion batteries have been developed with high energy and power densities to satisfy the need of electrochemical energy storage for portable electronics as well as automobiles. However, limited natural resources of lithium raw materials compelled researchers to investigate alternative battery chemistries beyond lithium, such as aluminum-ion batteries (AIBs). In this work, a set of novel gel-polymer electrolytes (GPEs) based on poly (ethylene oxide) (PEO) host polymer and aluminum sulphate ($\text{Al}_2(\text{SO}_4)_3$) ionic salt have been synthesized and characterized by electrochemical and optical means. The optimized 8PEO-10 $\text{Al}_2(\text{SO}_4)_3$ -40EC-42PC (wt.%) GPE composition showed an ambient temperature ionic conductivity of 0.25 mS cm^{-1} and the variation of ionic conductivity with inverse temperature showed Arrhenius behavior with almost same activation energies of about 0.14 ~ 0.15 eV for all the compositions studied in this work. DC polarization test on SS/GPE/SS configuration showed that the highest conducting composition is dominantly an ionic conductor ($t_{\text{ion}} \sim 0.99$) with negligible electronic conductivity, which is very much advantageous to avoid short circuits within the cell. Linear sweep voltammetry (LSV) studies showed that the electrochemical stability window of optimized GPE is about 3.7 V (Al/Al^{3+}). FTIR studies confirm the dissociation of ionic salt and the amorphous nature of these GPEs. The optimized GPE developed in this study with exceptional electrochemical stability and high ionic conductivity at ambient temperature has the great potential to be used in AIBs.

1. Introduction

In present day technological world, efficient and sustainable energy storage systems such as secondary batteries are crucial for stationary as well as portable applications. Although, lithium-ion batteries are the best choices for portable electronics as well as electric vehicles, limited availability and ever increasing prices of lithium raw materials pose major challenges [1-3]. Nowadays, several battery chemistries beyond lithium such as sodium, magnesium, zinc, aluminum, potassium and calcium are being explored, mostly at the laboratory R&D level. Among these, aluminum is the highest abundant material on the Earth's crust (8.1 wt.% compared to 0.002 wt.% of lithium; more than 4000 times compared to lithium), it also has the highest theoretical volumetric energy density (8040 mAh cm⁻³ for aluminum compared to 2046 mAh cm⁻³ for lithium) and good theoretical gravimetric energy density (2980 mAh g⁻¹ for aluminum compared to 3860 mAh g⁻¹ for lithium) [1,4,5]. Three-electron-transfer redox in aluminum is also an added advantage for efficient energy storage. Due to all these potential benefits, aluminum-ion batteries (AIBs) are heavily investigated these days.

Since the R&D works in the field of AIBs are at the early stages, several ongoing studies are mainly on design and characterization of suitable electrolyte and electrode materials. As far as the electrolytes are concerned, various types of electrolytes such as ionic liquids, polymer electrolytes, molten salts, aqueous and non-aqueous liquids have been studied in the recent past [6]. Solid electrolytes have low ionic conductivities at ambient temperature, liquid electrolytes have corrosion and leakage problems, ionic liquids are expensive and the molten salts have high operating temperatures. Chloroaluminate melts are the conventionally used electrolytes in AIBs. However, there are major challenges due to their extremely high corrosivity with electrodes, binders, separators and cell cases [5,7]. Hence, design of suitable electrolytes with reasonably good ambient temperature ionic conductivities is a hot topic in the field of AIBs. Nowadays, polymer based electrolytes are very commonly employed, typically in commercial lithium-ion batteries and many other prototype cells. Though the size of Al³⁺ (~ 106 pm) is smaller than that of Li⁺ (~ 152 pm) [8], which favor the Al³⁺ ionic mobility, dissolving aluminum salts in organic liquids is the primary challenge to synthesize polymer based electrolytes for AIBs.

Due to several advantages of having properties in the middle of solids and liquids, gel-polymer electrolytes (GPEs) are generally preferred for applications in various battery chemistries. There are a few reports in the literature on GPEs for AIBs, but almost all of those electrolytes have been synthesized by entrapping ionic liquids (ILs) within polymer matrices [4,5,7,9,10]. The work of Lin *et al.* [9] performed in 2015 was one of the ground breaking investigations on AIBs assembled with room temperature IL electrolytes. In this seminal work, Al//graphite cells were assembled with a chloroaluminate IL electrolyte; 1-ethyl-3-methylimidazolium chloride-AlCl₃ (EMImCl-AlCl₃). The power density, the energy density, the specific capacity and the Coulombic efficiency of the cell were reported as 3000 W kg⁻¹, 40 Wh kg⁻¹, 70 mAh g⁻¹ and 98%, respectively. Further, the cell showed stable cycling life up to 7500 charge/discharge cycles without decay and a drilling test on the cell while it is in operation confirmed that there are no safety hazards. Since then, several GPEs for AIBs have been synthesized by entrapping the same EMImCl-AlCl₃ IL in polymer matrices as summarized in the following paragraphs.

In 2016, Sun *et al.* [4] successfully prepared a GPE for AIBs with acrylamide as the functional monomer and the IL, EMImCl-AlCl₃ (1-1.5 molar ratio), as the plasticizer. It was found that the ionic conductivity increases with the amount of IL and the highest value recorded was 1.66 mS cm⁻¹ at 20 °C for the electrolyte containing 80 wt.% of the IL. The cyclic voltammetry (CV) studies showed that these GPEs exhibit reversible aluminum stripping/plating at 50 °C. In 2021, another set of GPEs for AIBs were synthesized by Elia *et al.* [7] using conventional solvent-casting method with polyacrylonitrile (PAN) polymer host and EMImCl-AlCl₃ IL. At room temperature, the ionic conductivity of the prepared GPE was estimated as 0.25 mS cm⁻¹ and the conductivity variation with inverse temperature showed Arrhenius behavior with an activation energy of 0.15 eV. It has also been reported that the prepared GPE showed lower corrosivity with stainless steel current collector compared to conventional EMImCl-AlCl₃ IL electrolyte. Further, the test cell, Al/GPE/pyrolytic graphite, showed good cycling ability exhibiting the suitability of the prepared GPE for secondary AIBs.

In 2021, Liu *et al.* [10] designed a polyamide based GPE (PAGPE) by dissolving polyamide into EMImCl-AlCl₃ room temperature IL mixed at various molar ratios of EMImCl and AlCl₃. The optimized PAGPE showed reversible Al stripping/plating, long term electrochemical stability and room temperature ionic conductivity of 6.61 mS cm⁻¹. The PAGPE electrolyte with Al//graphite electrodes worked well in a broad range of temperatures from -30 to 45 °C and the LSV studies revealed a wider electrochemical stability window of 2.6 V (vs Al/Al³⁺). In 2023, Zhang *et al.* [5] synthesized a GPE via a facile in-situ polymerization of cross-linked poly(ethyl acrylate) (PEA) in high molar ratio of EMImCl-AlCl₃ IL. This GPE showed an ionic conductivity of 1.46x10⁻³ S cm⁻¹, a wider electrochemical stability window of 3 V (vs Al/Al³⁺) and appreciable interfacial stability, at ambient temperature. The Al//graphite cells assembled with this GPE gave stable capacities of about 90 mAh g⁻¹ for 1000 cycles at the current density of 100 mA g⁻¹, at ambient temperature as well as at -10 °C.

As summarized above and also given in detail in the recent review articles [1,6], almost all the GPE works intended for AIBs are on ILs (mostly EMImCl-AlCl₃) entrapped within a polymer matrix. Since these ILs are highly expensive and the chloroaluminate is corrosive, alternative materials/methods to synthesize GPEs are highly

important for the development of next generation efficient and sustainable energy storage systems. In the present work, GPEs for AIBs have been synthesized for the first time by entrapping cheaper chlorine-free organic liquid based electrolyte, $\text{Al}_2(\text{SO}_4)_3$ -EC-PC, in a poly (ethylene oxide) (PEO) polymer matrix. To the best of our knowledge, there are no reports available in the literature on such organic liquid based GPEs for AIBs. Electrochemical and optical studies have been done on all the synthesized electrolytes and the optimized composition showed reasonably good ambient temperature ionic conductivity.

2. Experimental Procedure

All the starting materials such as poly (ethylene oxide) (molecular weight, 4×10^6), $\text{Al}_2(\text{SO}_4)_3$ (98%), EC (99%) and PC (99%) were obtained from Sigma-Aldrich (USA) and used as received. First of all, the required quantities of $\text{Al}_2(\text{SO}_4)_3$, EC and PC were magnetically stirred at room temperature for about 2-3 hours until the salt homogeneously dissolves into the EC-PC mixture. Then, the required amount of PEO was added and magnetically stirred again by gently heating the sample at around 75 °C for 6-7 hours until a clear transparent gel is obtained. All the GPE samples A, B, C and D (each of 1 g in weight) with different ether oxygen to Al^{3+} ion ratio (listed in Table 1) have been synthesized by using the same method mentioned above.

Table 1 Compositions of synthesized GPEs with different ether oxygen to Al^{3+} ion ratio

Sample	PEO (wt %)	$\text{Al}_2(\text{SO}_4)_3$ (wt %)	EC (wt %)	PC (wt %)
A	6	12	40	42
B	8	10	40	42
C	10	8	40	42
D	12	6	40	42

Electrochemical impedance spectroscopic (EIS) measurements were done by using Metrohm Autolab Potentiostat-Galvanostat (PGSTAT 128N, 10 μHz - 1 MHz, Netherlands) on disc shaped samples of diameter 5.4 mm and thickness 2.8 mm sandwiched between two spring-loaded stainless steel blocking electrodes (SS/GPE/SS) separated by a non-conducting spacer. The temperature of the samples was varied from 25 to 75 °C and the measurements were recorded at 10 °C intervals on heating. In order to make sure that the set and the actual temperatures are exactly the same, samples were kept at each set point for about an hour. The ionic conductivity values were estimated by using the respective bulk resistances extracted from the Nyquist plots.

Bruker - alpha II (USA) spectrometer at the attenuated total reflection (ATR) mode was used for recording FTIR spectra of all the samples in the wavenumber range from 600 to 2000 cm^{-1} . Room temperature DC polarization study on the highest conducting sample B was done under an applied voltage of 1V with stainless steel (SS) blocking electrodes. In order to determine the electrochemical stability window of the highest conducting sample, LSV study was performed on a cell configuration of $\text{Al}/\text{GPE}/\text{Al}$ with a scan rate of 5 mV s^{-1} .

3. Results and Discussion

3.1 Electrochemical Studies

Among various electrochemical techniques used to study the electrolytes for secondary batteries, EIS is one of the most commonly employed techniques typically used to estimate the ionic conductivity of the electrolytes. In this work, EIS data were recorded on all the GPE samples A, B, C and D with SS/GPE/SS configuration at 10 °C intervals on heating from 25 to 75 °C. Fig. 1(a) shows the Nyquist plots at ambient temperature for all the samples. The bulk resistance values have been extracted from these Nyquist plots by finding the intersection of the semi-circular portion with the low frequency spike. The ionic conductivity values have been estimated by using the respective bulk resistance values and Fig. 1(b) shows the corresponding σ vs $1/T$ plots for all the GPE samples studied in this work. As shown in Fig. 1(b), sample B shows the highest ionic conductivity at all experimental temperatures, possibly due to the optimized ionic concentration and enhanced mobility of ionic species within the polymer/salt complex. It was impossible to synthesize samples with PEO higher than 12 wt.% and less than 6 wt.% due to the formation of polymeric clusters and the difficulty in 'gel' formation, respectively. Evidently, σ vs $1/T$ plots for all the GPE samples shown in Fig. 1(b) follows Arrhenius behavior as in equation 1.

$$\sigma(T) = \sigma_0 e^{-E_a/kT} \quad (1)$$

where σ_0 is the conductivity pre-exponential factor, E_a is the activation energy for conduction and k is the Boltzmann constant.

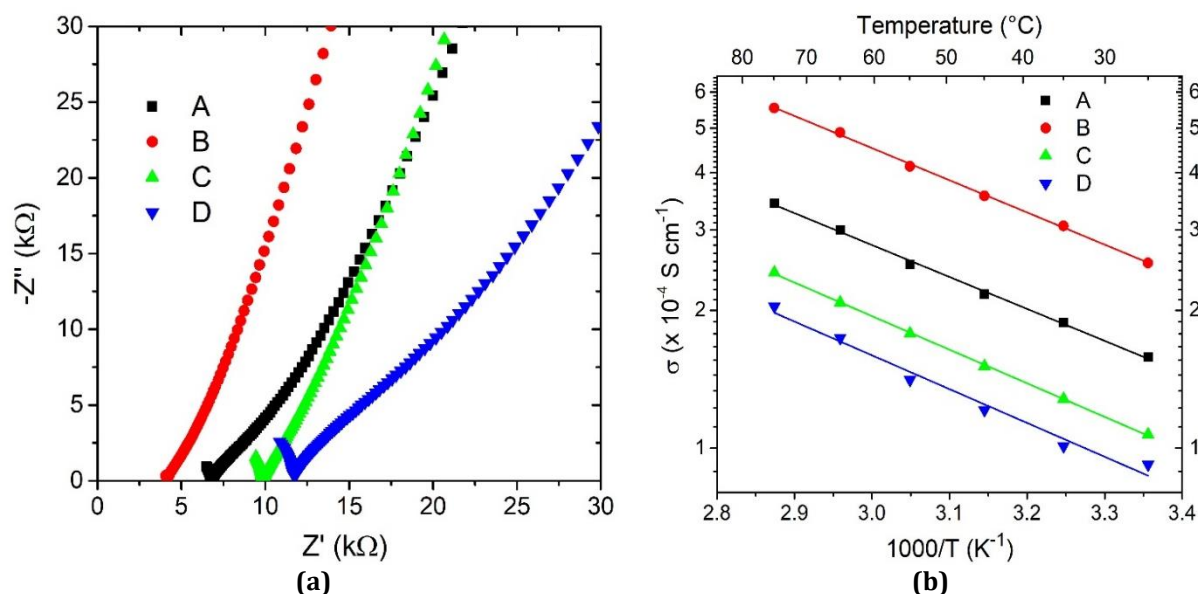


Fig. 1 (a) Nyquist plots at ambient temperature; and (b) σ vs $1/T$ plots, for the GPE samples A, B, C and D

Obviously, parallel lines with almost same slopes for samples A, B, C and D shown in Fig. 1(b) suggest that the activation energies are almost same for all the samples and also the calculated values of the activation energies seem to be very low (0.14 ~ 0.15 eV). The ambient temperature conductivity values and the activation energies for all the GPE samples have been listed in Table 2. Similar values of activation energies have been reported for some other Al^{3+} ion conducting GPE systems as well [5, 7, 11]. Low activation energies clearly indicate that the energy barrier for the migration of ions is lower, which facilitate the ionic mobility. This is mainly due to the completely amorphous nature of these 'gel' electrolytes. Almost the same values of activation energies for all the samples suggest that the activation process is mainly controlled by the EC/PC concentration in the electrolytes, not by the concentrations of polymer host and/or ionic salt. One can notice that the EC/PC concentration (82 wt.%) is the major component in all the GPEs studied in this work.

Table 2 Conductivity at ambient temperature (σ_{RT}), pre-exponential factor (σ_0) and the activation energy (E_a) of GPE samples synthesized with different ether oxygen to Al^{3+} ion ratio

Sample	σ_{RT} (S cm ⁻¹)	σ_0 (S cm ⁻¹)	E_a (eV)
A	1.6×10^{-4}	3.5×10^{-2}	0.14
B	2.5×10^{-4}	5.8×10^{-2}	0.14
C	1.1×10^{-4}	3.1×10^{-2}	0.15
D	1.0×10^{-4}	2.6×10^{-2}	0.15

As listed in Table 2, sample B showed the highest ambient temperature ionic conductivity of 0.25 mS cm^{-1} , which is comparable to that of a few other IL incorporated Al^{3+} ion conducting GPE systems reported in the literature. For example, a PAN polymer host and EMImCl-AlCl₃ IL based GPE system studied in 2021 showed an ambient temperature ionic conductivity of 0.25 mS cm^{-1} [7], which is exactly same as the highest conductivity value recorded in this work. It is also interesting to note that the activation energy reported for that system is 0.15 eV, almost same as that of the electrolyte samples investigated in this work. The electrolytes studied in this work consist of cheaper materials, the cost of organic liquids such as EC and PC are much cheaper compared to ILs. Further, GPE system reported in this work is free from chloride salts and hence corrosion problems with the electrodes and the current collectors can be minimized.

High dielectric constants of EC and PC help to reduce the Coulombic interactions between the ions and hence prevent the formation of crystalline phases and ion-aggregates. This leads to enhanced ionic conductivity. The ionic conductivity values estimated for the present Al^{3+} conducting GPEs are slightly lower than that of the commonly reported Li^{+} conducting solid and/or gel-polymer electrolyte systems [12-13]. Smaller size of Al^{3+} (106 pm) in comparison to that of Li^{+} (152 pm) and the three-electron-transfer redox in aluminum are favorable for better ionic conduction in aluminum systems. However, the lower ionic conductivity observed in present set of samples compared to that of lithium systems suggest that the heavy weight of Al^{3+} (26.98 g mol⁻¹) compared to that of Li^{+} (6.94 g mol⁻¹) [14] plays a critical role in constraining the Al^{3+} ionic mobility.

DC polarization test was performed on highest conducting composition (sample B) at ambient temperature with SS blocking electrodes to study the ionic and electronic contributions to the conductivity. Fig. 2(a) shows the normalized current vs time plot recorded under an externally applied voltage of 1V. From Fig. 2(a), the ionic transference number (t_i) was estimated by using Equation 2.

$$t_i = \frac{i_T - i_e}{i_T} \quad (2)$$

Here, i_T is the initial total current and i_e is the final residual current. The electronic transference number (t_e) was estimated by subtracting t_i from 1. For sample B, the estimated ionic transference number is 0.99 and the electronic transference number is 0.01. This clearly shows that the electrolyte is dominantly an ionic conductor with negligible electronic conductivity, which is desirable to avoid short-circuits within the electrolyte.

LSV data was recorded at ambient temperature on the highest conducting composition (sample B) with non-blocking aluminum electrodes in order to estimate the electrochemical stability window (ESW) of the electrolyte. Fig. 2(b) shows the LSV data recorded with 5 mV s⁻¹ scan rate and Al/GPE/Al configuration on sample B. Accordingly, the ESW of the highest conducting composition was estimated as 3.7 V (vs Al/Al³⁺). It is interesting to note that this value of ESW is higher than that of a few IL incorporated GPEs reported in the literature. Typically, Liu *et al.* [10] designed a polyamide based GPE (PAGPE) in 2021 by dissolving polyamide into EMImCl-AlCl₃ room temperature IL and their LSV studies on optimized PAGPE showed an ESW of 2.6 V (vs Al/Al³⁺). Another GPE synthesized *via* a facile in-situ polymerization of cross-linked poly (ethyl acrylate) (PEA) in high molar ratio of EMImCl-AlCl₃ IL showed an ESW of 3 V (vs Al/Al³⁺), at ambient temperature [5].

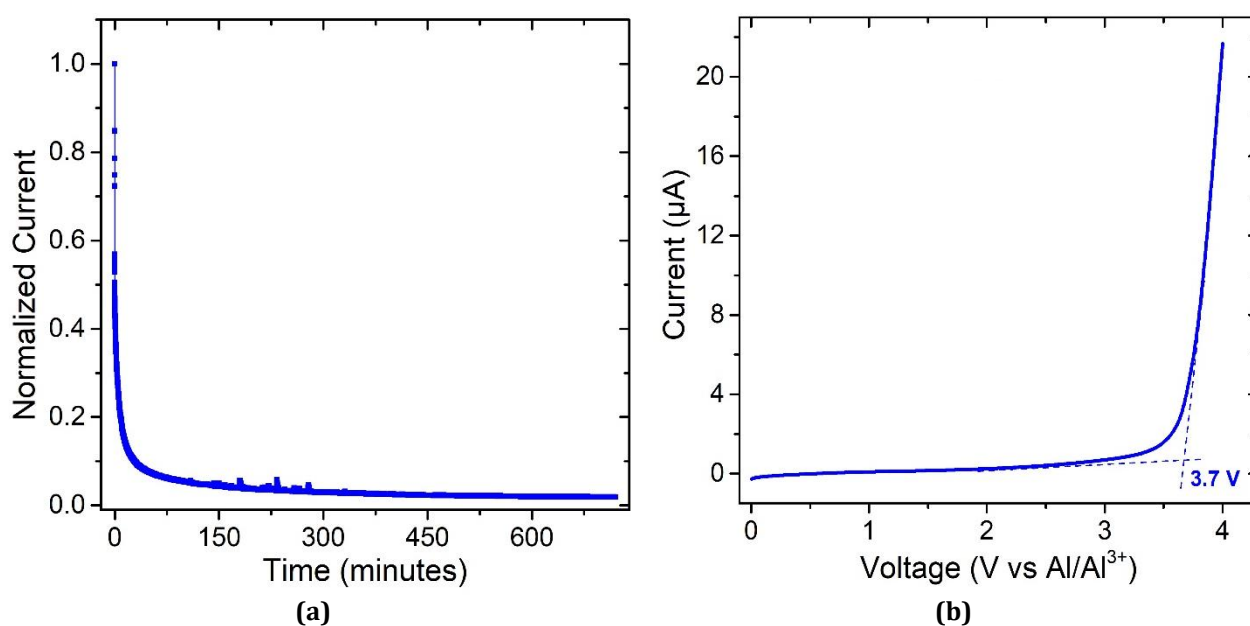


Fig. 2 (a) DC polarization data recorded with SS/GPE/SS configuration; and (b) Linear sweep voltammetry (LSV) data recorded with Al/GPE/Al configuration, on the highest conducting composition (sample B)

3.2 Optical Studies

FTIR studies help to investigate the ion-complexation arising from the dissociation of ionic salt within the polymer matrix. In this work, FTIR spectra were recorded in the wave-number range 600-2000 cm⁻¹ at ATR-FTIR mode on all the samples A, B, C and D and also on all the raw materials; PEO, Al₂(SO₄)₃, EC and PC (Fig. 3). To study the vibrational excitations in great detail, FTIR spectrum for the highest conducting composition (sample B) has been de-convoluted by using Gaussian-Lorentzian product function in Peak-Fit software and are shown in Fig. 4(a) and Fig. 4(b) in the wavenumber ranges 1000-1300 and 1700-1850 cm⁻¹, respectively along with relevant FTIR spectra for the raw materials.

The following mode-assignments have been made by comparing the FTIR spectra of GPEs with that of the raw materials and also by using available information in the relevant literature [15-17]. A summary of these assignments is given in Table 3.

- (i) Two sharp modes appearing at the wavenumbers 714 and 774 cm⁻¹ in all the GPEs and also in EC and PC (Fig. 3) have been assigned to the C=O bending vibrations arising from EC and PC.

- (ii) Two modes clearly visible in pure PEO at wavenumbers 843 and 960 cm^{-1} (Fig. 3) have slightly blue-shifted in all the GPEs. These modes have been assigned to CH_2 asymmetric and symmetric rocking vibrations, respectively. This is an indication of amorphous nature of the GPEs, because amorphous nature facilitates CH_2 rocking vibrations.
- (iii) The triplet of modes appearing at 1058, 1093 and 1145 cm^{-1} in pure PEO are due to C-O-C stretching vibrations. As clearly visible in the de-convoluted spectrum (Fig. 4(a)), these triplet of modes red-shifted in the GPEs compared with pure PEO. This red-shift may be due to the decrease in vibrational frequency of C-O-C stretching caused by the co-ordination of Al^{3+} ions with ether oxygen atoms in the PEO backbone.
- (iv) A sharp mode appearing at 1067 cm^{-1} (Fig. 4(a)) in EC is due to ring breathing vibration.
- (v) The modes at 1240 and 1279 cm^{-1} appearing in pure PEO (Fig. 4(a)) have red-shifted with very low intensities in the GPEs. These modes are arising from the CH_2 twisting vibrations.
- (vi) A doublet appearing at 1353 and 1390 cm^{-1} (Fig. 3) in PC and also in all the GPEs is due to CH_2 wagging.
- (vii) A small intense mode appearing at 1480 cm^{-1} in EC/PC and also in all the GPEs (Fig. 3) has been assigned to CH_2 scissoring. This mode appears at 1465 cm^{-1} in pure PEO and then blue-shifts due to complexation with ionic salt.
- (viii) The highest intense mode in all four GPEs is the doublet that can be seen in the wavenumber range 1700-1850 cm^{-1} (Figs. 3 and 4(b)), which arises from EC and PC. As shown in Fig. 4(b), the doublet of EC is enveloped within the broad mode of PC in the gel-polymer samples. These modes are coming from C=O stretching vibrations in EC and PC.

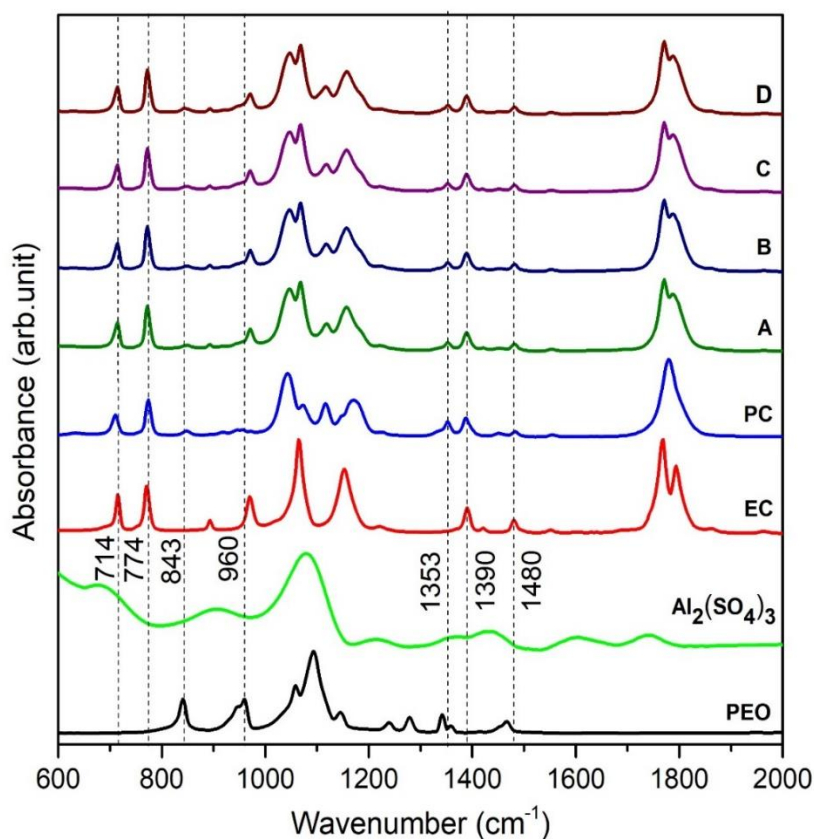


Fig. 3 FTIR spectra of GPE samples (A to D) and the raw materials (PEO, $\text{Al}_2(\text{SO}_4)_3$, EC and PC) in the wavenumber range 600 - 2000 cm^{-1}

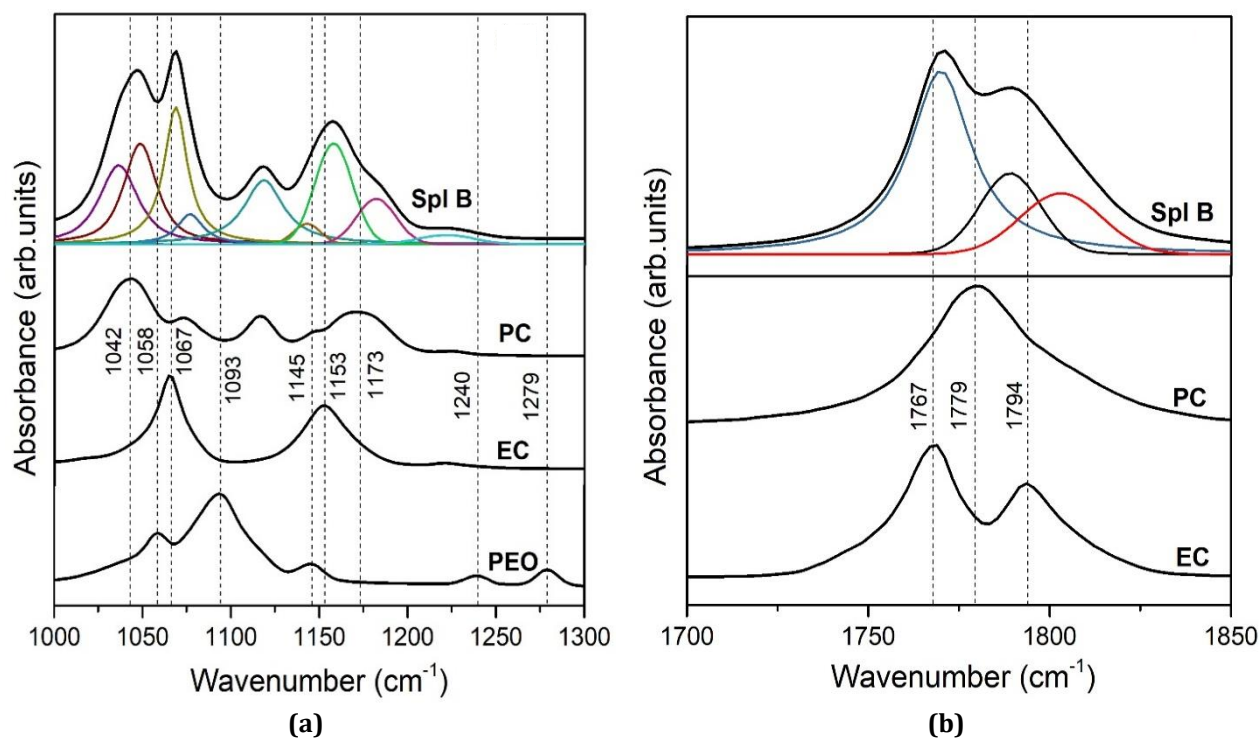


Fig. 4 Comparison of FTIR Peak-fit deconvolution for sample B with the FTIR spectra of relevant raw materials in the wavenumber range (a) 1000 - 1300 cm^{-1} ; and (b) 1700 - 1850 cm^{-1}

Table 3 Summary of FTIR vibrational mode assignments [15-17]

Wavenumber (cm^{-1})	Mode assignment
714 and 774	C=O bending in EC and PC
843	CH_2 asymmetric rocking in PEO
960	CH_2 symmetric rocking in PEO
1042	C-O-C stretching in PC ring
1058, 1093 and 1145	C-O-C stretching triplet in PEO
1067	Ring breathing in EC
1240 and 1279	CH_2 twisting doublet in PEO
1353 and 1390	CH_2 wagging doublet
1480	CH_2 scissoring
1767, 1779 and 1794	C=O stretching vibrations in EC and PC

4. Conclusion

In this work, a new set of Al^{3+} ion conducting GPEs based on PEO polymer host have been synthesized and characterized by electrochemical and optical means. The composition 8PEO-10 $\text{Al}_2(\text{SO}_4)_3$ -40EC-42PC (wt.%) showed highest ambient temperature ionic conductivity of 0.25 mS cm^{-1} and the variation of ionic conductivity with inverse temperature showed Arrhenius behavior with almost same activation energies ($\sim 0.14 - 0.15$ eV) for all four GPE compositions studied in this work. DC polarization test on SS/GPE/SS configuration showed that the highest conducting composition is dominantly an ionic conductor ($t_{\text{ion}} \sim 0.99$) with negligible electronic conductivity. LSV studies show that the electrochemical stability window of optimized GPE is about 3.7 V (Al/Al^{3+}) and the FTIR vibrational spectroscopy confirmed the dissociation of ionic salt into constituent ions and the amorphous nature of synthesized electrolytes.

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Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** K. Thenmolie, K. Vignarooban; **data collection:** K. Thenmolie, G. Sashikesh, V. Jathushan; **analysis and interpretation of results:** K. Thenmolie, J.H.T.B. Jayamaha, K. Prashanthan; **draft manuscript preparation:** K. Thenmolie, K. Vignarooban. All authors reviewed the results and approved the final version of the manuscript.

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