

Electrochemical and Spectroscopic Studies of Nanocomposites Laden with BaTiO₃-grafted-graphene Oxide

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ABSTRACT

The electrochemical behavior of lithium-sulfur batteries was investigated by means of electrochemical impedance spectroscopy (EIS). Films of NCPEs (PEO+LiCF₃SO₃+BaTiO₃-g-GO) were cast using a novel hotpress technique. The addition of filler in fractional amount to the solid polymer matrix at room temperature further enhances the ionic conductivity. The membranes were subjected to cycling, FT-IR and Raman spectroscopic analyses. The occurrence of ion-ion pairs interaction has been identified using FT-IR and Raman analysis. Cells with this electrolyte have a high coulombic efficiency of 98% and a high cycling stability. **Keywords :** FT-IR, Raman; ionic Conductivity, Cycling, Coulombic efficiency

I. INTRODUCTION

The escalating demand for energy from transport and electronic sectors has prompted researchers to find alternative energy conversion/storage devices across the world¹. Approximately 68% of today's electrical energy is supplied from fossil fuels. Recent increase in the demand for oil, associated with the price increase and environmental issues continue to exert pressure on an already stretched world energy infrastructure²⁻⁵. However, alternative energy sources such as solar and windmill are intermittent and seasonal which make them unreliable. Hence advanced energy conversion and storage devices are required to meetout the increasing energy demand and their utilization effectively. The electrochemical energy conversion and storage devices convert chemical energy into electrical energy. Fuel cells, supercapacitors and lithium batteries are recognized as strong contenders to fulfil the future energy requirement.

The benefits of the lithium-ion battery, such as highenergy density, good performance, no- memory effect, high single cell voltage, and long cycle life have qualified lithium-ion batteries as ultimate power source for portable electronic devices such as digital cameras, laptop computers, mobile phones, etc., gave the battery a rapid acceptance in the market^{6,7}. Lithium–sulfur (Li–S) energy storage, which can deliver a high theoretical energy density of 2600 Wh kg⁻¹, has been considered as one of the most promising candidates for the next-generation energy storage devices⁸⁻¹². The high energy density is achieved by utilizing the high-capacity sulfur cathode (1672 mA h g-1) and lithium metal anode (3860 mA h g⁻¹), as well as the lowest negative electrochemical potential of the anode (3.040 V vs. the standard hydrogen electrode. Furthermore, sulfur enjoys the advantages of abundant resources, low costs, and high biocompatibility, which are particularly attractive for reliable bulk energy storage applications ^{13–19}. The emergence of the Li–S battery is traced back to the 1960s, yet as compared to the rapid advancement of LIBs, it is still the incumbent technology^{8,19}. Successful operation of Li/S batteries has been achieved through the development of composites of sulfur with carbonaceous²⁰⁻²⁹ and polymeric³⁰⁻³³ materials. In these composites, the S particles are embedded into the conductive carbon or polymer matrices ^{20,27,34}, which enhance the electronic conductivity of the composite and hinder the dissolution of polysulfides into the electrolyte³⁵⁻³⁸.

Another strategy to improve the capacity and cyclability of Li/S batteries is the electrolyte optimization so as to reduce the loss of sulfur by dissolution in the liquid electrolyte^{36,39-43}. Among the possible electrolyte modifications, replacement of the common liquid organic electrolytes with polymer electrolytes has proved promising and efficient. Polymer electrolyte may generally be defined as a membrane that possesses transport properties comparable to that of common liquid ionic solutions⁴⁴. It is possible and convenient to group all the polymer systems into two broad categories, i.e., pure solid polymer electrolyte (SPE) and plasticized or gel polymer electrolyte systems (GPE). The first category, pure solid polymer electrolyte, is composed of lithium salts (e.g., LiClO₄, LiBF₄, LiPF₆, LiAsF₆, LiCF3SO3, LiN(CF3SO2)2, LiC(CF3SO2)3 dissolved in high molecular weight polyether hosts, (e.g., PEO and PPO) which acts as solid solvents⁴⁵.

In the present study, the characterization techniques for Lithium sulphur batteries for various fillers and concentrations are studied. In the present work, a unique hybrid nanoparticle consisting of BaTiO₃ which is chemically coupled to graphene oxide (GO) sheets was employed as a filler^{46,47}. This hybrid particle was suitably incorporated in a PEO matrix along with a lithium salt to design polymer electrolytes for all-solid-state lithium-batteries and its spectroscopic and electrochemical properties are studied.

II. METHODS AND MATERIAL

A. Preparation of NCPE

PEO-based nanocomposite polymer electrolyte (NCPE) were prepared by a novel dry (solution free) hot-press method as reported earlier⁴⁸. Precursors used were, PEO,LiCF₃SO₃,BaTiO₃-g-GO. The total weight of polymer, inert filler and lithium salt was set to be 100% by weight. The solution was continuously stirred in a magnetic stirrer for 6 hours at room temperature. The viscous solution was cast as a membrane. Upon evaporation of solvent the precursor was hot-pressed in order to get a uniform membrane. The thickness of the membrane was between 50-60µm. Subsequently, this membrane was stored in a vacuum oven for further characterization.

Table 1 : Composition of PEO,LiCF₃SO₃,BaTiO₃-g-GO

| Sample | PEO | LiCF ₃ SO ₃ | BaTiO₃-g- |
|--------|-------|-----------------------------------|-----------|
| | wt(%) | wt(%) | GO |
| S1 | 98 | 2 | 0 |
| S2 | 95 | 3 | 2 |
| S3 | 90 | 5 | 5 |
| S4 | 85 | 10 | 5 |
| S5 | 80 | 12 | 8 |

B. Characterization of NCPE

In the present study, sample S5 was used for both spectroscopic and electrochemical characterizations as this sample was found to be optimal in terms of ionic conductivity. The ionic conductivity of the samples was measured by sandwiching the samples between two stainless steel blocking electrodes. The measurements were performed using an electrochemical impedance analyzer (Bio-Logic InstrumentSP-31, France) between 100 kHz and 50 mHz for different temperatures between 30°C and 60 °C.

The compatibility of NCPE with lithium metal anode was studied by assembling symmetric cell composed of Li/NCPE/Li and measuring its interfacial resistance R_i as a function of time under open circuit condition at 60°C.

This interfacial resistance plays a vital role in determining the properties that include shelf life, safety, lithium deposition, dissolution, efficiency and cycle life.

The coin cells composed of Li/NCPE/S were assembled. The punched disks of cathode placed at the cathodic can and well polished disc of lithium metal(thickness: 0.75 mm, Aldrich) foil placed in anodic can of the coin cell keeping NCPE separator in between. The cells were assembled in a controlled, argon atmosphere dry-box (MBRAUN LabMaster 130) having both humidity and an oxygen content below 1ppm. After assembling, the cells were housed in argon filled glove box.

The Lithium/NCPE interface was analyzed by FT-IR and Raman, Thermo NICOLET Corporation, Nexus Model-670) by single internal reflection (SIR) mode.

III.RESULTS AND DISCUSSION

A. Ionic Conductivity

The conductivity of the polymer electrolyte can be calculated from the equation σ = L/(AR), where A is the area of the electrode, L is the thickness of the film and R is the bulk resistance.



Figure 1 : Variations of ionic conductivity as a function of inverse temperature of NCPE's

The ionic conductivity of NCPEs as a function of inverse temperature for various concentrations of PEO, LiCF₃SO₃ and BaTiO₃-g-GO are depicted in Figure 1.

In the present study, the observed increase in ionic conductivity is attributed to the interaction of Lewis acidic center of BaTiO₃-g-GO with anions of the LiCF₃SO₃ salt which reduces the crystallinity of the polymeric host⁴⁶⁻⁵⁰. This reduction in the value of crystallinity could be the reason for the increase in ionic conductivity.

B. Compatibility

Initially for the PEO+LiCF₃SO₃ complex the value of interfacial resistance, 'R_i' increases exponentially. The growth of the interfacial resistance does not follow a regular trend. The interfacial resistance of the NCPE has been significantly reduced upon the incorporation of BaTiO₃-g-GO and after 150 hours the 'R_i' values do not change much.



Figure 2. Variation of interfacial resistance as a function of time for the samples S1(PEO+LiCF₃SO₃) and S5 (PEO+LiCF₃SO₃+BaTiO3-g-GO)

This may be attributed to the morphological changes of the passivation films that varies with time to finally acquire a non-compact, possibly porous structure. This insulating layer will impede electrode reactions. This may very well have happened when excessive amount of the passive BaTiO₃ phase were introduced into the polymer matrix

C. Charge-Discharge

The prepared S-AC active material was mixed with poly (vinylidene fluoride), carbon black in N-methyl Pyrrolidinone (NMP, anhydrous,99.5%, Aldrich). The sulfur-carbon binder mixing ratio was 80-10-10 wt %. After homogenization, the slurry was coated onto a 20 μ m thick Aluminium current collector. The resulting positive electrode was dried at 55° C for 24 hours and then cut into 14 mm diameter disks. The coating thickness was about 80 μ m so as to obtain 100 μ m thick electrode after drying.

Figure 3. shows a well defined stable plateau at 2.4V vs Li⁺ on charge and 2.1V vs Li on discharge at different C-rates. A small drop in voltage indicates a lower interfacial resistance and is attributed to the better adhesion of the NCPE on the electrode



Figure 4. Discharge capacity as a function of cycle number PEO+LiCF₃SO₃+BaTiO₃-g-GO

Figure 4 shows an initial discharge capacity of 698 mAh g^{-1} at 0.1C- rate and it decreases to 489 mAh g^{-1} at 1C- rate with the Coulombic efficiency 99% and at 2C- rate it drastically reduces to 280 mAh g^{-1} .The cell offers a discharge capacity of 198 mAh g^{-1} at 5C- rate without much in fade in capacity. This fade in capacity with increase in C- rate is attributed to the composition of cathode material which has to be optimized and formation of SEI. The declining discharge capacity at higher C- rates may also be due to the solid electrolyte interface (SEI) film formation with electrolyte decomposition

The better cyclability with appreciable Coulombic efficiency at low C- rates is attributed to the slower diffusion of polysulfides to the anode than the total

electrochemical reaction time. Generally, measurement of Coulombic efficiency can be widely employed to quantify the shuttle process

D. FT-IR

FT-IR has been identified as a powerful tool to study the interaction and complexation between the polymer host, fillers and lithium salts. Since PEO has a tendency to absorb water extreme care had been taken to analyse the FTIR for the polymeric membranes. The spectra of electrolyte films reported here are free from water peaks as well as those due to residual acetonitrile solvent.



Figure 5. FT-IR spectra of PEO+LiCF₃SO₃+BaTiO₃g-GO

The FTIR spectra were recorded between 400 and 4000 cm⁻¹ at room temperature. The data is analysed only on the basis of the literature available for the - CH₂- scissoring, -C-O-C- stretching, -CH₂ twisting, and -CH₂- wagging modes of PEO.PEO exhibits -C-H stretching (between 2800 and 2935 cm⁻¹), asymmetric stretching (1950-1970 cm⁻¹), asymmetric bending (1450 cm⁻¹), CH₂ scissoring (1465-1485 cm⁻¹), C-O-C- stretching (1250-950 cm⁻¹), -CH₂- twisting (991 cm⁻¹), and -CH₂- wagging (842 cm⁻¹). The peak at 839cm⁻¹ attributed to CH₂ wagging, and corresponds to the Gauche conformation of PEO.³⁶ The spectral region 800-1000 cm⁻¹ depicts characteristic peaks of C-O

stretching and –CH₂-rocking modes which may be due to the presence of crystalline phase of PEO.

Upon addition of 2% of LiCF₃SO₃ in the PEO matrix the intensity of the peaks at 1239cm⁻¹ and 1278cm⁻¹ gets increased which confirms the CF₃ vibration and SO2 vibrations of LiCF3SO3. The strongest band of PEO at 1104cm⁻¹ due to asymmetric -C-O-Cstretching is strongly affected by cation complexation. The intensity of the intense broad peak at 2870 cm⁻¹ decreases with increasing content of BaTiO₃ as shown in figure. Apparent changes occurring to the characteristic peaks of the polymer systems, confirms the cooperative interactions between PEO, LiCF₃SO₃, and BaTiO₃^{51,52}.

The observed shifts and intensities in vibration of the FTIR spectrum of NCPE suggest that there is complete complexation of filler and salt with polymer PEO.

E. Raman



Figure 6. Raman spectra of PEO+LiCF₃SO₃+BaTiO₃-g-GO

The regions of Raman spectra between $500 \text{cm}^{-1} - 4000 \text{ cm}^{-1}\text{ of PEO: LiCF}_3\text{SO}_3$: BaTiO_3-g-GO, S1(98:2), S2(95:3:2), S3(90:5:5), S4(85:10:5), S5(80:12:8) samples are shown in figure 6.The SO₂ stretching modes of the uncoordinated anions appear at 1300

cm⁻¹, 1325 cm⁻¹, 1320 cm⁻¹ and 1343 cm⁻¹.It is also clear from figure that peaks at 1300 and 1480 cm⁻¹ represents the intensity of the D and G- Raman scattering of graphene oxide from BaTiO₃-g-GO⁵³.

The Raman spectra further support the structural change before and after the reduction of GO. The D-band was broadened and shifted to around 1310 cm⁻¹ and the G-band shifted to 1489 cm⁻¹, indicating the reduction in size of sp² domains due to extensive oxidation with PEO and salt. After reduction, the complexes show a higher D/G intensity ratio than PEO indicating that introduction of sp³ defects after functionalization⁵⁴

IV.CONCLUSION

The NCPE's composed of PEO+LiCF₃SO₃ + BaTiO₃-g-GO were prepared by a simple hot press technique subjected and were to spectroscopic and electrochemical characterizations. The ionic conductivity of polymeric membrane were increased upon addition of BaTiO₃-g-GO in the polymeric matrix. Among the samples analyzed the polymeric membrane with the composition of PEO: LiCF₃SO₃: BaTiO₃-g-GO (80:12:8) was found to be optimal in terms of ionic conductivity. The interfacial resistance "Ri" values have been reduced upon incorporation of fillers in the NCPEs and is attributed to the formation of an insulated layer of BaTiO₃-g-GO. This insulating layer will impede further electrode reactions. The formation of new compounds on the lithium surface also confirmed by FT-IR analysis. The cycling profile of Li/NCPE/S cells at 60°C showed that the cells composed of NCPEs (PEO+LiCF₃SO₃+ BaTiO₃-g-GO) are capable of delivering 689 mAh/g and may be a good candidate for electric vehicle applications.

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