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Ac Conductivity of Sodium perchlorate doped PEO composites: Battery Application

KEYWORDS: Ionic polymer, ac conductivity, Slurry technique, FTIR, SEM

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ABSTRACT *PEO – $NaClO_4$ composites have been prepared by slurry method using acetonitrile as solvent. The prepared composites is characterized by FTIR and surface morphology was carried out by SEM. Ac conductivity shows that 50 wt% has high conductivity due to ionic polarization and therefore it has low dielectric loss value of 1.5 × 10^{-8}.*

Introduction

Substantial research efforts have been devoted to improve ion conductivity in solid polymer electrolyte (SPE) materials at ambient temperature for potential application in energy storage by new types of high performance solid state batteries, energy conversion by the fuel cells, chemical sensors, electrochemical capacitors, electro-chromic windows or displays, an a Substantial research efforts have been devoted to improve ion conductivity in solid polymer electrolyte (SPE) materials at ambient temperature for potential application in energy storage by new types of high performance solid state batteries, energy conversion by the fuel cells, chemical sensors, electrochemical capacitors, electro-chromic windows or displays, analog memory devices and many others. To enhance the performance of SPEs in electrochemical utilities, several modifications are explored such as incorporation of nano- and inorganic ceramic fillers [1–3], and plasticizers [4], etc., in polymer electrolytes. In the present work author reported ac conductivity of PEO/$NaClO_4$ composites for possible battery application.

Experimental

Polyethylene oxide (relative molecular mass of 500 000) is dried under vacuum at 50 ^{0}C for 24 h before use. Sodium perchlorate ($NaClO_4$) is also dried under vacuum at 120 ^{0}C for 24 h to remove water from the crystal. Pure PEO and PEO/$NaClO_4$ composites are prepared by solution casting technique using anhydrous acetonitrile. First PEO is dissolved in anhydrous acetonitrile and continuously stirred for 5-6 hrs to obtain homogeneous solutions/gels. The gels were poured into glass plates in a dry room fume hood and left for 12 hrs to remove the majority of the acetonitrile. The PEO/$NaClO_4$ (90/0, 80/20, 70/30, 60/40 and 50/50) composites are prepared by adding different weight percentages $NaClO_4$ in homogeneous PEO solutions/gels. The gels are then poured into glass plates in a dry room fume hood and left for 12 hrs to remove the majority of the acetonitrile. The composites are then further dried at 60 ^{0}C under vacuum for 24 hrs. The composites thus obtained are stored in the dry room at 22 ^{0}C for several weeks prior to characterization. The thickness of PEO/$NaClO_4$ composites is found to be varies from 1 mm [5].

The FTIR spectra of pure PEO and PEO/$NaClO_4$ composites are recorded over the wavenumber range 400 to 4000 cm^{-1} using Perkin Elmer (model 1600) IR spectrometer in KBr medium at room temperature. For recording IR spectra the powder of samples are mixed with KBr in the ratio 1:25 to ensure the uniform dispersion of samples in KBr pellets. The mixed powders are the pressed in a cylindrical die to obtain clean discs of thickness 1 mm.

The morphology of pure PEO and PEO/$NaClO_4$ composites are investigated using Phillips XL30 ESEM scanning electronic microscope (SEM). To obtain SEM image the samples in the form of pellets are mounted on aluminum platform. The conducting gold is then sputtered on the sample to avoid charging at the sample surfaces. The samples are then examined under SEM and selected areas were photographed. The grain size is calculated using linear intercept technique and average grain size is calculated by the following equation.

$$\text{Grain size} = 1.56\ \frac{C}{MN} \quad (1)$$

where, C is the length of the test line(s), N is the number of intercepts and M is the magnification calculated from the reference scale printed on the micrograph.

3. Results and Discussion

3.1. Fourier Transform Infra red Spectroscopy

The FTIR spectra of pure PEO, $NaClO_4$ and PEO/$NaClO_4$ (50:50 wt.%) composite are shown in Fig 1(a-c). The FTIR spectra of pure PEO shows peaks at 3422 cm^{-1}, 2922 cm^{-1}, 2359 cm^{-1}, 2164 cm^{-1}, 1971 cm^{-1}, 1813 cm^{-1}, 1639 cm^{-1} 1467 cm^{-1}, 1344 cm^{-1},1120 cm^{-1}, 962 cm^{-1},642 cm^{-1} and 530 cm^{-1}. Since the PEO is highly hydrophilic a broad band appears at 3422cm^{-1}. The band at 2922 and 2359 cm^{-1} are respectively due to asymmetric and symmetric CH2 stretching. The band at 1467 cm^{-1} is due to the asymmetric CH2 bending and at 1344 cm^{-1} corresponds to the symmetric CH_2 wagging and some C- C stretching.

The FTIR spectra of pure $NaClO_4$ shows peaks at 3552 cm^{-1}, 3481 cm^{-1}, 3237 cm^{-1}, 2025 cm^{-1}, 1617 cm^{-1} and 479 cm^{-1}. The presence of Peak at 3552 cm^{-1} is due to the water absorption by $NaClO_4$. The peaks at 3481 cm^{-1} and 3237 cm^{-1} are due to O-H vibration and it is formed at high frequency range due to the splitting of water molecule. The peaks observed at 2025 cm^{-1} is due to the Na^+ ions, 1617 cm^{-1} and 479 cm^{-1} are due to chlorate (ClO_4–) ions.

The FTIR spectra of PEO/$NaClO_4$ (50:50 wt.%) composite shows prominent peaks at 3551 cm^{-1},3470 cm^{-1},3414 cm^{-1},2926 cm^{-1},1948 cm^{-1},1896 cm^{-1},1618 cm^{-1},1467 cm^{-1}, 1342 cm^{-1},1282 cm^{-1},1240 cm^{-1},966 cm^{-1},936 cm^{-1},623 and 484 cm^{-1}. Thus the FTIR spectra of PEO/$NaClO_4$ (50:50 wt %) composite shows the prominent of both PEO and $NaClO_4$ which confirm the formation of PEO - $NaClO_4$ composite.

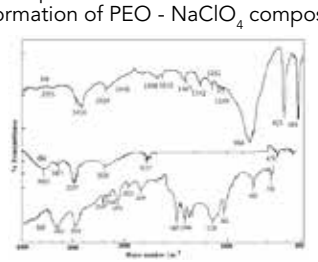

Figure 1 shows the FTIR spectra of PEO, $NaClO_4$ and PEO - $NaClO_4$ composites

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3.2 Scanning Electron microscopy

The scanning electron micrographs (SEM) of the PEO and PEO/$NaClO_4$ (50:50) composite are given in fig 2 (a-b). The SEM image of pure PEO shows highly crystalline and elongated rod like structure. Further the SEM image clearly shows considerable intragranular distance between two grains which indicates that PEO grains have less interaction with each other. The average grain size of PEO granules is estimated using the linear intercept formula and it is found to be 35 to 40μm. The SEM images of PEO: $NaClO_4$ (50:50 wt %) composites shows significant changes in morphology. The SEM image of PEO: $NaClO_4$ shows highly agglomerated structure. Further the grains are found to have irregular shape. The average grain size is estimated to be 2.7 mm. The granules are found to be interlinked with each other which decreases the intragranular distance [6]. The decrease in the intragranular distance between the grains may support the charge transfer mechanism from one grain to another grain.

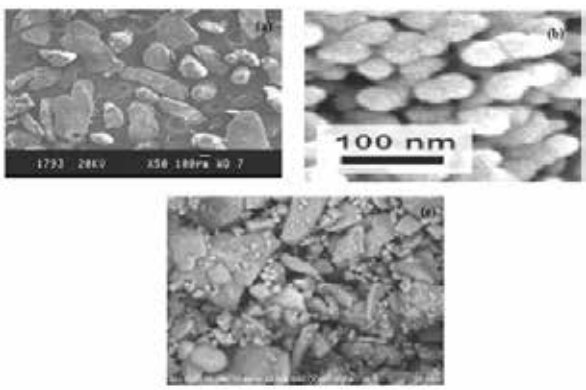

Figure 2 shows the SEM image of PEO, $NaClO_4$ and PEO - $NaClO_4$ composites

3.3 Ac conductivity

Figure 3 (a) shows the variation of ac conductivity as a function of frequency for Polyethylene oxide. The ac conductivity of Polyethylene oxide exhibit two phases in the frequency range 10^2 Hz to 10^6 Hz. In frequency between 10^2 Hz to 10^4 Hz, the conductivity values are almost constant and increases suddenly in the frequency range 10^4 – 10^6 Hz. Lattice polarization around a charge in localized state may be responsible for multiple phases of conductivity in Polyethylene oxide. Figure 3 (b) shows the variation of ac conductivity as a function of frequency for different weight % of PEO/$NaClO_4$ composites. It is observed that, the composite 40 wt % of PEO: $NaClO_4$, shows high conductivity which is due to ionic polarization and electrode polarization [7].

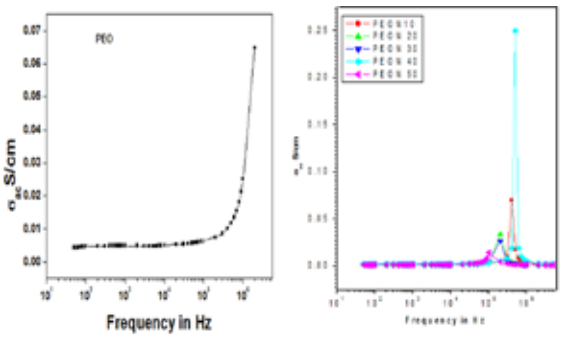

Figure 3 (a) shows the variation s_{ac} S/cm versus frequency for PEO and 3 (b) shows the variation of ac conductivity as a function of frequency for different weight % of PEO/ $NaClO_4$ composites.

Polyethylene oxide: Sodium chlorate ($NaClO_4$)

The variation of dielectric loss as a function of frequency for polyethylene oxide: Sodium chlorate ($NaClO_4$) composites (different wt %) is represented in Figure 4. Even though $NaClO_4$ is added up to 50 wt% in PEO, the behavior of dielectric loss with respect to frequency follows the same trend as that of pure PEO. At higher frequencies these composites exhibit almost zero dielectric loss which suggests that these composites are lossless materials at frequencies beyond 1 kHz.

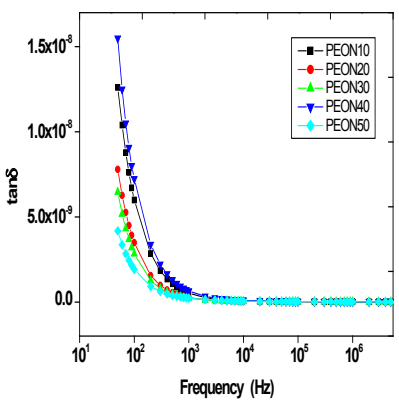

Figure 4 shows the variation of tan delta as a function of frequency for different weight % of PEO:$NaClO_4$ composites

3.4 Conclusion

Composites of $NaClO_4$ doped in PEO have been prepared by slurry method using acetonitrile as solvent. The prepared composites is further characterized to confirm the formation of composites by FTIR and surface morphology was carried out by SEM. Ac conductivity shows that 50 wt% has high conductivity due to ionic polarization and therefore it has low dielectric loss value.

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