Studies on structural and electrical properties of NaI doped PEO/CMC blend solid polymer electrolyte

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Abstract
Solid polymer blend electrolyte (SPE) films of Polyethylene oxide(PEO)/Carboxymethyl cellulose (NaCMC) doped with Sodium iodide (NaI), were prepared by solution cast method and their structural, optical and electrical properties were investigated using X-Ray diffraction, Fourier transform infrared spectroscopy, UV–Vis spectrophotometer and Electrical impedance analyser respectively. X-Ray diffraction spectra indicate that, deformation occurred in the structure of the blend followed by doping with NaI salt. The variation in intensity and shifting of characteristic bands in FTIR spectra provides the evidence for formation of complex due to the interaction occurred in the blend with the dopant. Optical energy band gap decreases in the blend samples upon doping and is confirmed by UV–Vis spectrophotometer analysis. Increase of dielectric constant and loss upon doping, exhibits usual behaviour of the polymer electrolytes. An increase in AC conductivity is observed upon doping and maximum conductivity of $9.41 \times 10^{-3}$ Scm$^{-1}$ is obtained for 10/90 sample doped with 0.03% Sodium iodide and DC conductivity for the same sample at room temperature is $1.05 \times 10^{-2}$ Scm$^{-1}$. In addition, I-T studies confirm the conductivity exhibited by the sample as due to ions and I-V studies show electrochemical stability in the range of good working potential. From the results obtained, it can be inferred that, the prepared sample can be used as polymer electrolytes in the manufacturing of energy storage devices.

Keywords Polymer blend · Interaction · Dielectric constant · AC Conductivity · Energy band gap

Introduction
Solid polymer blend electrolytes attracted many researchers due to their remarkable potential applications in the preparation of electrochemical devices such as high-density rechargeable batteries, fuel cells, super capacitors etc. [1]. Though the solid polymer electrolytes (SPE) have more applications over liquid or gel electrolytes in view of their good processability, high flexibility and strengthened resistance to volume change of the electrodes during the charge/discharge process [2], the main problem of SPEs is their poor room-temperature ionic conductivity ($10^{-5} – 10^{-7}$ Scm$^{-1}$) [3]. Numerous work has been carried out towards the preparation of highly potential solid polymer electrolyte materials. Among them, Polyethylene oxide based materials are the most familiar polymers due to the presence of etheric oxygen as Lewis base, promotes good dissolution of salt and serving to coordinate cations facilitating good ionic conductivity which is an important requisite for polymer electrolyte. Polyethylene oxide is associated with repeated oxoethylene group $-CH_2-CH_2-O-$ in which the polar group $-O-$ has ability to associate with metal ions. Polyethylene oxide shows high stability for chemical and electrochemical reactions due to the presence of $C-C$, $C-O$ and $C-H$ bonds [4]. However, in spite of these, Polyethylene oxide possesses higher degree of crystalline nature which
restricts to achieve higher ionic conductivity. One of the main requisites to prepare polymer electrode is to provide amorphous rich region in the material. In this context, various methods have been introduced to enhance the degree of amorphous nature in polymer. Among them, polymer blending is found to be a fruitful one because of its simplicity, low cost, environmental friendly, nontoxic, easiest processing etc., by maintaining its required physical property, flexibility and mechanical stability [5]. Therefore, an attempt has been made in the present study to enhance the amorphous nature of the Polyethylene oxide by blending it with Carboxymethyl cellulose and hence to fabricate the polymer electrolyte of good conductivity.

Carboxymethyl cellulose is a bio polymer derived from naturally abundant cellulose. Huge number of carboxylic groups (CH$_2$COONa) have been introduced into cellulose molecule by facilitating the reaction with sodium hydroxide and chloroacetic acid [6]. Carboxymethyl cellulose has typical properties such as film forming abilities, good mechanical strength and high flexibility. It is used in various products like creams, lotions and toothpaste formulation as it possesses good binding, thickening and stabilizing properties. It is widely used in improving moisturizing effect in cosmetic products due to its film forming abilities. Apart from these it has been considered as eco-friendly due to its biodegradable nature [7].

Polyethylene oxide based polymer blends complexed with alkali metal salts have been studied and reviewed by many workers [8–12]. From these surveys, it can be seen that, alkali metal salts namely, Li$^+$ and Na$^+$ play an important role in the development of polymer electrolytes. In the present paper we are reporting the results obtained from the study of new polymer blend electrolyte made with Polyethylene oxide and Carboxymethyl cellulose doped with sodium iodide for Na$^+$ conduction.

Materials and methods

Polyethylene oxide (PEO) with 200,000 molecular weight from Sigma Aldrich and Carboxymethyl cellulose (Na-CMC) with average molecular weight of 295,188 g/mol (calculated by Mark-Houwink Sakudara equation $[\eta] = kM_w^\alpha$, where $[\eta]$ is intrinsic viscosity, $k$ is $5.37 \times 10^{-4}$ and $\alpha=0.73$ are Mark-Houwink constants for CMC in water solvent) from SD Fine-Chem Ltd were procured. Specific wt% of PEO and Na-CMC were dissolved in doubly distilled water taken as solvent by stirring until homogeneous solution was formed. Sodium iodide (NaI) salt with 0.01, 0.02 and 0.03% weight fractions were added to the homogeneous polymeric solution and stirred vigorously for two hours and ensured the complete mixing so that homogeneous solution was formed. The solution obtained was casted on serially labelled and cleaned petri dishes. Further, these were dried in an incubator at a temperature slightly greater than room temperature for two days and then peeled off carefully, placed in desiccator to avoid moisture absorption for further characterizations.

Experiments

X-Ray diffraction scan was performed for the blend samples irradiating Cu-K$\alpha$ radiation with wavelength $\lambda = 1.5406$ Å using X-Ray diffractometer (MiniFlex II, Rigaku Corporation, Japan). FTIR characterization was carried out in the wavenumber range 4000-400 cm$^{-1}$ using Fourier Transform Infra-red spectrometer [spectrum two SN: 94012]. Optical energy band gaps were obtained by measuring absorbance using computerised double beam UV–Visible spectrophotometer (Perkin-Elmer lambda-35) in the wavelength range of 200-1100 nm. Dielectric property and AC conductivity were obtained by measuring parallel capacitance ($C_p$) and energy dissipation factor (tan $\delta$) using electrical impedance analyser (Wayne Kerr precision impedance analyser 6500B) in the frequency range 50 Hz–1 MHz. Using these data, electrical impedance and hence DC conductivity was measured. Further, the electrochemical stability window of samples was measured using current and voltage plot. In addition, the transport number was measured using Wagner DC polarization technique.

Results and discussion

Structural characteristics–X-ray diffraction studies

X-Ray diffraction is an important tool used to investigate the structure, phase, particle size analysis etc., of the sample. The degree of amorphous nature contributes the ionic conductivity of polymer electrolyte has also been studied by X-Ray diffraction. Since, CMC is having higher degree of amorphous nature compared to PEO, the blend consisting of higher wt% of CMC is expected to exhibit a broad hump representing amorphous nature. From Fig. 1a–c it can be noticed that, on decreasing wt% of CMC in the blend, the broad hump gradually disappears followed by the appearance of two new peaks at 19.22 and 23.43 degrees corresponding to characteristic peaks of PEO, originating from ordering of polyether side chains and strong intermolecular attraction between PEO chains through hydrogen bonding [13]. This confirms the interaction of PEO and CMC due to the formation of hydrogen bond upon blending. Hence 10/90 composition shows broad hump and new small peaks are formed at 50/50 composition. The intensity of these peaks reaches maximum for 90/10 composition of the blend.

The interaction of NaI with PEO/CMC blend can also be understood from the same figure. NaI is an inorganic metal
consisting of several diffraction peaks representing highly crystalline structure as reported in literature [14]. For the blend of 10/90 composition, the increase in NaI concentration leads to the formation of new peak in the diffraction pattern and this peak is appeared to be shifted towards lower 2θ value indicating the interaction of NaI with the blend. In 50/50 composition, two small sub peaks located at 23.08 and 23.35 in the pure blend without doping, become well distinguished for 0.01% and 0.02% and finally merged for 0.03% NaI doping. This represents change in structure which in turn signifies the interaction between NaI and polymer blend. Similar changes can be seen for 90/10 composition and in addition, the intensity of the peak at 19.22 gradually increases upon doping. Hence, the changes occurred in diffraction pattern for 10/90, 50/50 and 90/10 composition represent the interaction between the blend and the dopant NaI.

**Chemical structure and composition–FTIR spectroscopy**

The characteristic peaks related to PEO and CMC of the samples studied are evident from the FTIR spectra as shown in Fig. 2a–c. From the literature, it is observed that, pure NaI has characteristic peak at around 1620 cm\(^{-1}\). Upon doping of NaI in the blend of 10/90 composition, the characteristic peaks appeared and located at 1652 cm\(^{-1}\), 1657 cm\(^{-1}\) and 1659 cm\(^{-1}\) for 0.01%, 0.02% and 0.03% respectively with increase in the intensity. The peak located at 2887 cm\(^{-1}\) belonging to PEO/CMC representing CH symmetric stretching, splits into two peaks and located at 2884 cm\(^{-1}\)
1463 cm$^{-1}$, 2857 cm$^{-1}$ and 2917 cm$^{-1}$ upon doping with 0.01%, 0.02% and 0.03% of NaI. These additional peaks are corresponding to NaI diffraction peaks. The characteristic peak at 1463 cm$^{-1}$, gradually disappears representing CH$_2$ bending corresponding to PEO vanishes upon doping. In 50/50 composition, the peak appeared at 2856 cm$^{-1}$ is shifted to 2860 cm$^{-1}$, 2858 cm$^{-1}$ and 2860 cm$^{-1}$ upon doping with 0.01%, 0.02% and 0.03% of NaI respectively. The intensity of characteristic peak at 1463 cm$^{-1}$ representing CH$_2$ bending belonging to PEO, changes upon doping. This ensures the interaction between the blend and dopant. In 90/10 composition, the peak located at 2867 cm$^{-1}$ also shifted with the formation of new peak just beside upon doping and the separation between pre-existing and newly formed peaks are as shown in Fig. 2c. The very small peak located at 1359 cm$^{-1}$ for undoped blend representing CH$_3$ bending in PEO will become clearly observable upon doping. These changes occurred ensure the interaction between the blend and NaI. In all the compositions of the blend, the band representing hydrogen bond also changes with values and intensities and is attributed to the traces of moisture present in the NaI salt due to water impurities.

**Optical energy band gap**

Electronic transitions in polymers can be of two types namely direct and indirect transitions. The direct transition of electron is observed when the wave vector is zero (K = 0) i.e, the bottom of conduction band is exactly above the top of valence band and therefore energy and momentum are conserved. Whereas, indirect transition is observed when
The wave vector is nonzero \( (K \neq 0) \) i.e., the bottom of conduction band is not exactly above the top of valence band. However, several reports have suggested that, indirect transitions are involved in studied polymer blends [15–17].

The main purpose of studying the optical property of PEO/CMC blend before and after doping with NaI is to investigate the effect of the latter on the variation of optical energy band gap. It is evident from Fig. 3a–c that, the optical energy band gap is found to decrease gradually upon increasing the concentration of NaI. The optical energy band gap in 10/90 composition was found to increase from 4.82 eV to 4.97 eV upon doping with 0.01% NaI, whereas, its value found to decrease with further increase of dopant concentration and is found to be 4.63 eV and 4.53 eV respectively for 0.02% and 0.03% NaI. For 50/50 composition, it becomes 4.76 eV, 4.69 eV, 4.42 eV and 4.33 eV for pure, 0.01%, 0.02% and 0.03% of NaI respectively. Similarly for 90/10 composition, it becomes 5.09 eV, 4.99 eV, 4.66 eV and 4.40 eV for pure, 0.01%, 0.02% and 0.03% of NaI respectively. From these results, it can be seen that, optical band gap value increases slightly for 0.01% of dopant in 10/90 composition, later it decreases for 0.02% and 0.03% of dopant. Further, in 50/50 and 90/10 composition, optical energy band gap gradually decreases with the increase of NaI concentration. Therefore, it is evident that, increase of NaI concentration decreases the optical energy band gap and thus influences the optical conductivity. As the optical energy band gap is the minimum energy that is needed to excite the electron from valence band to conduction band, in the opposite sense, transition of electron to valence band results in the recombination process with hole and hence emits the energy difference in the form of photon. On the other hand, it may also be considered that, the material with wide optical energy band gap emits high energetic photons possessing lower wavelength. Since the obtained energy band gap is greater than 4 eV, these materials can be used in the fabrication of optoelectronic devices such as green & blue LED and laser. Because of their higher frequency, such materials may also be used in radio frequency applications notably in military radars.

### Dielectric constant and dielectric loss

The dielectric constant and dielectric loss of varied compositions of PEO/CMC blend with and without NaI doping at room temperature were investigated over wide range of frequency between 50 Hz-1 MHz using the following relations.

\[
\varepsilon' = \frac{C_p d}{\varepsilon_0 A}
\]  
\[
\varepsilon'' = \varepsilon'\tan\delta
\]

where, \( C_p \) is the parallel capacitance and \( \tan\delta \) is the dissipation factor which were obtained by electrical impedance spectroscope, \( d \) is the thickness of sample and \( A \) is the area of cross section of the electrode. The study of Dielectric constant and loss provides the information regarding the polarization effect at electrode–electrolyte interface and it helps to understand the electrical conductivity of the polymer.

![Fig. 3](image-url)  

*Fig. 3* The plot of optical energy band gap of PEO/Na-CMC polymer blend with and without doping for the composition a 10/90, b 50/50 and c 90/10.
electrolyte [18]. In the present study, the variation of dielectric constant and dielectric loss is frequency dependant and hence gives the evidence for space-charge polarisation. It is evident from Fig. 4a–f that, dielectric properties of the samples are high in low frequency and slightly decrease in intermediate frequency range. High value of dielectric constant and hence dielectric loss in low frequency region signifies the presence of electrode polarization and space-charge effect [19]. Meanwhile, these were found to decrease exponentially in higher frequency region and this may be due to the fact that, in higher frequency region dipoles do not have enough time to align themselves in the direction of applied alternative electric field. This result in decreasing of polarization and hence dielectric constant in intermediate frequency region and attains saturation in higher frequency range [20–23].

It is also evident from the figures that the dielectric constant and dielectric loss for PEO/CMC blends with different compositions increases with increasing concentration of NaI for the entire blend compositions and may be attributed to the arising of space charge polarization with increase in number of charge carriers due to higher concentration of doping. Thus, high value of dielectric constant was observed for all the blend compositions when doped with 0.03% of NaI.

AC conductivity

The AC conductivity of the blends was evaluated using the following relation from the values of dielectric constant (\(\varepsilon'\)) and dissipation energy factor (\(\tan \delta\)) which were measured by Electrical impedance spectroscopy.

\[
\sigma_{ac} = \varepsilon' \varepsilon_0 \omega \tan \delta
\]

where \(\varepsilon_0\) is permittivity of free space and \(\omega\) is angular frequency of applied electric field. The frequency dependant AC conductivity for the blend with and without doping for each blend composition can be seen from Fig. 5a–c. It is evident that, two distinct regions namely plateau (frequency independent) region at low frequency range and dispersion region at high frequency range are observed for all the samples. Similar behaviour is also observed for few polymeric samples [24]. The plateau region is due to the low conductivity in lower frequency region that could be attributed to the accumulation of charge carriers at the electrode–electrolyte interface and space-charge polarization at the blocking electrode. The dispersion of conductivity in higher frequency region is due to the increase of ionic conductivity associated with AC conductivity [25].

It can be noticed from the figures that, the maximum conductivity at each blend composition increases with doping concentration. Increasing concentration of dopant enhances the charge carrier density which in turn enhances the ionic conduction and hence increase in conductivity is observed. This has been as evidenced from the relation \(\Sigma n_i z_i \mu_i\), where \(n_i\), \(z_i\), and \(\mu_i\) are number of charge carriers, the ionic charge and the ionic mobility respectively.

This reveals that, doping of NaI with the blend is an effective technique to improve ion transport mechanism. The ion transportation mechanism takes place with the hopping of ion from one coordination site to another following with the creation of the vacancy in preoccupied site for new incoming ion. By this mechanism, the movement of ions can be expected to be coupled with the polymer chain segmental dynamics in solid polymer electrolyte [26–29].

Ionic conductivity- impedance analysis

The impedance analysis is an important and powerful method to investigate the ionic conductivity in solid polymer electrolyte samples and was carried out using complex impedance spectroscopy. In the present study the ionic conductivity was investigated by applying the electrical signal of 40 mV across the cell configuration SS|SPEiSS (SPE is sandwiched between two stainless steel electrodes) and the corresponding impedance plots were presented in terms of Cole–Cole plot and is as shown in Fig. 6a–c. The plot shows the variation of real component of complex impedance (\(Z'\)) along X axis and the imaginary component of it (\(Z''\)) along Y axis for 10/90, 50/50 and 90/10 compositions of PEO/Na-CMC blend for varied concentrations of NaI at different temperatures in the range of 303 K-323 K. In the plots, two well defined regions can be observed such that, a semicircle in higher frequency region and a spike with the angle of inclination less than 90° with real axis indicates inhomogeneous nature of electrode–electrolyte interface. In higher frequency region, the point of intersection of semicircle with real axis provides the value of bulk resistance (\(R_b\)) of polymer electrolyte sample and this point of intersection was determined in the present case by using ZSimpWin (version 3.21) software. The ionic conductivity of the polymer electrolyte was calculated using the relation

\[
\sigma = \frac{r}{R_b \Lambda}
\]

where, ‘t’ is thickness of sample in meter, ‘\(\Lambda\)’ is area of the sample in m² and ‘\(R_b\)’ is bulk resistance of solid polymer electrolyte sample [30].

From the figure it can be observed that, there will be a decrease in impedance with the increase of concentration of NaI. In the lower concentration, the onset of semicircle indicates the higher value of impedance. The semi circles are well defined, possessing low value of
Fig. 4 The plot of Dielectric constant a 10/90, b 50/50, c 90/10 and Dielectric loss of d 10/90, e 50/50, f 90/10 verses frequency for PEO/Na-CMC polymer blend with and without doping
impedance in higher concentration, indicating the higher ionic conductivity.

From the figure, it can be seen that, the 10/90 composition blend without dopant exhibits higher impedance and hence less ionic conductivity ($6.06 \times 10^{-3}$ Scm$^{-1}$). Further, with the increase of dopant concentration, the bulk resistance ($R_b$) decreases and this leads to the increase of conductivity. The same kind of result is observed for 50/50 and 90/10 composition. The polymer electrolyte sample of 10/90 composition doped with 0.03% exhibits higher value of ionic conductivity of $1.05 \times 10^{-2}$ Scm$^{-1}$.

**Ion transport number analysis (conductivity of SPE)**

This is the most convenient and widely used method as suggested by Wagner to analyse the conductivity exhibited either by electrons or ions in solid polymer electrolyte material. The conductivity analysis was carried out by measuring the ion-transport number using the DC polarization technique in which the polarization current was recorded with respect to time with specific time interval. The plot of current with time for PEO/Na-CMC blend for 10/90 composition doped with 0.03%
NaI is shown in Fig. 7. The transport number analysis was done specifically for this sample because of its highly conducting nature when compared to rest of the samples. Under biasing condition, where the voltage is below the decomposition potential of electrolyte and until the steady state is reached, both ions and electrons are in motion and hence the current is due to both ions and electrons. When the bias voltage is greater than decomposition potential of the electrolyte, the ions get accumulated across the oppositely charged electrodes and blocked at the electrode – electrolyte interface, leading to the polarization and hence in this state, the electrons are only contributing to the current. Therefore, the initial total current (due to ions and electrons) decreases with the increase of time due to the depletion of ionic species in the electrolyte and then becomes constant [31]. The total ionic transference number was then determined using the relation,

$$t_{ion} = 1 - \frac{I_f}{I_i}$$  \hspace{1cm} (5)$$

where $I_f$ is final current and $I_i$ is initial current. Meanwhile, the electronic tranference number was determined using the relation,

$$t_{electron} = 1 - t_{ion}$$ \hspace{1cm} (6)$$

From the figure, the transport number due to ions was found to be 0.9932 for the sample characterized.

**Electrochemical stability window**

The consistency in the potential of solid polymer electrolyte is an important necessary parameter in the study of energy storage devices. The voltage limit called the electrochemical stability window (ESW) can be determined using current – voltage behaviour of respective SPE sample. The results of

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**Fig. 6** Cole–Cole plot of PEO/Na-CMC blend with (a) 10/90 (b) 50/50 and (c) 90/10 composition with varied concentration of NaI dopant

**Fig. 7** I-T characteristics of PEO/NaCMC blend of 10/90 composition doped with 0.03% NaI

**Fig. 8** I-V characteristics of PEO/NaCMC blend of 10/90 composition doped with 0.03% NaI
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**Data availability** The authors hereby confirm that, no datasets were generated nor used, instead, the manuscript comprises only experimental results which can be made available upon readers request.

**Declarations**

**Competing interests** The authors have no relevant financial or non-financial interests to disclose.

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**Table 1** The electrochemical stability potential of PEO/Na-CMC blend with varied composition of NaI

| Sample     | ESW   |
|------------|-------|
| 10/90      | 2.83  |
| 10/90+0.01%| 2.18  |
| 10/90+0.02%| 2.106 |
| 10/90+0.03%| 2.34  |

I-V characteristics of the samples of the polymer blend composition with different dopant concentration are shown in Fig. 8. For the purpose of characterization, the SPE samples were sandwiched between two stainless steel (SS) electrodes in the cell configuration of SS|SPE|SS. The voltage stability window was determined by the point of intercept on the X-axis in the region of sudden rise of current. The experimentally determined values of ESW have been tabulated in Table 1. These values are on an average in good working potential range and hence the samples can be used in the preparation of Sodium batteries [32].

**Conclusion**

The solid polymer electrolyte of PEO/Na-CMC blend with varied compositions and doped with different concentration of NaI was prepared using solution cast method. X-Ray diffraction studies reveal the variation of intensity and formation of new peaks upon doping. FTIR spectra showed the formation of complex between the dopant and blend. Optical studies revealed that, the optical energy band decreases with increase of dopant concentration. Since, these blend sample materials are found to exhibit higher optical energy gap, these may be used in the fabrication of optoelectronic devices. In addition, the dielectric constant and loss were found to decrease with increasing frequency and after reaching a minimum they get saturated. The ionic conductivity is found to increase upon increasing the concentration of dopant and maximum conductivity is observed with 10/90 composite sample doped with 0.03% NaI. The highest conductivity observed in this sample is greatly contributed from the ions. The I-V characteristic studies suggest that the prepared sample has good electrochemical stability potential. Hence, this sample can be used as polymer electrolytes in the manufacturing of energy storage devices.
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