

Physico-mechanical, AC-conductivity and microstructural properties of FeCl₃ doped HPMC polymer films

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(Received June 26, 2012, Revised October 12, 2012, Accepted November 21, 2012)

Abstract. The transition metal salt doped solid polymer electrolyte [TSPE] were prepared with HPMC as a host polymer. The virgin and doped films were prepared by solution-casting method and investigated using wide angle X-ray scattering method. Micro structural parameters like lattice strain (g%), stacking/twin faults, the average number of unit cells counted in a direction perpendicular to the Bragg's plane (hkl) spacing of (hkl) planes dhkl, crystallite size Ds, distortion width, standard deviation were determined by whole pattern powder fitting (WPPF) method, which is an extension of single order method. It is found that the crystallite size decreases with the increase in the content of FeCl₃. This decrease is due to increase in localized breaking of polymer network which also accounts for the amorphous nature of the material. The filler inorganic salt FeCl₃ acts as plasticizer. FTIR study also confirms and justifies the interaction between the polymer and in-organic salt in the matrix. Physical properties like mechanical stability and Ac conductivity in these films are in conformity with the X-ray results.

Keywords: doping; whole powder pattern fitting; AC conductivity; tensile strength; crystallite size

1. Introduction

Conducting polymers are a class of materials which conducts without being heated. The era of conducting polymers began with the invention of ionic conduction in Polyethylene based electrolyte in 1973 by Wright (Armand 1994). In recent years, there is an increased interest in the research activity in the conducting polymers to bring out new materials which are suitable for electronics, opto-electronics, and electrical devices (Mika 2007, Scrosati 1993). Use of polymers as conducting materials solves many problems such as cost, processability, leakage, power loss and fabrication, weight of the device, good mechanical properties (Rajendran 2002). Barring a few conducting polymers, majority of the polymers is non-conductors or having remarkably low conductivity. Blending two or three polymers to get a binary or ternary system or doping an inorganic salts or use of plasticizer, in the polymers and their blend matrix do change the

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conducting aspect of a polymer. This procedure inhibits more number of charge carriers and also provides a more flexible backbone for polymer network. HPMC is chosen in our study as a host polymer in the preparation of HPMC/FeCl₃ films, since not many reports of investigations are available. The results of the investigation do correlate AC conductivity, stability and microstructure details of these blends do suggest that there is indeed interesting results with regard to conducting polymers in keeping view of other social obligations like polymer doped with inorganic salts are degradable and environmental friendly.

2. Materials and methods

HPMC and FeCl₃ samples were purchased from Loba chem Mumbai. Pure and doped films were prepared by using solvent cast method (Tanabe 2002), (Guo 1989). HPMC (5%wt) dissolved in 100 ml of distilled water with continuous stirring. After complete dissolution the solution was filtered using filter paper to remove undissolved particles. FeCl₃ of different (%wt) 0.2 g-0.8 g dissolved in distilled water, and added to 100ml of 5% HPMC solution and stirred continuously using magnetic stirrer for 20 min to ensure uniform mixing of the solutions in the polymer matrix. Solution was allowed for a while and then it is poured on to the clean glass plate and allowed to dry for a week. After drying, the films were peeled out of the glass plate and stored in a desiccators to avoid moisture. The prepared films appears to be yellowish transparent. This is an indication that the inorganic salt FeCl₃ has fully dissolved creating a new polymer matrix constituted by one phase (Assessment 2000).

3. Experimental procedure

3.1 X-ray diffraction

X-ray diffraction recordings of the samples were carried out using Rigaku miniflex11 diffractometer with Ni filtered CuK α radiation of wavelength 1.5406 \AA , with graphite monochromatic. Samples were scanned in the 2θ range 6 $^{\circ}$ -40 $^{\circ}$ with a scan speed of 5 $^{\circ}$ /min and in step of 0.02 $^{\circ}$. Specifications used for recording are 30 kV and 15 mA. X-ray pattern of blend films contain both amorphous and crystalline region in the form of one broad band and other sharp peaks. Using in-house developed software each of the mentioned x-ray pattern of polymer blends are analyzed.

3.2 FTIR spectra

FTIR spectra of these films were recorded using Perkin-Elmer spectrometer [spectrum 65] with a resolution of 4 cm⁻¹. The spectra were obtained in the wave number range 400 cm⁻¹ to 4000 cm⁻¹.

3.3 Mechanical properties

Mechanical properties such as tensile strength, Young's Modulus, stiffness, percentage of elongation at break were measured using Universal testing machine (UTM).

3.4 UV-VIS spectra

UV-VIS spectra of all the films were recorded using UV-VIS spectrophotometer model 6080 in the wave length range of 200 nm-600 nm in air medium at room temperature.

3.5 AC conductivity measurements

AC conductivity measurements were made using Hiok (Japan) Model 3532-50 programmable computer interfaced digital LCR meter in the frequency range from 100 Hz to 5 MHz, at room temperature.

4. Results and discussions

4.1 X-ray diffraction studies

XRD Patterns of HPMC/FeCl₃ polymer electrolyte is shown in Fig. 1. there are two sharp peaks at 14°, 17° and a broad peak around 20° were observed in the XRD patterns of salt free HPMC, indicates semi crystalline nature of HPMC (Krumova 2000). In the salt added system sharp peaks disappear and only broad peak is observed. With increase in concentration of the salt broadness increases and there is a decrease in the intensity. This clearly shows amorphous nature of the HPMC film increases due to the reduction in the crystallinity of the film. Essentially due to the breaking of the bonds and hence the polymer network which results in disruption of the semi crystalline structure of the film by FeCl₃. Hodge *et al.* (1996) have established a correlation between the intensity of the peak and degree of crystallinity. They observe that the intensity of XRD pattern decreases as the amorphous nature increases with the addition of salt. Amorphous nature results in greater ionic diffusivity with high ionic conductivity, as amorphous polymer have flexible backbone (Mohammed 2003). No new peaks corresponding to FeCl₃ have been observed which indicates a complete dissociation of salt in the polymer matrix. The calculated values of

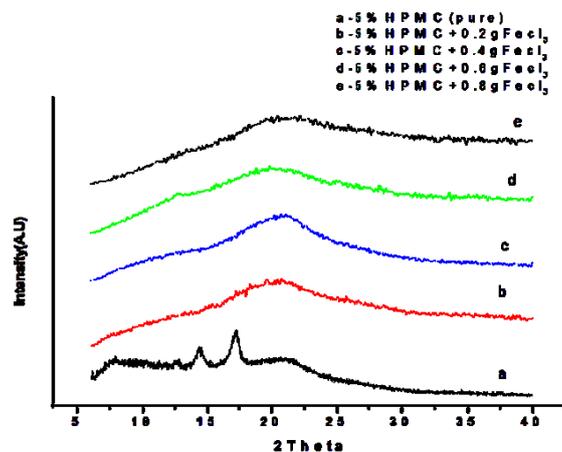
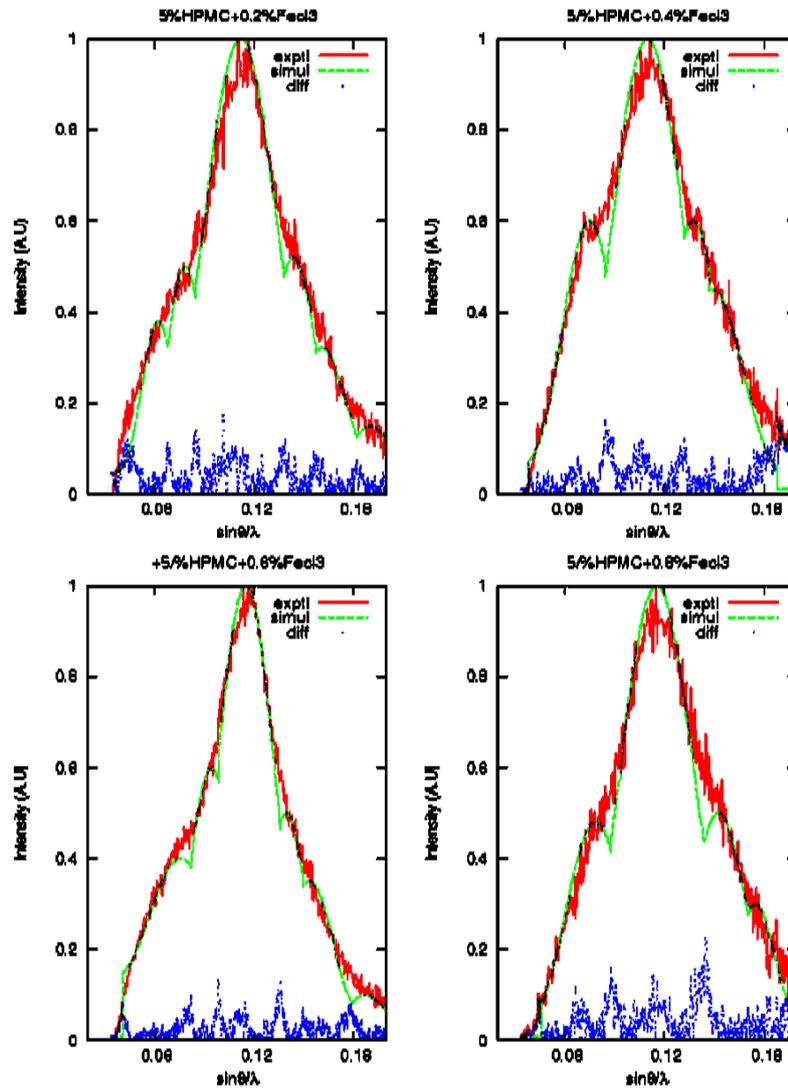


Fig. 1 XRD Pattern of HPMC and FeCl₃ doped HPMC films

Table 1 Microstructural parameters of FeCl₃ doped HPMC films using Whole powder pattern fitting method

| Sample | Din Å° | N | G(%) | α | DS = Nx | αd | β | Δ |
|--------------------------------|--------|------|------|----------|---------|------------|----------|----------|
| HPMC | 4.27 | 4.75 | 0.1 | 4.00 | 20.28 | 1.93E-06 | 4.57E-05 | 0.09 |
| HPMC + 0.2 g FeCl ₃ | 4.33 | 2.83 | 0.1 | 8.32 | 12.25 | 8.21E-09 | 2.18E-07 | 1.14E-03 |
| HPMC + 0.4 g FeCl ₃ | 4.52 | 2.20 | 0.0 | 8.25 | 9.94 | 2.48E-08 | 1.44E-05 | 1.59E-03 |
| HPMC + 0.6 g FeCl ₃ | 4.45 | 2.23 | 0.1 | 4.13 | 9.92 | 1.30E-07 | 2.19E-07 | 9.98E-04 |
| HPMC + 0.8 g FeCl ₃ | 4.21 | 1.86 | 0.1 | 5.91 | 7.83 | 2.61E-07 | 2.40E-04 | 1.08E-03 |

Fig. 2 Whole pattern fitting for pure HPMC and FeCl₃ doped HPMC films

crystallite size from line profile analysis (Hall 1991, Somashekar 1989, Divakara 2009, Souza 2006) were listed in Table 1. These values indicate that there is a decrease in the crystallite size as the concentration of FeCl_3 increases. Obtained data from the line profile analysis were further used in the refinement by whole powder pattern fitting method. The goodness of the fit was appreciated in Fig. 2.

4.2 Fourier transform infrared spectroscopic studies

IR Spectra is an important tool in understanding the structure and purity of a compound. It provides information about the complex and interaction between the different constituents of the blend matrix. FTIR spectra of the starting material HPMC and doped HPMC films were given in Fig. 3. Several changes were observed after comparing the spectrum of doped HPMC films with that of the pure films. The absorption band 3367 cm^{-1} observed in pure films which is due to intermolecular Hydrogen bonded O-H stretching frequency, shifted to 3350 cm^{-1} , 3339 cm^{-1} , 3340 cm^{-1} , in the 0.2 g, 0.4 g, 0.8 g FeCl_3 doped HPMC films respectively. The O-H bend of -CH₂ in pure HPMC shows an absorption band at 2910.55 cm^{-1} is now shifted to 2904.5 cm^{-1} , 2907.55 cm^{-1} , 2907 cm^{-1} and 2910.5 cm^{-1} . The C-H bend of -CH₂ in pure HPMC exhibited absorption at 1455.98 cm^{-1} , has shifted to 1455 in 0.2 g FeCl_3 doped HPMC films and cannot be observed in other samples of higher concentration of FeCl_3 . C-O stretching occurs at 1096.5 cm^{-1} ; in pure film has now shifted to 1101 cm^{-1} , 1190.5 cm^{-1} , 1190.5 cm^{-1} and 1183.5 cm^{-1} in the blended films respectively. The peak pertaining to C-Cl is observed in the complex films only at 850 cm^{-1} . C-Cl stretching exhibits change in its intensity behavior with changing the concentration of FeCl_3 . Absorption band appeared at 948.5 cm^{-1} due to O-H bond in pure film has shifted to 947.0 cm^{-1} in 0.2 g FeCl_3 doped films and disappears in other blended films of higher concentration. From the above analysis we assert that indeed there is a complex formation in polymer blends.

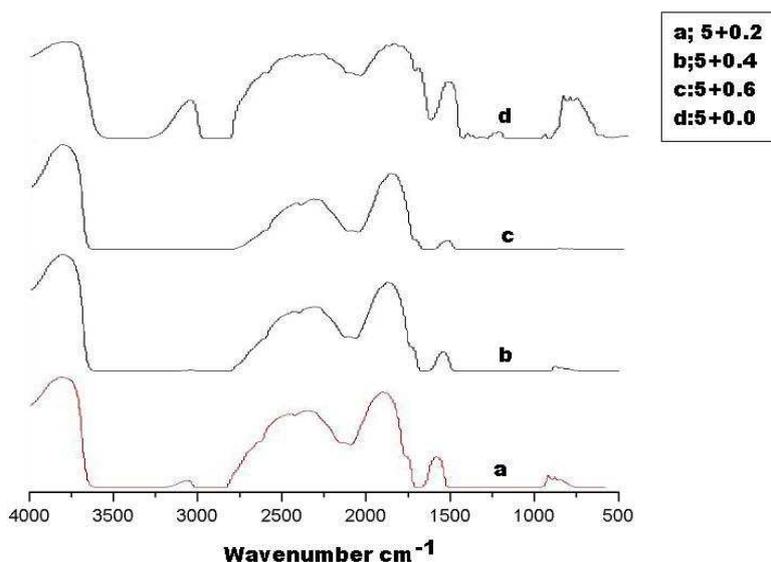


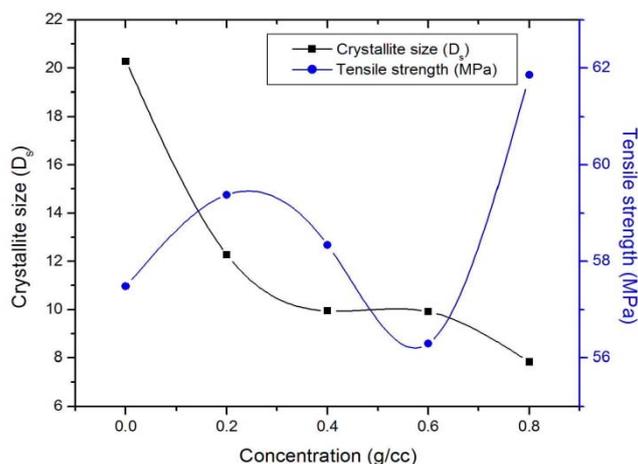
Fig. 3 FTIR of HPMC and FeCl_3 doped HPMC films

Table 2 Spectroscopic data of HPMC and FeCl₃ doped HPMC Films

| 5%HPMC | 5%HPMC + 0.2 g FeCl ₃ | 5%HPMC + 0.4g FeCl ₃ | 5%HPMC + 0.6 g FeCl ₃ | 5%HPMC + 0.8 g FeCl ₃ | correlation |
|---------|----------------------------------|---------------------------------|----------------------------------|----------------------------------|-----------------|
| 3367 | 3350 | 3339 | 3339 | 3340.5 | O-H stretch |
| 2910.55 | 2904.5 | 2907.5 | 2907.5 | 2010.5 | C-H Stretch |
| 2055.48 | 2059.28 | 2059.56 | 2059.26 | 2050.69 | C-H Stretch |
| 1611.94 | 1651.50 | 1646. | 1646 | 1644.5 | -C = C- Stretch |
| 1455.98 | 1455 | — | — | — | C-H bend |
| 1375.65 | 1354 | — | — | — | C-H rock |
| 1096.50 | 1101 | 1190.5 | 1190.5 | 1183.5 | C-O Stretch |
| 948.50 | 947 | — | — | — | O-H bend |
| 850.98 | 850.99 | 850.76 | 850.76 | 851.27 | C-Cl Stretch |

Table 3 Mechanical properties of HPMC and FeCl₃ doped HPMC films

| Sample | Tensile strength [MPa] | Young's modulus [MPa] | Stiffness | %of Elongation at break |
|-----------------------------------|------------------------|-----------------------|-----------|-------------------------|
| 5% HPMC | 57.48 | 1687.08 | 93.72 | 5.80 |
| 5% HPMC + 0.2 g FeCl ₃ | 59.38 | 1840.67 | 81.80 | 5.905 |
| 5%HPMC + 0.4 g FeCl ₃ | 58.29 | 2003.54 | 89.04 | 4.33 |
| 5%HPMC + 0.6 g FeCl ₃ | 56.29 | 2879.77 | 127.99 | 3.56 |
| 5%HPMC + 0.8 g FeCl ₃ | 64.50 | 3867.80 | 171.90 | 13.17 |

Fig. 4 Mechanical properties of HPMC and FeCl₃ doped HPMC films

4.3 Mechanical properties

Mechanical properties such as tensile strength (TS), Young's Modulus (σ), stiffness (S), % elongation at break (Eb) have the following behavior. Tensile strength decreases first and then increases as the concentration of FeCl₃ increases in the HPMC matrix. Tensile strength of the films was calculated by dividing the maximum load for break the film by cross sectional area. Young's

Modulus is the ratio of stress to strain at the linear portion of the curve, or slope of the linear portion of the curve of the stress strain. Elongation at break was calculated by the ratio of the film elongation at rupture to initial gauge length and multiplied by 100. The XRD analysis suggests that increase in flexible backbone which supports the increase of mechanical properties of the films. Table 3 shows mechanical properties of blends. Fig. 4 clearly shows the variation of Tensile strength and crystallite size with concentration of the salt in HPMC films. Fig. 5 shows the variation of Young's modulus with concentration of salt.

4.4 UV-VIS absorption spectraanalysis

Optical absorption is a useful method for investigating the induced transition and providing information about the band structure and optical energy gap in the material. The energies of the orbital's involved in electronic transitions have fixed values and as energy is quantized. Valance electrons can generally found in any one of these electron orbitals, single or σ , π , and n -orbitals (non bond). When an electromagnetic radiation of suitable frequency is absorbed, a transition occurs from one of these orbitals to an empty orbital, usually an anti bonding orbital π^* , σ^* . The exact energy difference between the orbitals depends on the atoms present and the nature of the bonding system. The electronic transition that are found in the visible and Ultraviolet region are $\sigma \rightarrow \sigma^*$, $n \rightarrow \pi^*$, and $\pi \rightarrow \pi^*$. The optical transitions results from the presence of impurities have energies in the visible part of the spectrum, and the defects are referred to as color centers (Hema 2008). The UV-VIS spectrophotometer scan of pure and FeCl_3 doped films were done in the wavelength range from 200 nm to 600 nm. The obtained spectra were shown in the Fig. 6. The observed spectra contains main absorption peak for all the films which shifts towards a longer wavelength side with the increase of concentration of FeCl_3 . The shift indicates that there is an interaction of in-organic salt with the polymer network and due to change in crystallinity.

4.4.1 Optical energy band gap

In-organic salt like FeCl_3 induces the Microstructural modification in the polymer and their

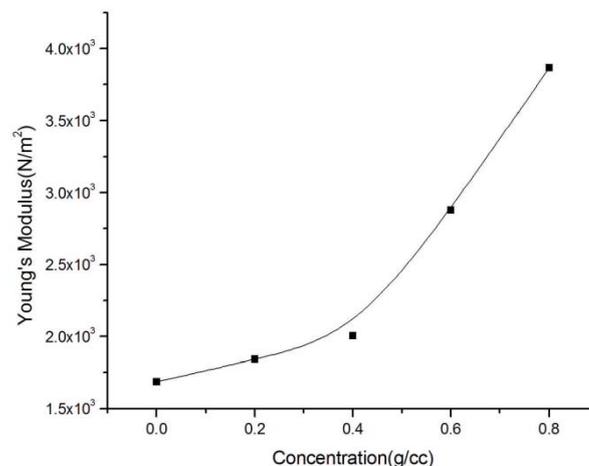


Fig. 5 Variation of Young's Modulus with concentration of FeCl_3 , in FeCl_3 doped HPMC film

blends, changes the polymer network structure which results in the optical band gap. Optical band gap can be calculated by plotting $(\alpha h\nu)^{1/2}$ against $h\nu$ (Srivastava 2000), Sangappa (2008) near absorption edge as shown in Fig. 7. By extrapolating the straight regions to x-axis corresponds to optical band gap. These values are given in Table 4. It is evident that energy gap decreases with concentration of.

4.5 Ac conductivity study

The measured conductance $G(\omega)$ from 50 Hz to 1 MHz was used to calculate AC conductivity $\sigma(\omega)$ using the following equation

$$\sigma(\omega) = G(\omega) d / A$$

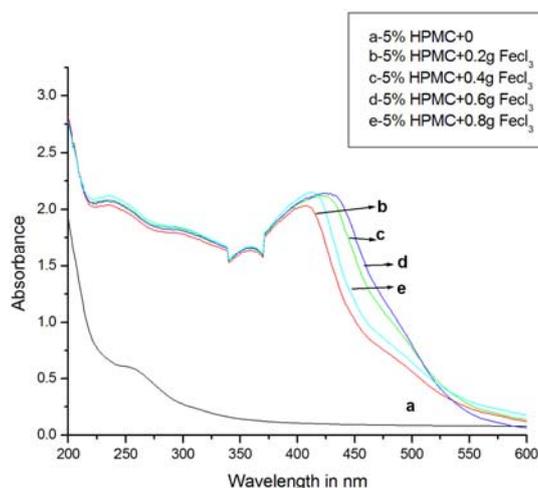


Fig. 6 UV-VIS spectra of pure HPMC and FeCl_3 doped HPMC films

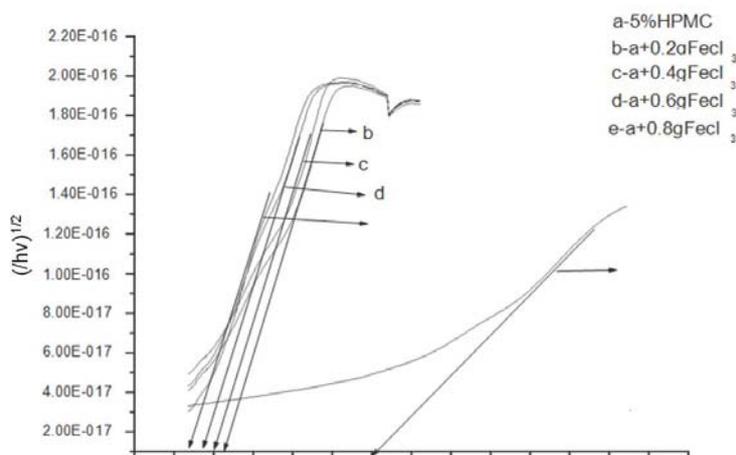


Fig. 7 plot of $(\alpha h\nu)^{1/2}$ against $h\nu$

Table 4 Optical energy band gap of HPMC and FeCl₃ doped HPMC films

| Sample | indirect energy gap in ev | direct energy gap in ev |
|------------------------------|---------------------------|-------------------------|
| HPMC+0 | 3.00 | 3.95 |
| HPMC +0.2gFeCl ₃ | 2.25 | 2.75 |
| HPMC +0.4g FeCl ₃ | 2.20 | 2.65 |
| HPMC +0.6g FeCl ₃ | 2.15 | 2.58 |
| HPMC +0.8g FeCl ₃ | 2.10 | 2.50 |

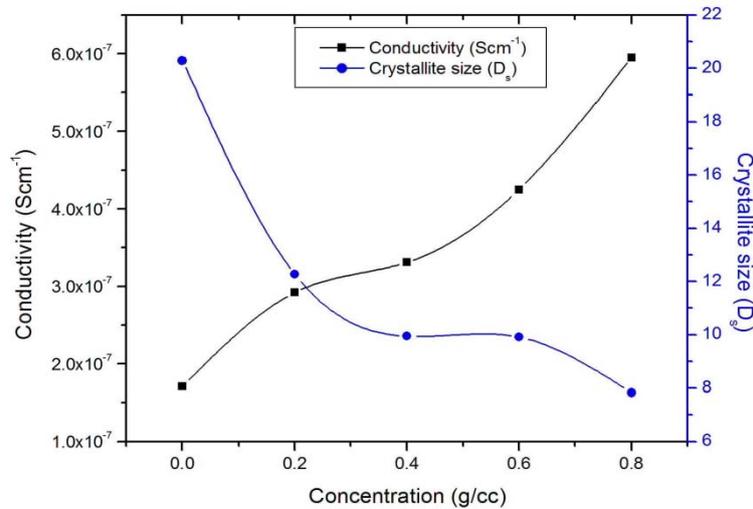


Fig. 8 Variation of AC conductivity and crystallite size with concentration of FeCl₃ in FeCl₃ doped HPMC films

where d is the thickness of the film, A is the area of cross-section. Fig. 7 shows the AC conductivity of HPMC/FeCl₃ doped films. Frequency dependence of AC conductivity is given in Figs. 11 and 12. It is observed that the conductivity increases with the frequency. Increase of conductivity with frequency and concentration is common for polymeric materials. There are two trends in the present polymer blends. The first one up to 104 Hz frequency independent conductivity due to free charges available in the doped films and for above 104 Hz frequency dependent conductivity due to the trapped charges which is active only in the higher frequency region. Fig. 8 shows the variation of conductivity and crystallite size as a function of concentration and Variation of conductivity as a function of concentration of FeCl₃ is given in Fig. 9. The average conductivity at room temperature for different concentrations of FeCl₃ is given in Table 4. It can be observed that the addition of FeCl₃ does significantly improve the ionic conductivity. Addition of salt into the polymer matrix increases the amorphous nature (Park 2009), leading to more flexible nature of the films. Flexibility increases the segmental motion of the polymer which increases the ionic mobility (Puteh 2005). Salt dissociation is an additional factor in enhancement of ionic conductivity. The dissociation of salt gives rise to more number of Fe³⁺ ions to the electrolyte. The pure HPMC has a conductivity 1.95E-07 Scm⁻¹ and it increases to 6.0E-07 Scm⁻¹ with the addition of FeCl₃. The highest conductivity of 6.0 E-07 Scm⁻¹ observed for 0.8 g FeCl₃ added HPMC film. We have made a comparison of conductivity of our sample with the

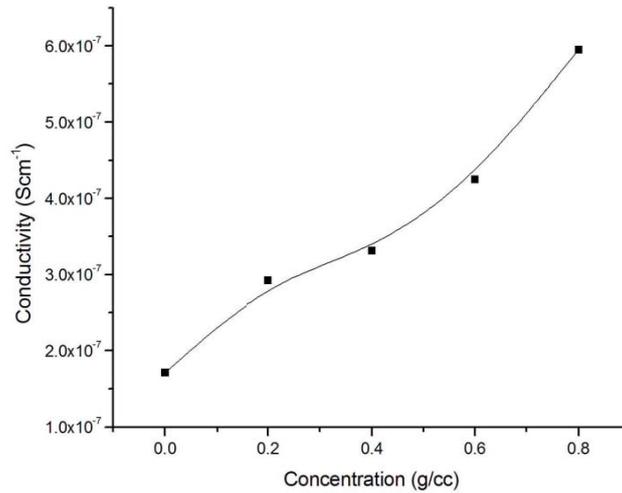


Fig. 9 Variation of AC conductivity with concentration of FeCl_3 , in FeCl_3 doped HPMC films

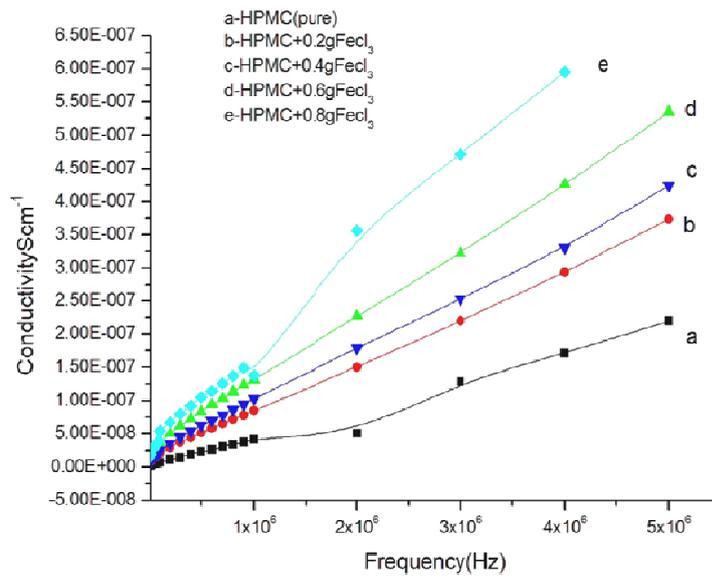


Fig. 10 AC conductivity of pure and FeCl_3 doped HPMC

Table 5 Comparison of AC conductivity of other polymer electrolyte with conductivity of our sample

| Sample | AC conductivity(Scm ⁻¹) | Reference |
|-------------------------------|-------------------------------------|---------------|
| PVA | 2×10^{-9} | (Balaji 2009) |
| PVA:NaF | 3.99×10^{-4} | (Balaji 2009) |
| PEO:PVP PEO:PVP:NaF | 5.7×10^{-4} | (Hema 2009) |
| PVA:N H4 Br PVA:N H4 I | 2.5×10^{-3} | (Kiran 2011) |
| Our Sample | 8.25×10^{-9} | (Hema 2009) |
| HPMC: FeCl_3 (0.8 g) | 1.19×10^{-7} | (Hema 2010) |
| | 6×10^{-7} | |

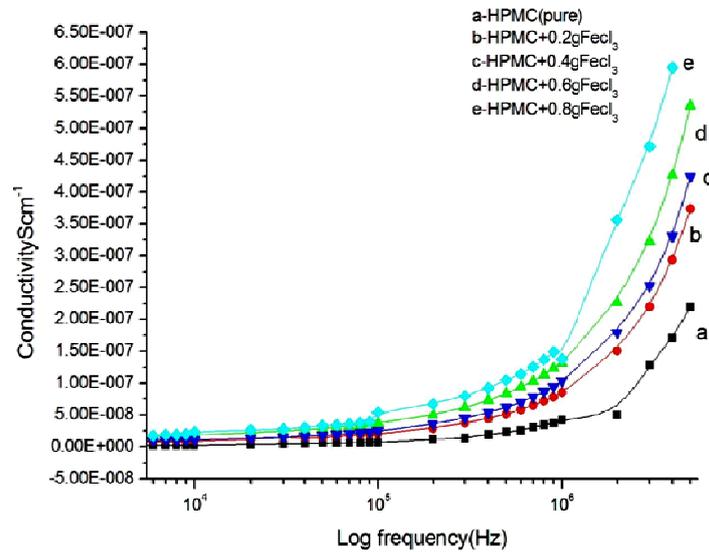


Fig. 11 Variation of log frequency with AC conductivity

conductivity of other polymers and their blends with various salts (Balaji 2009, Kiran 2011, Hema 2009, Hema 2008) available Table 5. This clearly indicates polymer blends here is a better candidate for conducting polymer. Another advantage is degradable. Using this polymer electrolyte in designing electrical components, one can reduce the problem of e-waste.

5. Conclusions

Extensive study on physical properties, FTIR, and AC conductivity of FeCl_3 doped HPMC films are carried out in this paper. In all the doped films AC conductivity increases as concentration of FeCl_3 increases. The doping of FeCl_3 in to HPMC destroys crystalline like order of HPMC and increases amorphous nature of films. FTIR spectra shows shift in some of the bands and decrease in the intensity of the peaks in doped films. The complex formation in the polymer matrix has been elucidated from XRD and FTIR Studies. The increase in conductivity in doped films has been well correlated with the increase in amorphous nature of the films. Increase in conductivity with frequency corresponds to more stability in films. HPMC polymer film doped with 0.8 g FeCl_3 has good mechanical strength and is desirable and promising candidate for the fabrication of degradable devices.

Acknowledgments

The corresponding author would like to acknowledge the following for supporting his research: Deanship of Scientific Research at Tafila Technical University (TTU), Ministry of Higher Education and Scientific Research, Jordan Atomic Energy Commission (JAEC), Abdul Hameed

Shoman Foundation, Jordan and the Middle East Desalination Research Center (MEDRC), Oman. The Editor in Chief and the reviewers of the AMR journal are greatly acknowledged to their valuable comments.

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