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Structural, Optical and Electrical conductivity Studies of Mn²⁺ Ions Doped PVA/MAA: EA Polymer Blend Films

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Abstract- Pure and different concentrations of Mn^{2+} (1.0, 2.0, 3.0, 4.0 and 5.0 mol%) doped PVA/MAA:EA polymer blend films were prepared by solution casting method. The structural analysis of the prepared samples by X-ray Diffraction reveals broadening of diffraction peak which implies a lower degree of crystallinity with higher Mn^{2+} concentration. UV-Vis spectral analysis shows a significant red shift in the optical band gap with increase in Mn^{2+} concentration. FTIR spectrum exhibits bands characteristic of stretching and bending vibrations O–H, C–H, C=C and C–O groups and the changes in the FTIR spectrum with dopant concentration indicate the complexation of dopant with the polymer blend. The conductivity study shows that the doping of Mn^{2+} ions into the polymer blend system enhances its ionic conductivity from 2.46 X 10^{-8} Scm⁻¹ for pure film to 1.41 X 10^{-7} Scm⁻¹ for 5 mol% of Mn^{2+} ions at room temperature, which is due to the increase in mobile charge carriers and their increased mobility which is explained in terms of an increase in the amorphicity.

Keywords- PVA/MAA:EA polymer blend, Mn doping, optical band gap, amorphicity and electrical conductivity.

I. INTRODUCTION

In the field of polymer or polymer blend additives, lot of research work has been carried out earlier towards the development and modification of innovative materials of significant importance. Transition metal ion doped polymers are considered as potential materials for both theoretical and experimental research and found to be essential because of their increasing technological applications. Doping polymer blends with transition metal ions have significant effect on their physical properties. Changes in physical properties of polymer blends are considered to be dependent on chemical nature of dopants and methods of preparation in which those interact with host matrices. An attention has been focused on the structural, thermal and optical properties of doped polymer blend films, since they possess potential properties useful in the advanced industrial applications [1, 2]. An important feature of polyvinyl alcohol (PVA) is the presence of crystalline and amorphous regions and its physical properties which are resulting from crystal amorphous interfacial effects [3], the presence of OH groups and the hydrogen bond formation. This explains various phenomena in electronics, optics and solid state physics. The copolymer Methacrylic Acid- Ethyl Acrylate (MAA:EA) has drawn researchers attention because of its good environmental stability, easy process ability and transparency.

Many studies have been reported in literature about the electrical conductivity and dielectric properties of polymer blends with regard to storage and dissipation of electric and magnetic energies in materials which explains numerous phenomena in optics, electronics and solid state physics. In order to store electric charges dielectrics are used whereas insulators are used to block flow of electric charges. Dopants are used with polymer blend for several reasons; improved processing, density control, optical effect, thermal conductivity, electrical and paramagnetic properties, etc. [4]. Mn is considered to be a good candidate for one or two dimensional phenomena and for optical memory devices [5, 6] based on the importance and relevance of the PVA/MAA:EA polymer blend as a suitable matrix for transition metal ion doping. To the best of our knowledge little systematic investigation has been carried out on metal/polymer composites.

Therefore, the present work is focused on the preparation and characterization e.g. structural, spectroscopic and electrical of PVA/MAA:EA polymer blend films doped with Manganese by the cost effective simple solution casting method. The as-prepared samples were analyzed by employing XRD, UV-Visible Spectrometer, FTIR and Impedance Spectroscopy to recognize the structure, optical band gap energy, position of functional groups, electrical conductivity and dielectric characteristics respectively.

Organization of this research paper has been done into five distinct sections: Introduction, Related work, Materials and Methods, Results with Discussion and Conclusions. Introduction section provides the brief review of literature related to our present investigation. Related work section elaborates on some work done by the various researchers on thermal, structural, optical and electrical studies of pure and doped PVA/MAA:EA polymer blend films. Materials and methods section contains the materials used for the preparation of Mn²⁺ ions doped PVA/MAA:EA polymer blend films, details of experimental procedure as well as the characterizations techniques employed for the analysis of prepared Mn²⁺ ions doped PVA/MAA:EA polymer blend films by solution casting method. Results and Discussion section provides a brief details on the various characterization of Mn^{2+} ions doped PVA/MAA:EA polymer blend films and the analysis. Conclusion section provides the major conclusions drawn from the results.

II. RELATED WORK

Some of the related works done by various researchers are Bajaj et al. 1994 [7] studied the thermal behaviour of MAA: EA copolymer. Wang et al. 2003 [8] studied the interaction between MAA:EA copolymer and bromide dopant. Madhava Kumar et al. 2016 [9, 10] studied the influence of Mn²⁺ incorporation on the structural, thermal, optical and electrical properties of MAA:EA copolymer films. Further to that, Siddaiah et al. 2018 [11] studied the thermal, structural, morphological and optical properties of PVA/MAA:EA polymer blend, reported that the (50:50) polymer blend composition is optimum and also studied the thermal, structural, optical and electrical properties of PVA/MAA:EA polymer blend filled with lithium Perchlorate [12].

III. EXPERIMENTAL

3.1 Preparation of the material

Pure and Mn^{2+} ions (1.0, 2.0, 3.0, 4.0 and 5.0 mol%) doped PVA/MAA:EA polymer blend films were prepared at room temperature by solution casting method. The polyvinyl alcohol (PVA) from Merck-Germany with molecular weight 14000 and Methacrylic Acid - Ethyl Acrylate copolymer (1:1) dispersion 30 percent having a mean relative molecular weight of about 250000 (Purchased from Merck Millipore India Ltd.) are water soluble polymers. Polyvinyl alcohol (PVA) polymer blended with MAA:EA copolymer in the concentration of 50:50 wt % (PVA:MAAEA) was prepared at room temperature by using distilled water. Five millilitres of PVA solution added to five millilitres of MAA: EA solution and then the solution was stirred magnetically for 10-12 hours to get a homogeneous mixture. The desired concentrations of MnSO₄ solution were prepared at room temperature by using distilled water. Then, different concentrations of (1.0, 2.0, 3.0, 4.0 and 5.0 mol %) dopant solution was added to the mixture solution. The resulting solution was magnetically stirred for 3-4 hours in order to get a homogeneous mixture and then cast onto polypropylene dishes. The solution was slow evaporated at RT to get the doped polymer blend films.

3.2 Characterization

In order to investigate the nature of the transition metal ion doped polymer blend films, X-ray diffraction measurements were carried out using a Siemens D5000 diffractometer with Cu K_a radiation ($\lambda = 1.5406$ Å). The films were scanned at 20 angles between 5 and 35° with a step size of 0.02°. UV-Vis absorption spectra of pure and doped films were recorded in the range 200-800 nm at room temperature using JASCO UV-Vis spectrophotometer (model- V.700). FTIR spectra of these films were recorded using a Perkin-Elmer FTIR spectrometer. The spectra were taken over a wavenumber range 500 – 4000 cm⁻¹. The impedance measurements were conducted using a computer controlled phase sensitive multimeter (PSM 1700) in the frequency range of 1 Hz–5 MHz at room temperature.

IV. RESULTS AND DISCUSSION

4.1 X-Ray Diffraction studies





The X-Ray diffraction analysis is used to determine the complexation and crystallization of the polymer matrix. Figure 1 shows the XRD patterns of the samples in the range of $2\theta = 5-35^{\circ}$. It is clear from the figure that, a broad diffraction peak was observed at $2\theta = 19.43^{\circ}$ and this may be attributed to the semi-crystalline nature of the blend [9]. It can also be observed that, compared to pure film the peak at $2\theta = 19.43^{\circ}$ has been increased in its broadness and decreased in its intensity with the gradual increase in dopant Mn^{2+} concentration [13]. This reveals the decrease of the degree of the films [14]. This may be attributed to the strong

interaction between polymer blend and Mn^{2+} ions, which results in the degree of intermolecular interaction, which implied a change in the degree of crystallization combined with increase in the amorphous regions. This behaviour demonstrates that the complexation between Mn^{2+} ions and the blend takes place in the amorphous region.

4.2 Ultraviolet and Visible studies

UV-Vis absorption spectra of prepared Mn^{2+} ions doped PVA/MAA:EA polymer blend films at room temperature in the wavelength range 200–800 nm are shown in Figure 2(A). It is well known that the fundamental absorption is due to the excitation of electron from the valence band to the conduction band, which can be used to determine the value of absorption edges and band gaps of the films to provide useful information about the band structure in both crystalline and non-crystalline state.



Figure 2. (A) Ultra Violet absorption edge, (B) α vs hυ plots and (c) $(\alpha h \upsilon)^{1/2}$ vs hυ plots of pure (a) and different concentrations 1.0 mol% (b), 2.0 mol% (c), 3.0 mol% (d), 4.0 mol% (e) and 5.0 mol% (f) of Mn²⁺ doped PVA/MAA: EA polymer blend films.

It is observed from Figure 2(A) that, there is an increase in absorbance for the polymer blend films due to doping, which may be attributed to electronic transition from the bonding molecular orbit. A hump or shoulder at 210 nm may be due to

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Samples	Absorption edge (eV)	Optical band gap energy (Indirect) (eV)
Pure(50-50)	5.14	5.01
1.0 mol%	5.08	4.95
2.0 mol%	5.06	4.91
3.0 mol%	5.01	4.85
4.0 mol%	4.90	4.76
5.0 mol%	4.72	4.51

Table 1. Absorption edge and optical band gap values of pure and

different concentrations of Mn²⁺ ions doped polymer blend films

 $n \rightarrow \pi^*$ electronic transitions [15, 16]. A broad band is observed at ~ 272 nm and it may be assigned to $\pi \rightarrow \pi^*$ transition [15, 16]. In the present system, the spectra of the Mn^{2+} ions doped PVA/MAA: EA polymer blend films exhibit a red shift for the sharp absorption edge toward longer wavelength with increase in Mn^{2+} content. This red shift for the absorption edge of the doped composite samples indicates the complexation between Mn^{2+} ions and the polymeric matrix. In addition, it indicates the change in the optical energy gap, which is caused due to the change in crystallinity within the polymer matrix of blend [15, 16], which was confirmed by XRD studies.

This means that the width of forbidden band decreases with increasing the Mn^{2+} ions content. The absorption coefficient $\alpha(\nu)$ can be obtained using the Beer–Lambert's relation [16]:

$$\alpha(\nu) = 2.303^* \, (A \,/\, d \,) \tag{1}$$

where A is the absorbance and d is the film thickness.

From the position of the absorption edge in the linear portions of α vs. hu curves as shown in Figure 2(B), it is observed that the absorption edge for pure film lies at 5.14 eV whereas for 1.0, 2.0, 3.0, 4.0 and 5.0 mol% Mn²⁺ doped PVA/MAA:EA polymer blend films, it lies at 5.08, 5.06, 5.01, 4.90 and 4.72 eV respectively.

For indirect transitions, interaction takes place with lattice vibrations (phonons); thus the wave vector of the electron can change in the optical transition and momentum change will be taken or given by of the electron can change in the optical transition and momentum change will be taken or given by phonons. In other words, if minimum of the conduction band lies in a different part of K-space from the maximum of the valence band, a direct optical transition from the top of the valence band to the bottom of the conduction band is forbidden. For indirect transition which requires phonon assistance, absorption coefficient has the following dependence on the photon energy [16, 17]:

$$(\alpha h v)^{1/2} = C (h v - E_{gi})$$
(2)

Where hv is the energy of phonon, E_{gi} is the indirect band gap associated with transition, *C* is the constant and α is the absorption coefficient. The indirect band gaps were obtained from the plots of square root of the product of absorption coefficient and photon energy versus photon energy as shown in Figure 2(C). For pure (PVA/MAA:EA) polymer blend film the indirect band gap lies at 5.01 eV while for doped films the values vary from 4.95 to 4.51 eV (Table 1). Optical absorption edge and optical band gap showed decreasing trend with increase in concentration, thus indicating that with increase in concentration of Mn²⁺, PVA/MAA:EA polymer blend film becomes more semiconducting in nature.

Figure 2(C) shows that, the band gap decreases with the increase in the dopant concentration. It can be explained that, the decrease in energy gap with increasing Mn²⁺ concentration is due to the mobility gap variation in the doped films. The calculated E_{gi} values of the films are summarized in Table 1. From this table, it is clear that these energy values are decreased in general with increasing Mn²⁺ ions content as compared with that of pure PVA/MAA:EA polymer blend. It is remarkable that these observations were in agreement with XRD analysis. This decrease in optical band gap may also be attributed to defects formation in the doped samples. These defects create localized states in the optical band gap and the density of these localized states increases with increase in dopant concentration resulting a decrease in the energy gap [18]. The decrease in energy gap value in Mn²⁺ ions doped PVA/MAA:EA polymer blend films makes them efficient materials for optoelectronic devices and electrochemical applications such as polymer electrolyte. This is due to the fact that such devices require the band gap tenability [19].

4.3 FTIR studies

The FTIR spectra for pure and Mn^{2+} (1.0, 2.0, 3.0, 4.0 and 5.0 mol%) doped PVA/MAA:EA polymer blend films are shown in Figure 3. The spectra exhibit bands characteristics of stretching, bending vibrations of O-H, C-H, C=O, CH₃-O and $CH_2+\alpha-CH_3$ groups. The observed FTIR band positions and their assignments are presented in Table 2. In pure film a strong band observed at 1452 cm⁻¹ has been assigned to bending mode of vibration corresponding to CH₃ group, while another strong band observed at 1492 cm⁻¹ has been attributed to C-H bending of CH₂ [20, 21]. The FTIR absorption bands attributed to carbonyl stretching of the acid and ester units have appeared as weak bands at 1601 cm⁻¹ range [17]. A broad band observed at 2923 cm⁻¹, arising from C-H stretching frequency shows the presence of methylene group [22]. Asymmetric C-H stretching of methyl groups give a peak at 3026 cm⁻¹ and C-H stretching of the methylene group gives two peaks at 3059 and 3082 m^{-1} [23]. The following changes in the spectral features have been observed for Mn²⁺ doped films. Compared to pure film, Mn²⁺ doped films do not exhibit any change in their band position but the intensity of bands varies considerably with varying Mn²⁺ concentration. The intensity of the band observed at 1601 cm⁻¹ which corresponds to C=O stretching of carboxylic acid, has increased with increasing Mn²⁻ content. The effect of dopant on the modes of vibration is observed in terms of increase in the intensity and broadening of the band, which results from the formation of cross links between the Mn²⁺ cations and oxygen atoms of carbonyl groups. This indicates increase in the basicity of C=O groups with the increased Mn²⁺ ion concentration, implying that the carbonyl group is able to act as a strong electron donor to interact with Mn²⁺ cation. This leads to decrease the crystallinity and increase the amorphous content [20]. In addition, small bands are observed at 1579 and 1857 cm^{-1} . This



Figure 3. FTIR spectra of pure (a) and different concentrations 1.0 mol% (b), 2.0 mol% (c), 3.0 mol% (d), 4.0 mol% (e) and 5.0 mol% (f) of Mn²⁺ doped PVA/MAA:EA polymer blend films.

indicates the transformation of crystalline regions of PVA/MAA:EA polymer blend into amorphous regions due to the addition of Mn^{2+} ions [20]. All these changes in the FTIR spectra are clear indication for the complexation of polymer blend [9]. The compatibility between the polymer matrix and the transition metal ion dopant has great influence on the properties (mechanical, thermal, optical, morphological) of the polymer blend films.

Table 2. FTIR peaks assignment of pure and different molar
concentrations of Mn ²⁺ doped PVA/MAA: EA polymer blend
films.

Wavenumber (cm ⁻¹)	Band assignment
1452	CH ₃ –O bending
1492	C–H bending of CH ₂
1579	C=C stretching

1601	C=O stretching
1857	C–H stretching
1937	C–O stretching
1942	C–O stretching
2923	C–H stretching of methylene group
3026	C–H stretching methyl group
3059	C–H stretching methylene group
3082	C–H stretching methylene group

4.4. Electrical studies

4.4.1 AC conductivity studies

AC impedance spectroscopy is an important characterization technique to study ionic conductivity of doped polymer blend films. Figure 4 shows an impedance plot between pure and different concentration of Mn²⁺ doped PVA/MAA:EA polymer blend films at room temperatures in the frequency range of 1Hz - 5MHz. It is observed that the idealized impedance plane plots of Z^{II} as a function of Z^{I} , i.e. Cole – Cole plots of the films (Z' and Z'' denote the real andimaginary parts of the complex impedance Z*), contain a semi circular arc and inclined spike, both of which are characteristic behaviour of ionic conductivity of solids with blocking electrodes [24]. The semicircle indicates the parallel combination of bulk resistance (due to the migration of ions) and bulk capacitance (due to the immobile polymer chains). Hence the frequency



Figure 4. Nyquist impedance plots of pure and different concentrations of Mn^{2+} doped PVA/MAA:EA polymer blend films at room-temperature.

response of the given sample could be represented by an equivalent circuit consisting of a parallel combination of the

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circuit elements R (resistance) and C (capacitance). The presence of the depressed semi circle reveals the non-Debye nature of the sample [25] due to the potential well for each site, through which the ion transport occurs. The inclined spike represents formation of double layer capacitance at the electrode–electrolyte interface due to the migration of ions at low frequency. The capacitance values are in the range of PF, which represents the bulk response of the sample [26]. The electrode double layer at each interface possesses increasing impedance against ion transfer with the decrease in frequency, which, in the Nyquist plot of impedance spectra, was represented by an inclined spike. Furthermore, inclination of the spike at an angle less than 90 °C to the real axis is due to the roughness of the electrode–electrolyte interface [27].

The ionic conductivity of pure and Mn^{2+} doped PVA/MAA:EA polymer blend films is calculated from the relation;

$$\sigma = \frac{l}{R_b A} \tag{3}$$

where l, is the thickness of the film, A, the area of the film and R_b, the bulk resistance of the film material which is obtained from the intercept on the real axis at the high frequency end of the Nyquist plot of complex impedance [28].



Figure 5. Variation of AC electrical conductivity (σ) of the doped samples with frequency of Mn²⁺ doped PVA/MAA:EA polymer blend films at room temperature.

Figure 5 shows the variation of AC electrical conductivity of the doped samples with frequency at room temperature. Figure shows existence of mobile charge carriers, which can be transported by hopping through defect sites along polymer blend chain [29]. Also, there is an enhancement in the ionic conductivity of polymer blend films by adding Mn^{2+} ions, which is due to the increase in mobile charge carriers and the charge carrier mobility enhancement, as well as the improvement of amorphicity. This can be explained by the general conductivity relation $\sigma = n_i q_i \mu_i$ where n_i is the number of charge carriers, q_i is the charge of mobile charge carrier and μ_i is the mobility of charge carriers. According to this relation, the improvement in the ionic conductivity of polymer blend films can be achieved by increasing n_i and μ_i because q_i is the same for all charge carriers in the polymer blend system.

4.4.2 Composition dependence of conductivity:

The variation of conductivity (σ) with Mn²⁺ concentration at room temperature is shown in Figure 6. From the figure, it is noticed that the conductivity of pure film is about 2.46 X 10⁻ 8 Scm⁻¹ at room temperature and increases to 1.41 X 10⁻⁷ Scm⁻¹ for 5 mol% of Mn^{2+} ions. The increase in ionic conductivity with increase in Mn^{2+} concentration is attributed to a reduction in crystallinity of polymer blend films and also to an increase in number of mobile charge carriers. The coordination interactions of ether oxygen atoms of PVA/MAA:EA polymer blend with Mn²⁺ cations, which result in a reduction in crystallinity of PVA/MAA:EA polymer blend, are responsible for the increase in ionic conductivity. The maximum conductivity shows the maximum and effective interaction between oxygen atoms and Mn²⁺ cations [13]. A reduction in crystallinity of PVA/MAA:EA polymer blend can also be seen from the XRD analysis that shows a decrement in the intensity of sharp crystalline peaks with the



Figure 6. Variation of conductivity (σ) with different concentration of Mn²⁺ doped PVA/MAA:EA polymer blend films at room temperature.

addition of Mn^{2+} ions, which results in a dominant amorphous phase in the polymer blend. A polymer chain in the amorphous phase is more flexible, which results in an increase in segmental motion of the polymer, which facilitates higher ionic mobility [30]. The increment in conductivity with increase in dopant concentration is due to the rise in the number of charge carriers as shown in Figure 8. The conductivity data of pure and Mn^{2+} doped PVA/MAA:EA polymer blend at room temperature is presented in Table 3.

4.4.3 Dielectric studies

The dielectric property indicates the amount of charge that can be stored by a material and it can be used as an indicator to prove that the increase in conductivity is due to an increase in the charge carriers or free mobile ions. If dielectric property of the material increases, the amount of charge stored by the material will also increase. The dielectric response is generally described by the complex permittivity $\varepsilon^* = \varepsilon' - i\varepsilon''$, where real ε' and imaginary ε'' components are the storage and loss of energy in each cycle of applied electric field. Figure 7 (A) and (B) shows that the values of ε' and ε'' are very high at low frequency but at high frequency these are relatively constant with similar behaviour that has been reported in a number of polymers [31]. Such high value of ε' at lower frequency may be due to the availability of enough time for the dipoles to align themselves with field before it changes, so dielectric constant is high but at higher frequency dielectric constant

decreases due to available of shorter time for them [32].



Figure 7. Variation of dielectric constant ε' (A) and dielectric loss ε'' (B) with frequency of different concentration of Mn^{2+} doped PVA/MAA:EA polymer blend films at room temperature.

Table 3. Conductivity values of pure and different concentrations
of Mn ²⁺ doped PVA/MAA:EA polymer blend films at room
temperature

temperature.		
Concentration in mol% of Mn ²⁺ :PVA/MAA:EA	Conductivity at 303 K (S cm ⁻¹)	
Pure	2.46 x 10 ⁻⁸	
1	3.68 x 10 ⁻⁸	
2	5.09 x 10 ⁻⁸	
3	6.8 x 10 ⁻⁸	
4	8.47 x 10 ⁻⁸	
5	1.41 x 10 ⁻⁷	

V. CONCLUSIONS AND FUTURE SCOPE

Pure and Mn²⁺ ions doped PVA/MAA:EA polymer blend films were successfully prepared by solution casting technique and were characterised by various techniques. The structural properties of these films were examined by X-ray diffraction patterns. The enhanced amorphous nature of the polymer blend films has been identified from the broadening of X-ray diffraction patterns with increase of Mn²⁺ ions. UV-Vis absorption spectra shown that indirect band gap values shifted to lower energies on doping with Mn²⁺ ions, thus indicating that with increase in concentration of Mn^{2} ions, PVA/MAA:EA polymer blend film becomes more semiconducting in nature. The FTIR spectra exhibit bands characteristic of stretching, bending vibrations of O – H, C – H, C = O, CH₃ – O and CH₂ + α – CH₃ groups. The changes in FTIR signal intensity with increasing dopant concentration indicate the increase in the basicity of C=O groups, implying that the carbonyl group is able to act as a strong electron donor to interact with Mn²⁺ cation. This leads to decrease in the crystallinity and increase in the amorphous content. The increase in the conductivity with an increase in concentration of Mn²⁺ was explained in terms of an increase in the amorphicity. Thus, these changes show a high sensitivity of these films to doping that would suggest the applicability in magnetic and/or optical devices, fabricating solid state batteries and other electrochemical devices. We plan to study the effect of another transition metals Fe and VO as doping materials on thermal, structural, spectroscopic and electrical properties of PVA/MAA:EA polymer blend as our future work.

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