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Absorption, Emission and Stability Investigations on Insulating - Conducting Polymer Blends

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Abstract

Insulating -conducting polymer blends exhibit fascinating optical properties. Poly [2-methoxy-5(3', 7'-dimethyloctyloxy)-1, 4- phenylenevinylene] [MDMO-PPV] is a widely used conducting polymer with band gap of about 2eV. The thin films of MDMO-PPV prepared from chloroform has the absorption maximum at about 490nm and emission maximum at around 590nm. We have prepared free standing films of MDMO-PPV doped at 0.5wt%, 1wt%, 1.5wt%, 2wt%, 2.5wt% and 3.0wt % of 1:1 PMMA/PVAc non conducting polymer matrix by solution cast method. Absorption peaks are slowly red shifted from 499.4nm for 0.5 wt% sample to about 514.2nm for 3.0 wt% sample. Red shift is also observed in emission spectrum (fluorescence spectrum) ranging from 586nm to about 593nm with the increased doping level. Considerable Stoke's shift has been noticed which plays an important role inhibiting fluorescence quenching. New material exhibits multiband gap structure that are calculated using Tauc's plot. Least direct band gap of 1.93 eV and indirect band gap of about 1.27eV are noticed. Thermogravimetric and Differential Scanning calorimetry affirmed slight variations in the stability of the samples compared to that of pure PMMA/PVAc films. There is no such drastic change in the thermal

stability, may be because of very small variation in the doping concentrations. Observed broad band absorption and also efficient emission in the visible region along with the tailored absorption -emission properties can be used in the designing a new materials of desired properties.

Key words : MDMO-PPV, PMMA, PVAc, Stoke's shift

Introduction

Polymer blending is a versatile, simple and low cost approach in designing a new materials with required novel properties to meet the needs of changing technologies and markets. The property of blends are superior relative to the component polymers and also these properties can be tailored by varying the composition ratios or by addition of dopants like salts, nanoparticles, dyes *etc*. [1,2,3,4]. Though the optically transparent PMMA is very useful in industrial and medical applications, its rigidity, hardness and poor thermal stability has restrained its use at high temperature. On blending the PMMA with PVAc its thermal instability can be overcome such that blend can be used for devices that can withstand high temperature. Addition of PVAc also improves the photochemical stability, photo cross linking and photo-oxidation. Literature reports that 50/50 PMMA/PVAc blend is a good matrix with optimized qualities [5,6,7]. H.M Zidan et al reported that PMMA /PVAc blend form an important and unique pair of polymers although they are chemically different [8]. R.M. Ahmed et al reported that PMMA/PVAc blend has modified the optical properties of its homo polymers which is suggested to be a good host matrix for the solar concentrators [9]. Doping PMMA/PVAC (1:1) matrix with metallic fillers like carbon black, grapheme, nano fillers, dyes or quantum dots will result in a material suitable for optoelectronic devices [10,11,12].

It would be an interesting research to replace the inorganic fillers by organic conducting polymers because of their intrinsic electrical conductivity and also their availability with wide variation in color. The properties like insolubility, infusibility and non processability of the conducting polymers can thus be overcome by doping them on to inert polymer matrix [10, 13]. Gumyusenge Aristide et al reported that a blend of conjugated polymers and insulating matrix having high glass-transition temperature are displaying temperature-insensitive charge transport behavior with hole mobility exceeding 2.0 $cm^2/V{\cdot}s$ that are used in thin film transistors [14]. Mincheol Chang et al reported increase in the charge transport properties of 5% P3HT doped Polystyrene [15].

Among conjugated polymers, Light Emitting Polymers(LEPs) are attractive in the field of research because of high absorption coefficient, a high PL efficiency and a large Stokes' shift. The large Stoke's shift can reduce self quenching of its PL emission. Among LEPs poly [2-methoxy-5(3', 7'-dimethyloctyloxy)-1, 4-phenylene vinylene] (MDMO-PPV) is a polymer with long side chains that form flexible films for organic electronics. It is a piezo-resistant polymer with band gap of about 2.2 eV [16, 17, 18]. Such a band gap material is suitable for preparing photoconductive or photoemissive layers, energy harvesting and displaying devices like solar cells, LEDs, optical pump lasers and potentially polymer diode lasers [19]. Preparing MDMO-PPV doped PMMA/PVAc will result in a novel Conjugate/ Inert polymer matric with good optoelectronic properties. Use of chloroform as the common solvent is preferred. Yang Haiyang et al reported that the chloroform is the best solvent for preparing

PMMA/PVAc because of attractive interaction between PMMA and PVAc in chloroform [20]. The investigations on optical absorption and emission properties of this system will definitely shed a light on device applications and Industrial growth.

Experimental

Chemicals used : Poly [2-methoxy-5(3', 7'-dimethyloctyloxy)-1, 4- phenylenevinylene] (mol. wt 120000) is purchased from Sigma Aldrich Corporation. Poly methyl methacrylate (mol. wt 15000) and Polyvinyl Acetate (mol. wt. 50000) are procured from Himedia Laoratories India and Alfa Aesar, England respectively. Common solvent used is the chloroform (mol.wt.119.38) procured from Rankem Chemicals.

Preparation of host solution : 4g of PMMA and 4g PVAc are separately dissolved in 200cc of chloroform. These solutions are magnetically stirred at room temperature for about 24 hours. These solutions are mixed with each other with wight ratio 1:1 and again magnetically stirred for two days to ensure complete blending.

Preparation of dopant solution: Dopant solution is prepared by dissolving 50mg of MDMO-PPV in 200cc of chloroform and subjected to magnetic stirring for about 48 hours at room temperature.

MDMO-PPV/PMMA-PVAc films *:* 1:1 PMMA/PVAC host solutions are doped with MDMO-PPV solutions in the weight ratios 0.5%, 1%,1.5%,2% and 3% in separate beakers. These are magnetically stirred at room temperature for nearly two days. Blended solutions are transferred to petri plates, dried in hot air oven at about 40^{o}C and then tested for different characterizations.

Characterizations : UV-Visible absorption studies are done using JASCO UV-Vis NIR 670 from Karnatak University Dharwad. Photoluminescence studies are accomplished with HITACHI(Japan) model F7000 Spectrophotometer available in Department of Physics Karnatak University Dharwad. Thermal properties are characterized by Universal TA-SDT Q 600, PURSE Lab Mangalore University.

Result and Discussion

UV-Visible Spectroscopy :

Absorption spectra gives insight into band structure and the energy gap in a sample. The spectrum at lower energies reflect atomic vibration while the higher energy spectrum about the electronic energy levels. For polymeric composites, broadband spectrum with peaks of maximum intensity account for transition among electronic states and minimum intensities indicate the vibrational transitions in the materials. Both PMMA and PVAc have intense absorption bands in UV region without any absorption in visible region. Absorption peaks occur at 230nm for undoped PMMA and at 270nm for PVAc. But these bands have been shifted to lower wavelength region in the 1:1 blend of these homopolymers indicating strong intermolecular interaction between the PMMA and PVAc [21]. The thin film of MDMO-PPV prepared from chloroform has the absorption maximum at about 490nm. Omer reported thin films of a conjugated polymer MDMO-PPV prepared from chloroform, absorption maximum peak (λmax) values of 491[22]. Quist et al. reported a MDMO-PPV with an absorption coefficient at λ = 500 nm[23].The Figure 1 represents the UV-Visible absorption spectra of different weight percentage of MPDMO-PPV doped PMMA/PVAc blend. All the doped samples have absorption extending from UV to Visible region with a small peak at 340nm and intense broad band peak around 500nm. The rate of absorption

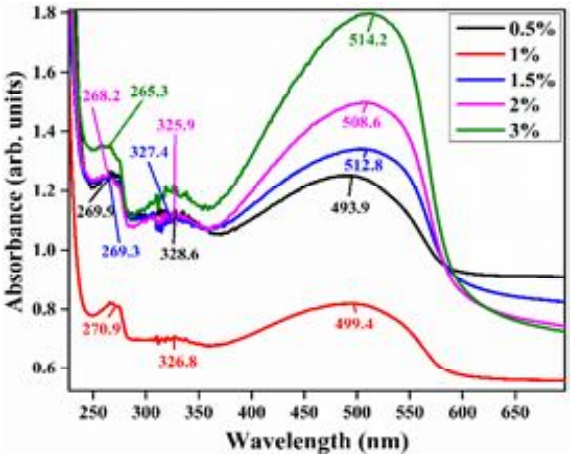

Figure 1 (a)

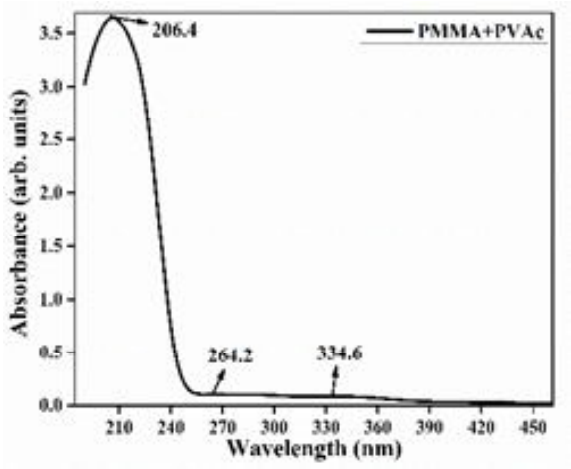

Figure 1(b)

Figure 1. (a) Overlay of UV-Visible absorption (b) UV-Visible spectrum of pure PMMA/PVAc film

increases with doping concentration and also the absorption peak gets red shifted. Maximum shift is observed for 3% MDMO-PPV doped PMMA/ PVAc film with peak at about 525.5nm and onset wavelength 606.43 nm. [24]

Fluorescence spectroscopy :

Fluorescence spectroscopy elucidates the electronic and vibrational energy levels in a sample. The de-excitation of the excited molecule is followed by transition to different vibrational levels of the ground state. This results in the emission of various wavelengths and also of varying intensities. Analysis of these frequencies and relative intensities can give insight into the electronic and vibrational levels of the sample. Since there is loss of energy during emission process the emission line is shifted towards longer wavelength. The difference between the frequency of exciting photon to that of emitted photon is called Stokes shift inferring the of energy lost during excitation and emission process [25,26]. Figure 2 represents the fluorescence emission spectra of different weight percentage of MPDMOPPV doped PMMA/PVAc blend. The fluorescence is caused by an exciting wavelength of 340nm. Intensity peaks arise due to the vibrational transitions 0-1 bands [27]. Small Red shift in the emission wavelength is noticed for samples with increase in doping level. 3wt % doped film has emission at 593nm approaching the usual emission wavelength of the pure MDMO-PPV. Saxena et al. reported the solution of MDMO-PPV polymer in chloroform with a peak value of emission spectra of 592 nm [28] Intensity of emission is increased with the increase in doping level of LEP. Shift in PL spectra are supposed to be controlled by the conformational changes of polymeric chain, aggregation and the solute –solvent interactions for different solvents. Since the solvent for all the samples are same, the position of peaks can be attributed to the formation of aggregates [29]. Table 1 depicts optical parameters of samples.

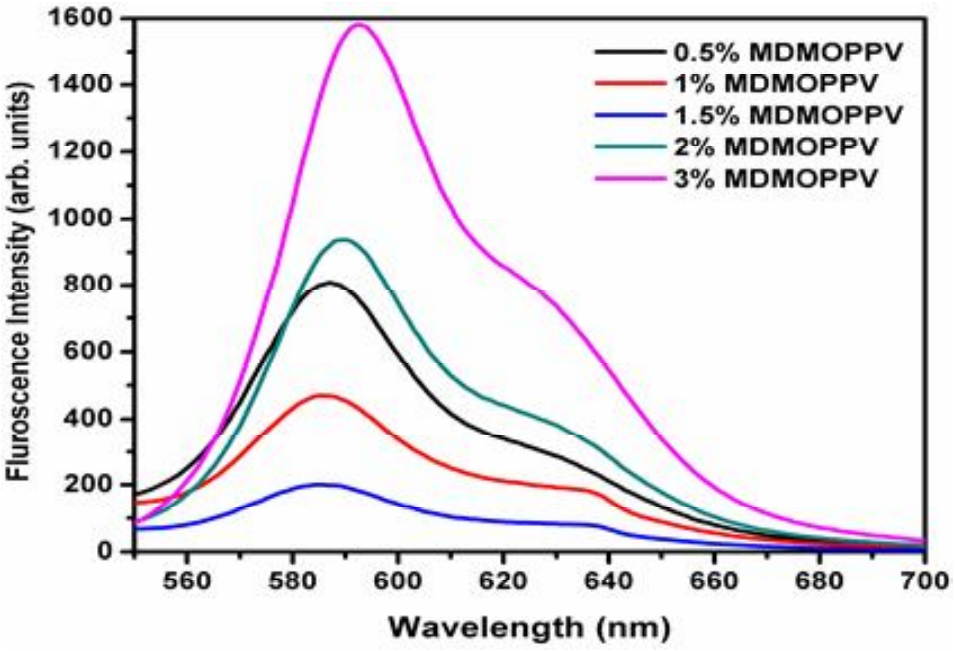

Figure 2. Overlay of Fluoroscence emission spectra of MPDMOPPV doped PMMA/PVAc films

Table-1. Optical parameters of different wt % MPDMOPPV doped PMMA/PVAc blend.

Sample	Absorption maximum λ_{ab} in nm	Emission maximum λ_{em} in nm	Stoke's shift $\Delta\lambda$ in nm
0.5 wt%	499.4	587	87.6
1.0 wt%	501.7	586	84.3
1.5 wt%	514.0	586	72.0
2.0 wt%	520.5	590	69.5
3.0 wt%	525.5	593	67.5

Band gap analysis :

The plot of lnα with hν is depicted in figure 3. The graph confirms the possibility of multi band gap regions in samples. Both direct and indirect band gap values are calculated in higher and lower energy regions. Figure 4(a) and Figure 4(b) are the Tauc's plot for direct band gap in the higher and lower energy regions respectively. Similarly Figure 5(a) and Figure 5(b) represent Tauc's plot for indirect band gaps in higher and lower energy regions. The values of band gaps are depicted in the table 2.

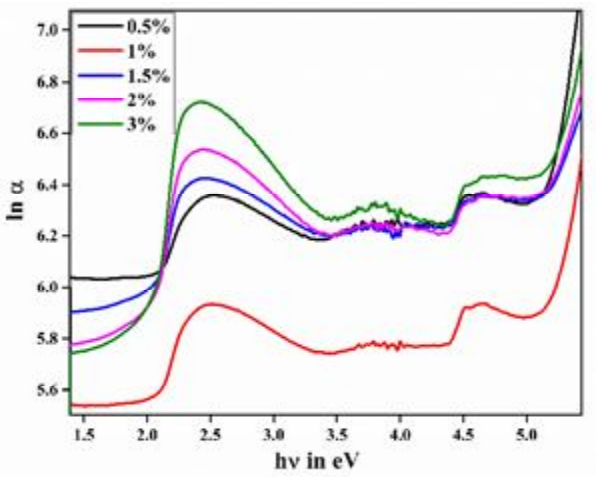

Figure 3. Plot of lnα with hν

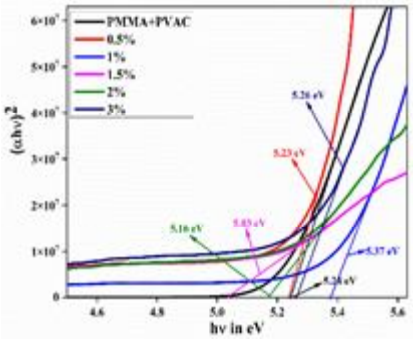

Figure 4(a)

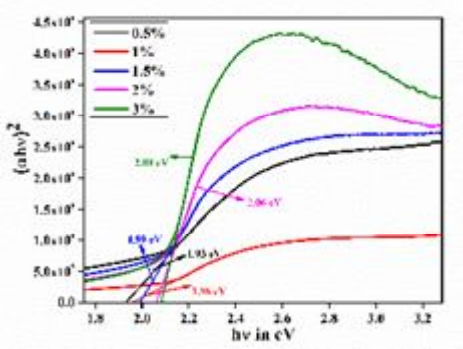

Figure 4(b)

Figure 4(a) Tauc's plot for direct band gap at higher energy region
4(b) Tauc's plot for direct band gap at lower energy region.

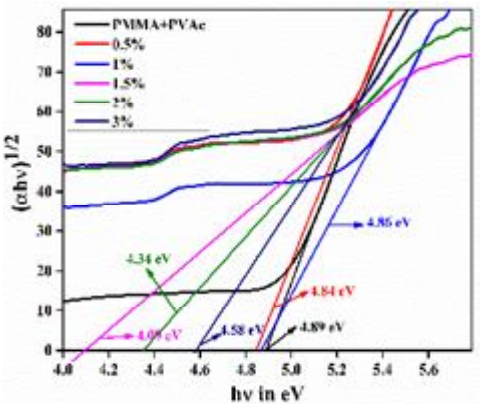

Figure 5(a).

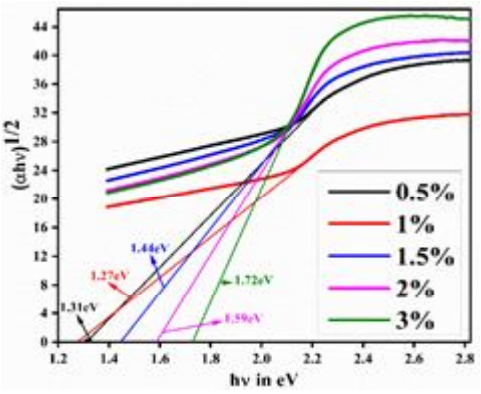

Figure 5(b).

Figure 5(a)Tauc's plot for indirect band gap at higher energy region
5(b)Tauc's plot for indirect band gap at lower energy region

Table-2. Direct and Indirect band gap values

Sample	$E_g - direct$		$E_g - indirect$	
	Lowest	highest	lowest	highest
PMMA/PVAc	———	5.24	———	4.89
0.5%	1.93	5.23	1.31	4.84
1.0%	1.96	5.37	1.27	4.86
1.5%	1.99	5.03	1.44	4.05
2.0%	2.06	5.16	1.59	4.34
3.0%	2.08	5.26	1.72	4.58

Thermal properties :

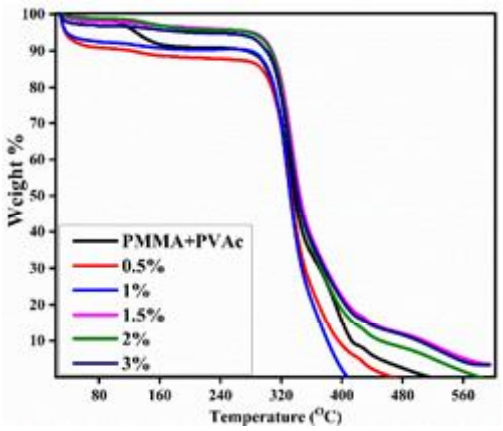

Figure 6(a) Overlay of TGcurves

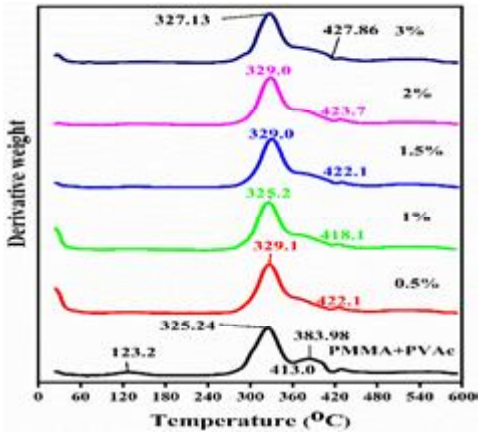

Figure 6(b) Overlay of Derivative TG

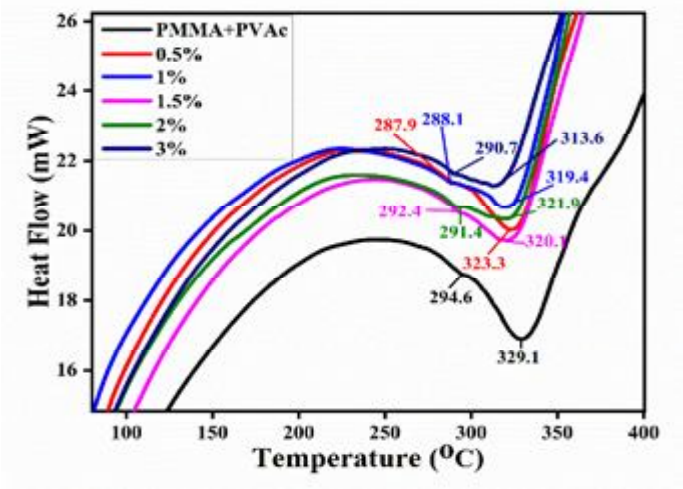

Figure 6(c) Overlay of DSC thermograms

Table-3. Melting points and maximum decomposition temperature

Sample	Tm in ^oC	Td in ^oC
PMMA/PVAc	294.6	329.1
0.5 %	287.9	323.3
1.0 %	288.1	319.4
1.5 %	292.4	320.1
2.0 %	291.4	321.9
3.0 %	290.7	313.6

Literature reports that PVAc is thermally stable relative to PMMA because of their structural difference. PVAc has acetate group linked through C-O bond while PMMA has acrylate group through C-C bond dissociation energy of C-C bond is smaller compared to C-O bond [30]. Three steps degradation behavior is observed for pure PMMa/PVAc with degradation peaks at 130 oC, 325.5 oC ,390 oC, and. The first peak at 130 oC corresponds to the scission of the weak head-to- head bonds and the second one at 325.5 oC corresponds to depolymerization by the unsaturated vinyl chain ends. The third peak at 390 oC corresponds to random main chain scission [30, 31]. Overlapping TGA curves {Figure 6(a)} in the most of the temperature range indicates that the pure and doped samples exhibit almost similar degradation behavior because of very small doping concentrations of LEP. However net increase in thermal stability can be observed which increases with increase in doping level. It is interesting to see that degradation of doped samples changes into two step degradation as indicated by the disappearance of first decomposition peak corresponding to PMMA/PVAC. This indicates that doped samples are more stable than PMMA/ PVAc blend. The observed small shift in the second level degradation peak towards higher temperature is also noticed inferring the increased resistance to depolimerization of vinyl chain ends. Literature reports the glass transition temperature foor pure PMMA is around 106 whereas that for pure PVAc is around 46 and for blends blends the glass transition peak proportionally lies between that for the homopolymers. Tg decreases with increasing PVAc content [30,31,32,33]. Table-3 indicates melting point and maximum decomposition temperatures.

Conclusion

Pure MDMO-PPV film cast from chloroform has the absorption maximum at 491nm and onset wavelength 577nm with band gap 2.15 eV as quoted in literature. MDMO-PPV doped PMMA/PVAc films show red shift in the absorption maximum. Maximum red shift is observed for 3% doped film. Band gap gets considerably reduced for doped films. Minimum band gap of 1.93eV to 1.27 eV is observed for doped films. Red shift is also observed in fluorescence spectra ie wavelength corresponding to emission maximum is also red shifted. Large Stoke's shifts are noticed for all samples. Stoke's shift ranging from 67.5 nm to 87.6nm noticed in the experiment. Higher values of Stoke's shift is helpful in preventing the fluorescence quenching. Insulating PMMA/PVAc environment modifies the photo physical properties of MDMO-PPV. Broad absorption in the visible region can be used in the preparation of solar cell material (photoactive material) and solar concentrator. As the host matrix changes from nonconducting to conducting phase, the films can be used in the construction of transparent electrodes. Doping the system with nanoparticles or with ionic liquids, the electric conductivity of the samples can be enhanced. The new material can be used as an emission layer in LED or in display devises.

Scope for future work :

Absorption and emission properties of these MDMO-PPV doped PMMA/PVAc films can be further tailored by addition of gold nano

particles or other metal oxide nanoparticles. More solar absorption can be achieved due to Surface Plasmon Resonance (SPR) of these nano particles. Even by doping with ionic liquids the electrical conductivity can be enhanced. There are various light emitting polymers like MEH-PPV, P3HT, PFO etc. to be used as dopants on the same PMMA/PVAc host matrix and a relative study of the sample properties can be reported. The experiment can be extended using different common solvents or by changing host matrix also.

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