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Characterization of Conducting Polypyrrole Based Insulating Polymercomposites

Dr.Neeraja M. Haridas
Dept of Physics Santaji Mahavidyalaya

Abstract: *The present study reveals with preparation methods, study of structural, optical and thermal properties of polypyrrole filled PMMA thin film composite. The surface morphology of PPy-PMMA thin films were analyzed using scanning electron microscopy (SEM), tunneling electron microscopy (TEM). The FTIR study gave the signature of the presence of PPy in PMMA. From the analysis of absorption spectra the band gap of polypyrrole filled PMMA have been found to lie between 2.81 to 3.2eV.The decrease in band gap energy suggests a transition from insulating nature to semiconducting nature. Thermal stability of polypyrrole filled PMMA was investigated by TGA.*

Keywords: *PPy, PMMA, Optical band gap, TGA*

I. Introduction

Conducting polymers (CPs) were first produced inthe mid-1970s [1] as a novel generation of organicmaterials that have both electrical and opticalproperties similar to those of metals and inorganicsemiconductors, but which also exhibit the attractiveproperties associated with conventional polymers, such as ease of synthesis and flexibility in processing.Polypyrrole (PPy) is one of the most widely usedconductive polymers, due to its high chemical andphysical stability, low toxicity of the monomer andits easy chemical or electrochemical synthesis, evenin aqueous solutions [2, 3, 4]. PPy may be preparedby chemical or electrochemical oxidation. It hasbeen proven that by inserting doping anions (Figure1) like PTS (Para-toluene-sulfonic acid) theconductivity of PPy can be increased up to 10^2S/cm, [5, 6, 7].Applications of PPy were essentially extendedduring last years and include now such differentfields of science and technology as corrosionprotection of metals, development of individualelectronic devices e.g. diodes, metallization,electromagnetic interference shielding, biosensors,tissue engineering scaffolds, neural probes, drugdeliverydevices, and bio-actuators[8, 9,10].

Experimental Details

Polypyrrole was synthesized by chemical oxidative method from pyrrole monomer using ammonium per sulfate as oxidant and p-Toluene sulphonic acid as a dopant. Aqueous solution of pyrrole 0.5 ml in 7.5 ml water was added instantly to a solution of ammonium per sulphate (150 mg) and p-toluene sulphonic acid (500mg). After 5 minutes the product was recovered by filtration, washed with water and dried at 70^0 c for 12 hours [11].

Preparation of sample:

To prepare the sample of undoped PMMA (series II), 1 gm of PMMA taken in 15 ml of tetrahydro furan, stirred continuously at 353 K for 12 hrs. till the homogeneous transparent solution is obtained The films were prepared by pouring the solution on a cleaned optically plane glass plate.

The glass plate was kept over a pool of mercury for perfect leveling, so as to ensure uniform thickness. The whole assembly was placed in a closed chamber to protect from dust. The solution was allowed to evaporate at room temperature. The film on the glass substrate was kept for 12 hrs at 313 K for outgassing and another 12 hrs at room temperature to remove the traces of solvent. It was cut into small pieces of desired size, which were then washed with ethyl alcohol to remove surface impurities.

For composite samples of 0.5 weight % polypyrrole filled PMMA 0.995 gm of PMMA and 0.005 gm of PPy was added in 15ml THF and the solution was kept at 393 K for 12 hrs and with continuous stirring for maximum dispersion, the thin films were prepared by the same method as discussed.[12]

Thickness measurement:

The thickness of the sample was measured by the compound microscope in conjunction with an occulometer having a least count of 15.38 μm similar to method reported by Sangawar (2007).The thickness of the sample was kept constant throughout the work and is of the order of 46.14 μm.[13]

Electrode coating:

The electrode coating on the film of measured thickness was done by using quick drying and highly conducting silver paint supplied by Eltecks Corporation, Bangalore. A mask of circular aperture of 2.5 cm diameter was used while coating, to ensure uniformity in the size of the coated silver electrode.

Structural characterization

The morphology of PPy filled composite thin films were observed by JEOL-JSM-6380A. The samples were gold coated with high vacuum sputting instrument. The electron gun was set at 10 kV. The micrographs of the samples were taken at the magnification of 100, 200, 500, 1000 within resolution of 10, 50, 100 µm to identify crakes, holes and other changes on the surface. The FTIR was recorded on KBr pellet sample in the range of 4000-400 $cm^{-1}$by Perkin Elmmerfourier transform infrared spectrometer.Thethermogravimetry analysis of polymer composites was carried out on TGA Perkin-Elmer Diamond TGDTA. The transmission spectra of polypyrrole filled PMMA recorded in the region of wavelength 200-700nm on Hitachi 330 UV-Vis spectrophotometer at room temperature.

II. Result and Discussion

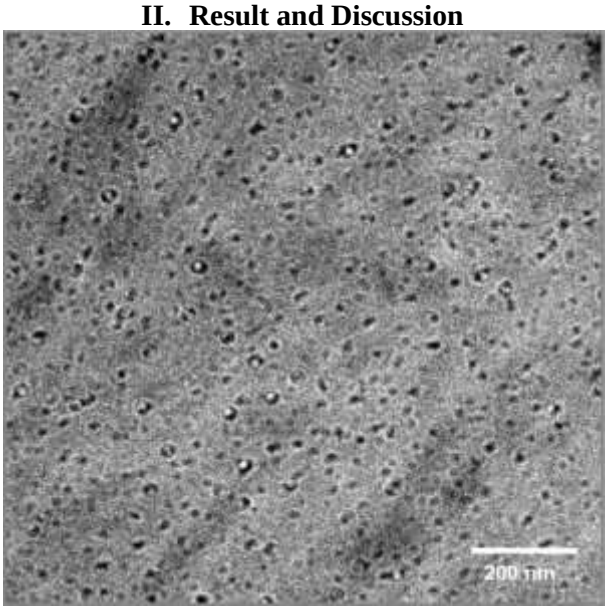

Figure 1 SEM for polypyrrole

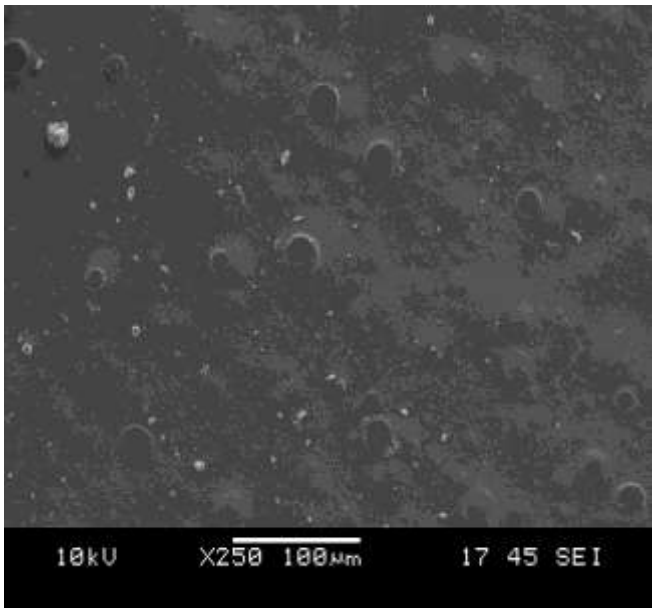

Figure 2 SEM for 10wt% polypyrrole filled PMMA

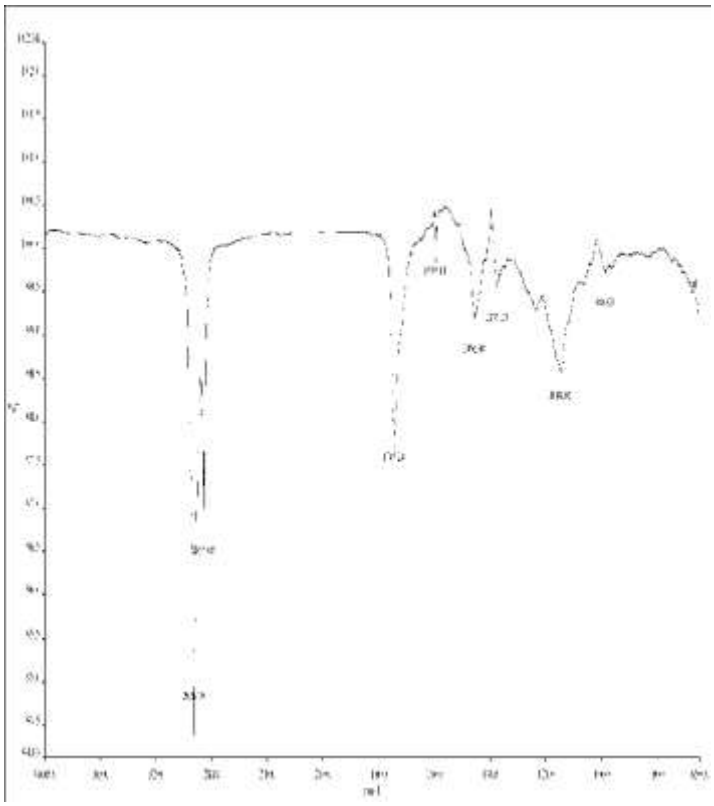

Figure 3 FTIR of unfilled PMMA

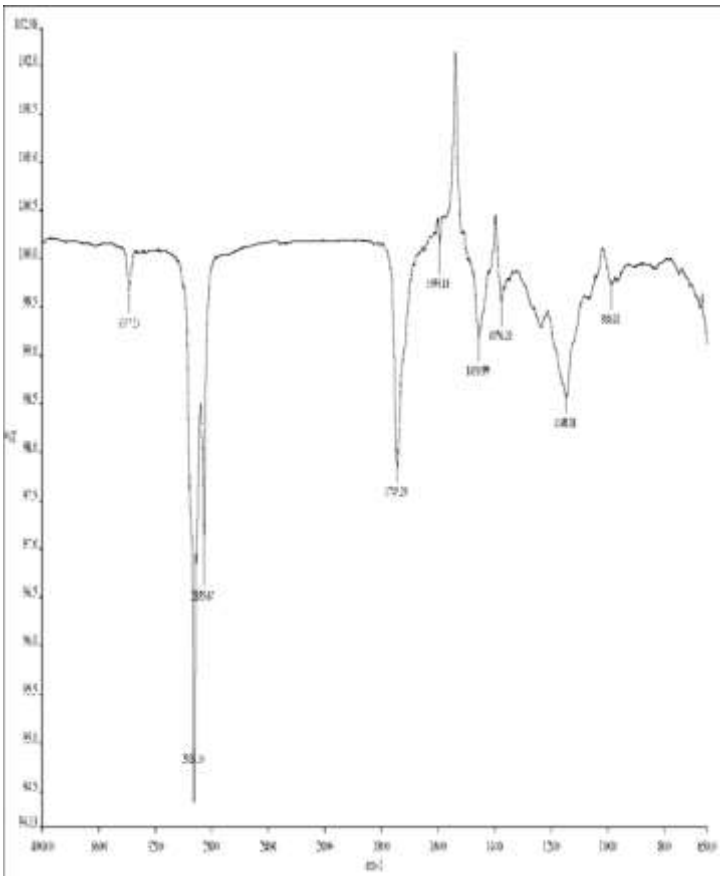

Figure 4 FTIR of 10 wt% of polypyrrole filled PMMA

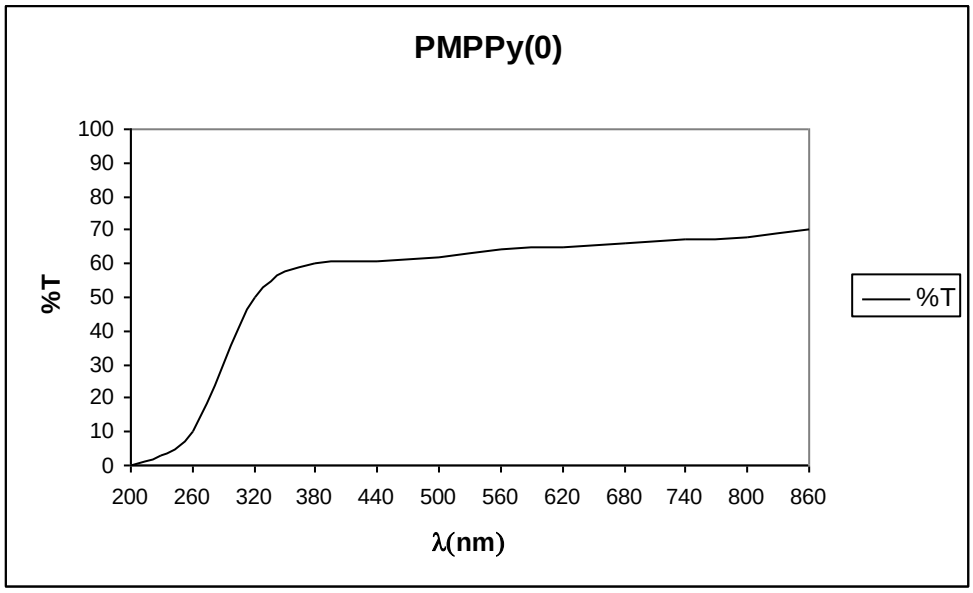

Figure 5 Transmittance spectra for unfilled PMMA thin film

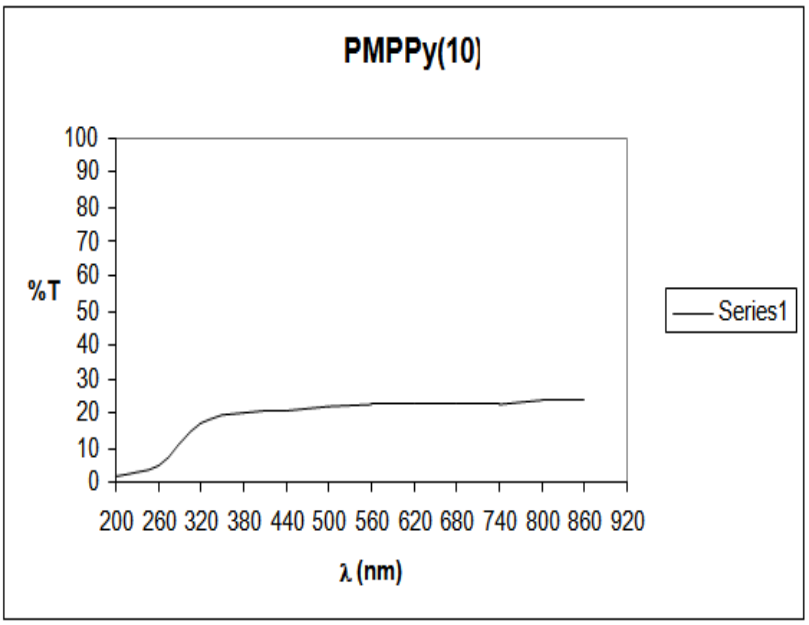

Figure 6 Transmittance spectra for10 wt% polypyrrole filled PMMA thin film

Figure 1 and 2 shows SEM for unfilled PMMA and 10 wt% of PPY filled PMMA respectively. SEM shows the individual particles are present in the matrix. This was due to poor adhesion between polypyrrole and the polymer matrix. Since no crystal structure formation is observed in the micrographs, confirms the amorphous nature of all samples of PPy filled composite thin films. The micrographs exhibit presence of clusters of PPy embedded into host polymer.

Figure 3 and 4 shows FTIR of unfilled and 10 wt% of Polypyrrole filled PMMA. The FTIR reveals of successful incorporation of Polypyrrole into PMMA.

Figure 5 show transmittance spectra for unfilled PMMA thin films. The spectra shows that optical transmittance is almost constant at higher wavelength and it starts decreasing from 350 nm. The maximum percent of transmission is about 72%.

Figure 6 represents the UV transmittance spectra for 10 wt% of PPy filled PMMA matrix respectively. Two characteristic peaks are not clearly observed in Fig. 6.5.1(c) but the percentage of transmittance has been remarkably decreased. This shows that with increase in percentage of filler the transmittance decreases, which indicates the successful dispersion of PPy in PMMA. The absorptionpeak 302 nm attributed π - π * inter band

transition. The absorption peak at 327 nm is attributed to the transition of electron fromthe highest occupied molecular orbital (HOMO) to the lowest unoccupiedmolecular orbital (LUMO) which is relatedto π - π * electron transition18-21

In present study, direct band gap energy value are calculated by taking r = ½. From the band gap energy values it is clear that the E_g values decreases from 3.81 to 2.13 eV for unfilled and PPy filled PMMA.

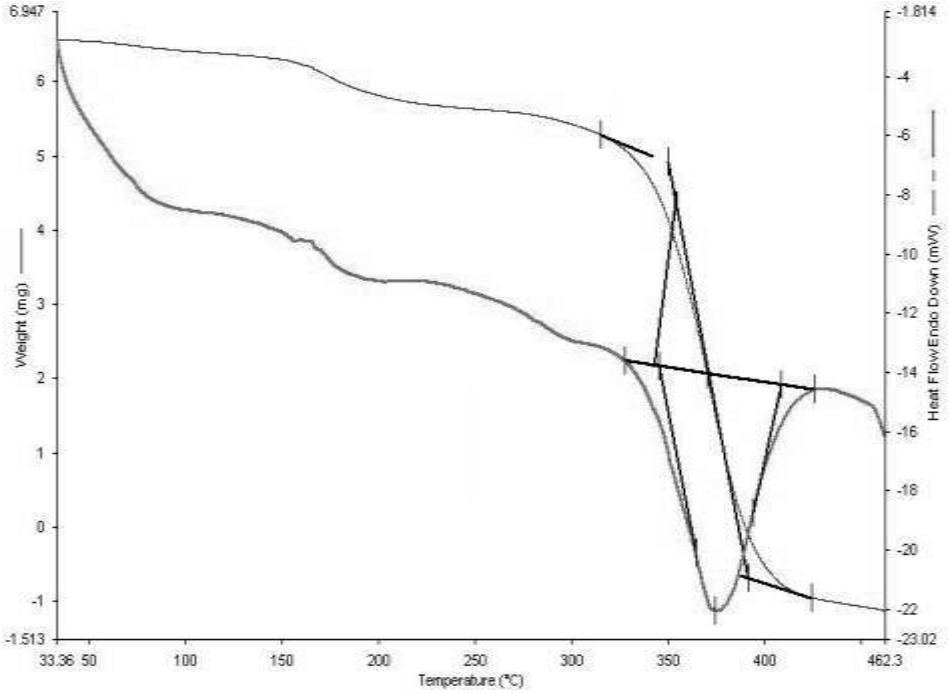

Figure 5 TGA curves for unfilled PMMA thin film

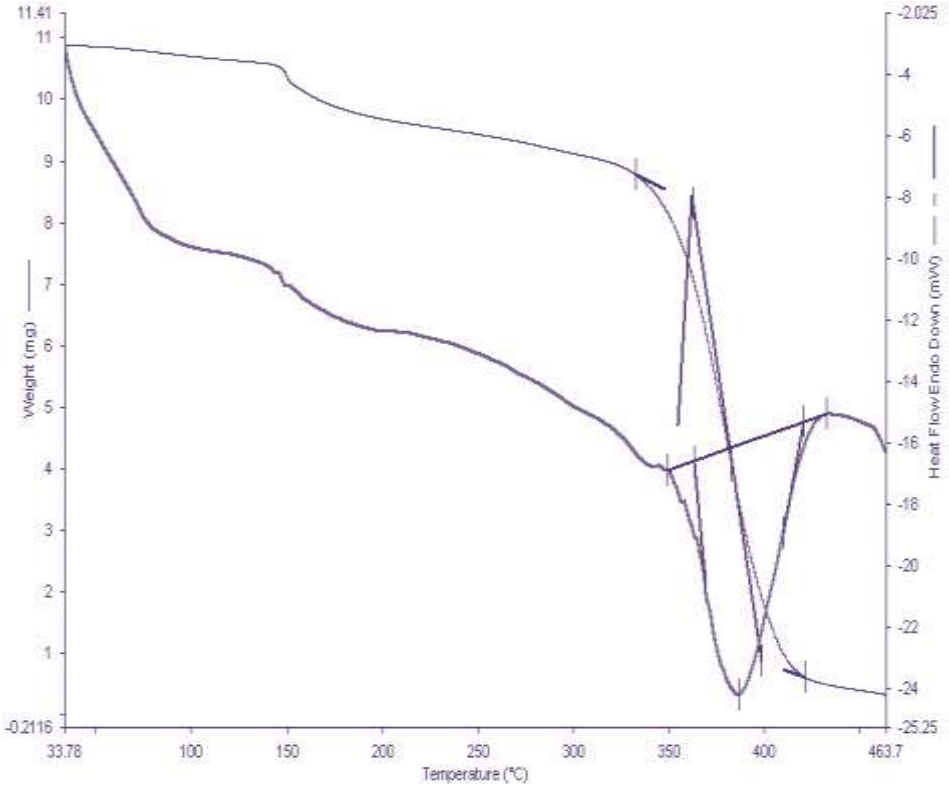

Figure 6: TGA curves for10 wt% of PPY filled PMMA thin film

The TGA/DTA curves for PMPPy(0) is shown in fig. 5, from the TGA curve it is observed that the mass loss for PMPPy(0) starts at 342.67°C and continues very slowly at temperature below 400°C. Above 400°C, this process takes place very rapidly. The mass loss at onset temperature is 20.75%. There is considerable weight loss due to degradation of the large chain of molecules into small fragments.

All these findings indicates that due to addition of PPy in PMMA the composition temperature of PPy increases with decreasing loss of % mass with increasing concentration of PPy.

From the TGA curves, three major transitions corresponding to glass transition temperature (Tg), melting temperature (Tm) and vapourization temperature were observed for different concentrations of PPy.

In present study we have not focused on vaporization temperature. In fact almost all polymers will thermally degrade before they vaporize. For unfilled PMMA a large and sharp endothermic peak at 374.35°C and a clearly baseline gap at 100°C corresponds to melting temperature and glass transition temperature respectively [Raju et al (2007)]. For 10 wt% of PPY in PMMA the endothermic peaks of thermo grams of PMMA became sharper and the corresponding melting temperatures are in the range 376°C to 387°C. The glass transition temperatures are in the range 99-100°C.

III. Conclusion

We have demonstrated that surface modification play important roles in controlling the properties of a wide range of materials, and in regulating the performance of a large variety of devices. We have focused our attention on the surface functionalization of polymerscomposite and their use for modifying various other substrate surfaces for optoelectronic and sensing applications. Even this short paper has revealed the versatility of surface modification for making sophisticated materials with good bulk and surface properties and devices with desirable features for specific applications.

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