



Study of biocompatible polymer blend films with tuneable band gap

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In present communication, an attempt is made to prepare and study structural, optical, and electrical properties of biocompatible polyvinylchloride (PVC)-polyvinylpyrrolidone (PVP) blend films taken in ratio of 1:1 doped with conducting polymer Polypyrrole (PPy). The XRD spectra revealed amorphous nature of all the films under investigation. The Fourier transform infra-red spectroscopy (FTIR) study of conducting polymer doped blend films indicate a significant change in the intermolecular/intramolecular interactions taking place. The study on electrical properties of conducting polymer doped blend films show better conduction of current as compared to that of blend film without conducting polymer and dopant concentration dependent electrical conductivity and conduction mechanism. The results of optical study of conducting polymer doped blend films revealed the possibility of band gap modification from wide band gap to small band gap with suitable doping concentration. These biocompatible blend films with tuneable optical and electrical properties can be potential material for use in fields of photonic biosensors, solar cell and optoelectronics field.

Keywords: Polyvinylchloride, Polyvinylpyrrolidone, Polypyrrole, blend films, bandgap

1 Introduction

A paradigm shift, in the way research is now advancing is seen wherein researchers are moving towards 'polymer based organic green electronics' in which focus is on using: (i) processes consuming less energy and (ii) polymers which are non-toxic, biodegradable and environment friendly^{1,2}. Major technical challenge one faces in applications of polymers in field of electronics is that the solution-processible polymeric materials usually have very low conductivity compared with inorganic counterparts. The high resistance offered by polymers lowers device's performance, and Joule's heat generated by current flow through them decreases its lifetime. Thus, it is required to develop polymer (organic)-based electronic materials with high conductivity. Conducting polymers which possess extended π -conjugation along their backbone exhibit electrical conductivities from semi-conducting to metallic range³. Conducting polymers own properties such as tuneable electrical conductivity, low density, non-corrosiveness, nominal cost, and good thermal and environmental stability. However, conducting polymers offer limitations such as poor mechanical strength, insolubility and infusibility^{4,5}. To overcome these

limitations, conducting polymers are normally blended with other polymers for practical applications.

Insulating biocompatible polymers doped with conducting polymers with tuneable optical and electrical properties can be potential material for use in optoelectronics such as in organic light emitting diodes (OLED), liquid crystal displays (LCD), novel chip-integrated photonic biosensors, electroluminescence, solar cells, and energy storage devices^{1,2,4,6}.

The primary objective of the present study is to prepare biocompatible polymer blend film of Polyvinylchloride (PVC) and Polyvinylpyrrolidone (PVP) taken in ratio 1:1 doped with biocompatible conducting polymer Polypyrrole (PPy), using environment friendly materials and processes. The structural, electrical and optical properties of these films are studied in detail. The study reveals suitability of PPy doped PVC-PVP polymer blend films for photonic biosensors and solar cell applications due to their modified band gap.

2 Experimental

A) Materials

Amongst thermoplastic polymers, Polyvinyl chloride (PVC) is one of the flexible and evergreen polymers containing good chemical and

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environmental properties⁶⁻⁸. In present work Polyvinylchloride (PVC) with average molecular weight 62,000 gm/mol) supplied by Sigma Aldrich was used as an insulating matrix. Polyvinylpyrrolidone (PVP) a conjugated polymer is known to have growing pharmaceutical, biomedical, electronic and optical importance. Films of PVP exhibit good optical properties. PVP deserves a special attention in field of electronics as it exhibits high dielectric strength, good charge storage capacity and dopant dependent electrical properties^{9,10}. In present work, PVP supplied by *Loba Chemie* having average molecular weight of 40,000 gm/mol was blended with PVC.

Amongst conducting polymers, Polypyrrole (PPy) is one of the new generations conducting polymer which possess good sensing capability and also exhibits high reversible redox behaviour which is very important for many applications. PPy also exhibits high biocompatibility^{5,11} and have shown to possess stimulus-responsive properties¹². In present work, Pyrrole monomer supplied by Spectrochem, India, was used as received. Tetrahydrofuran purchased from HPLC was used as solvent and Anhydrous Ferric Chloride purchased from Otto Chemicals, was used as oxidant for polymerization of Pyrrole monomer.

B) Sample Preparation

The polymer blends in present work were prepared in form of films, owing to the versatility that they offer when used as coatings or flexible substrates/encapsulants/implants. In this study, an attempt was made to keep the process as much environment friendly as possible. Therefore, the polymers were selected with a focus of their being low cost, non-toxic, biocompatible and the ones which can be degraded or recycled. Also, the method of preparation of films adapted here was simple and cost effective *i.e.* the films were prepared by solution cast technique using a common solvent THF. The sample preparation was carried out at low temperatures (28°C to 60°C). PPy doped PVC-PVP (taken in ratio 1:1) films were prepared by mixing oxidative polymerization method^{13,14}. The weight percentage concentration of PPy in PVC-PVP was kept as 10%, 20%, 30%, 40% and 50%. All the films were kept in desiccator until use.

C) Characterization Techniques

A. Structural characterizations

The structural properties of the polymer films were investigated by X-ray diffraction (XRD) and Fourier

transformed infrared (FTIR) spectroscopy. X-ray diffraction (XRD) studies of polymer films in present work were carried out using a Bruker AXS D8 Advance Powder X-ray diffractometer using CuK α radiation ($\lambda = 1.5406 \text{ \AA}$). The XRD patterns of the films were recorded in the 2θ range of 10-90° with step width of 0.020° and step time 64 seconds. FTIR was recorded at room temperature in the wave number ranging from 500 to 4000 cm^{-1} with potassium bromide (KBr) as solvent using an Agilent Cary 630 FTIR Spectrometer. FTIR spectroscopy is used to identify the functional groups present in polymer blends and to verify complexation taking place between constituents of polymers in blends.

B. Electrical Characterization

The conduction current (I) as a function of Voltage (V) was measured using Keithley 2400 source meter for PPy doped PVC-PVP films at room temperature. The I - V data of these films is used to analyse the effect of PPy doping and to get an insight on the type of conduction mechanism taking place in these films.

C. Optical Characterization

In the present study the optical absorption and transmission profiles of polymer films were recorded at room temperature in the spectral range of wavelength 185-900 nm using Shimadzu 2600 spectrophotometer. Absorbance, A is defined as the ratio of intensity of light absorbed I_A by the sample to the incident intensity of light I_0 ¹⁵

$$A = \frac{I_A}{I_0} \quad \dots (1)$$

Absorption coefficient (α) is given as:

$$\alpha = \frac{2.303 A}{d} \quad \dots (2)$$

where d is the thickness of sample

Transmittance T, is given by:

$$T = \exp(-2.303 A) \quad \dots (3)$$

The optical measurements of the polymer films were utilized to determine optical parameters such as absorption coefficient (α), optical energy band gap (E_{opt}) and Urbach energy (E_u).

3 Results And Discussions

A. XRD Analysis

PVC-PVP-PPy polyblend film comprises of three different types of polymers. The X-Ray diffraction study of these films was undertaken in order to

identify the phase (crystalline or amorphous) of the final product. The X-ray diffraction patterns for undoped and PPy doped PVC-PVP blend film (Fig. 1) exhibited broad peaks at $2(\theta)$ values around 25° and 42° . Moreover, it was also observed that as the percentage weight concentration of PPy in PVC-PVP blend films was increased the peak around 42° was seen to be slowly diminishing. The overall analysis of XRD spectra revealed amorphous nature of all the films under investigation

B. FTIR analysis

The FTIR spectra of PVC-PVP blend film is shown in Fig. 2(a) whereas Fig. 2(b) and (c) show the spectra of PVC-PVP-PPy films for 10% and 30% weight percentage concentrations of PPy in PVC-PVP. The spectra of PPy doped PVC-PVP film indicates a peak

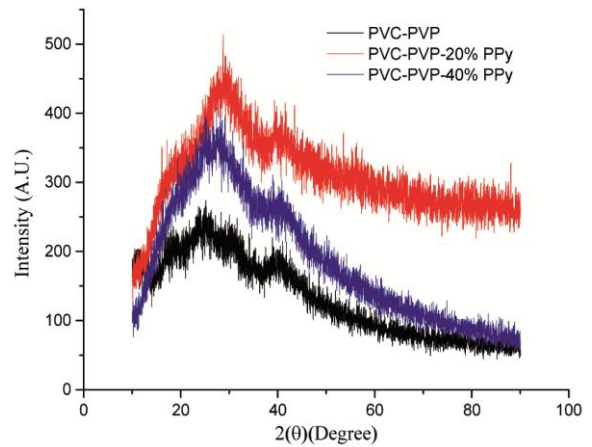


Fig. 1 — XRD patterns of PVC-PVP blend film and PPy doped PVC-PVP blend films

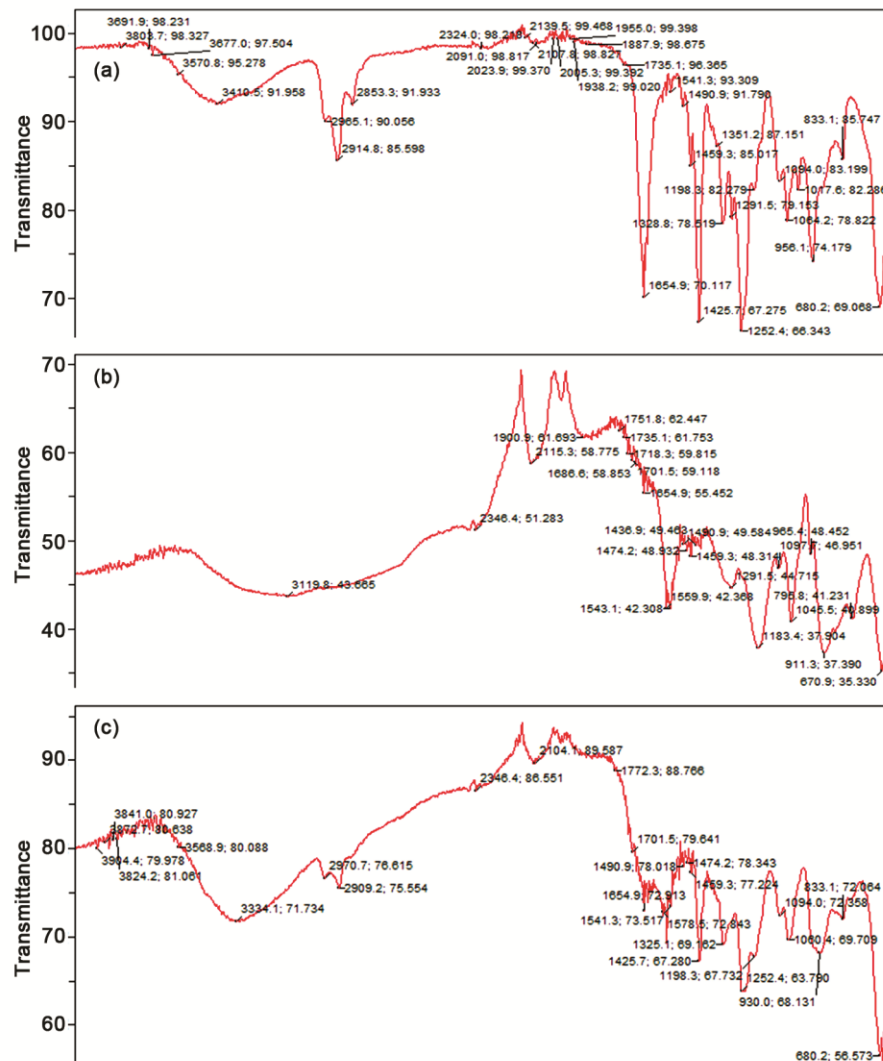


Fig. 2 — (a) FTIR spectra of PVC-PVP film (b- c) FTIR spectra of PPy doped PVC-PVP films with 10% and 30 % weight concentration of PPy

around 1559 cm^{-1} which stands for typical Polypyrrole ring vibrations and is seen to grow with the weight percentage concentration of PPy in the blend films¹⁶. The peaks at 911 cm^{-1} in Fig.2(b), 930 cm^{-1} in Fig.2(c) represent =C-H out of plane vibration mode indicating polymerization of Pyrrole¹⁶. Peaks at 1045 cm^{-1} in Fig. 2(b), 1094 cm^{-1} in Fig.2 (c) represent N-H in plane deformation¹⁷. The peaks at 1183 cm^{-1} in Fig. 2(b), 1198 cm^{-1} in Fig.2(c) represent doped state of PPy.

Some new peaks at 2346 cm^{-1} , 2113 cm^{-1} and 2104 cm^{-1} and 1541 cm^{-1} are also seen in FTIR spectra of PVC-PVP-PPy blend films. The appearance of new peaks along with changes in existing peaks in FTIR spectra is a direct indication of complexation taking place between three polymers *i.e.* PVC, PVP and PPy.

C. Electrical properties

Fig. 3 shows graph of $\ln I$ versus $\ln V$ for PVC-PVP-PPy films. Electrical conductivity in conducting polymers involves movement of positively charged carriers or electrons along the polymer chain and hopping of these charge carriers between polymeric chains. When PVC-PVP is doped with Pyrrole monomer, it forms a donor-acceptor complex in the conjugated system. Further, when Pyrrole monomer incorporated PVC-PVP film is dipped in aqueous solution of FeCl_3 , the polymerization of Pyrrole initiates. The presence of FeCl_3 leads to the formation of conjugation defects along the backbone of the polymer chain. The defect involves the rearrangement of the double bond in the conjugated system and provides a way for charge carrier to move along the chain. As a result, quasi particles are created which are called polarons. Polarons have low mobility and so PVC-PVP films with low concentration of PPy exhibit low conductivity. When percentage weight concentration of Pyrrole is increased, more polarons are formed and they combine or ionize to form spin less dications *i.e.* bipolarons which extend over few

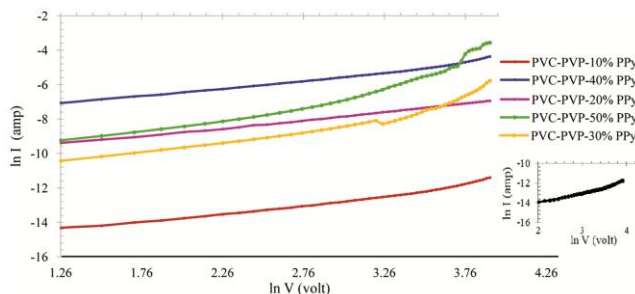


Fig. 3 — Graph of $\ln I$ versus $\ln V$ for PVC-PVP-PPy poly blend films (inset shows graph for PVC-PVP)

rings. A single bipolaron is more stable than double polarons. The higher value of conduction current exhibited by PVC-PVP films doped with higher concentration of PPy results from the motion of such spin less bipolarons. Moreover, as the magnitude of the applied voltage increases the number of polarons and bipolarons which results in increase in conduction current¹⁸⁻¹⁹. Comparison of graphs of $\ln I$ versus $\ln V$ for PVC-PVP and PPy doped PVC-PVP films reveal that PVC-PVP film doped with conducting polymer PPy show better conduction of current as compared to that of PVC-PVP.

Study of possible Charge Conduction Mechanism

The electrical conduction in metals is mainly due to the excitation of electrons from valence band to the conduction band. However, in case of organic solids the conductivity due to electron exciting from valence band to conduction band is negligible²⁰. The electrical conduction mechanism in such materials is rather complex which usually has been explained in terms of electron liberation from the traps in bulk material *i.e.* Poole-Frenkel mechanism or emission of electrons from cathode *i.e.* Schottky-Richardson mechanism²⁰. To identify possible charge transfer mechanisms in the films under study, both Poole-Frenkel emission and Schottky emission mechanisms have been investigated.

(i) Poole-Frenkel Mechanism

Poole-Frenkel conduction mechanism results from field enhanced excitation of trapped charges into the conduction band of the polymer. This mechanism, deals with the conduction in such materials where defect /impurity generated electron traps are involved. The structural defects in the material cause additional energy states close to the band edge which are called traps. These traps restrict the current flow because of the capture and emission process, thereby contributing in conducting electric current in the material^{21,22}. The current-voltage relationship for Poole-Frenkel mechanism is given as^{23,24}.

$$J = B \exp \left[-\frac{\phi}{kT} + \beta_{PF} E^{1/2} \right] \quad \dots (4)$$

where B is constant, β_{PF} is Poole-Frenkel field lowering coefficient and other symbols have their usual meanings. Poole-Frenkel field lowering coefficient β_{PF} is given as

$$\beta_{PF} = \left(\frac{e}{kT} \right) \left(\frac{e}{\pi \epsilon \epsilon_0 d} \right)^{1/2} \quad \dots (5)$$

The Poole-Frenkel mechanism predicts a field dependent electrical conductivity as^{23,25}.

$$\sigma = \sigma_0 \exp \left[\frac{\beta_{PF} E^{1/2}}{2kT} \right] \quad \dots (6)$$

$$\log \sigma = \log \sigma_0 + \frac{\beta_{PF} E^{1/2}}{2kT} \quad \dots (7)$$

The Poole- Frenkel conduction mechanism is characterized by the linearity of plot of $\log \sigma$ versus $E^{1/2}$. The $\ln \sigma$ versus $E^{1/2}$ plots for PPy doped PVC-PVP blend films are shown in (Fig. 4).

For PVC-PVP-PPy blend films, plots of $\ln \sigma$ versus $E^{1/2}$ with low weight percentage concentration of PPy, this mechanism does not seem to contribute significantly to the electrical conduction in lower field region as the slopes are negative showing the effect of PVC-PVP matrix to be dominant in polyblend film. However, for polyblend film with higher weight percentage concentration of PPy, $\ln \sigma$ show appreciable dependence on $E^{1/2}$ and also gave positive slopes suggesting that the electrical conduction in these films take place by Poole - Frenkel mechanism.

(iii) Schottky -Richardson Mechanism

Conduction of current due to emission of electron from electrode (metal) to the dielectric (polymer) is called Schottky-Richardson or thermionic emission and is one of the most observed conduction mechanism in polymer films²⁵. The Schottky-Richardson current- voltage relation is expressed as²⁴

$$\log J = \log A T^2 - \frac{\phi_s}{kT} + \beta_{SR} E^{1/2} \quad \dots (8)$$

Where J is the current density, A is the effective Richardson constant, T is the absolute temperature, ϕ_s is the Schottky barrier height, k is Boltzmann's Constant, E is electric field across the polymer and β_{SR} is Schottky-Richardson field lowering constant and is given as^{23,24}

$$\beta_{SR} = \left(\frac{e}{kT} \right) \left[\frac{e}{4\pi\epsilon_0\epsilon_r d} \right]^{1/2} \quad \dots (9)$$

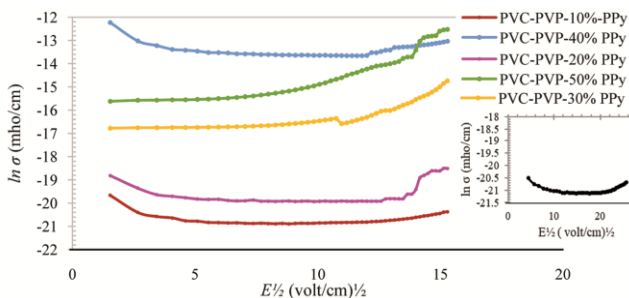


Fig. 4 — Graph of $\ln \sigma$ versus $E^{1/2}$ PPy doped PVC-PVP blend films (inset shows graph for PVC-PVP film)

where e is the electronic charge, ϵ_0 the permittivity of free space, ϵ_r relative permittivity and d is the thickness.

The Schottky plots ($\ln J$ versus $E^{1/2}$) should be a straight line with a positive slope characterizing the Schottky- Richardson conduction mechanism. Schottky plots for PVC-PVP-PPy blend films are shown in Fig. 5, which are straight lines with positive slope indicating the applicability of Schottky-Richardson conduction mechanism to these films.

Further, to understand the actual conduction mechanism involved in these blend films, amongst Poole-Frenkel and Schottky Richardson, the theoretical values of β_{SR} and β_{PF} were calculated using Eq. 4 and 9 and their experimental values (β_{exp}) were estimated from the slopes of graphs of $\text{Log } I$ versus $V^{1/2}$ which are shown in Fig. 6.

The values of β_{SR} , β_{PF} and β_{exp} of films under investigation are appended in Table 1. From Table 1 it is seen that for PVC-PVP blend film with 50% weight concentration of each PVC and PVP, the electrical conduction is mainly dominated by Pool-Frenkel mechanism. However, in case of PVC-PVP films with

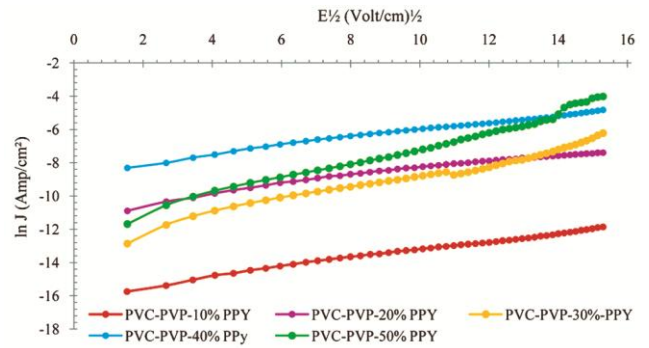


Fig. 5 — Graph of $\ln J$ versus $E^{1/2}$ for PPy doped PVC-PVP blend films (inset shows graph for PVC-PVP film)

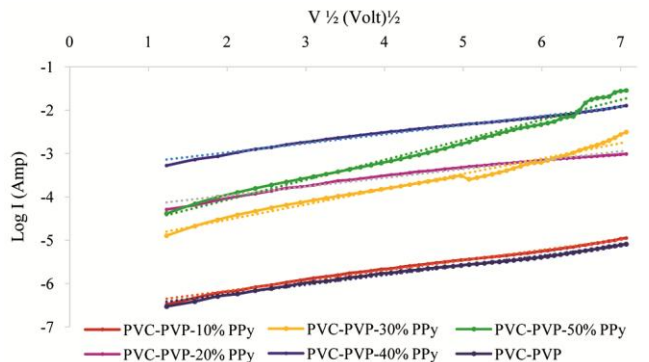


Fig. 6 — Graph of $\text{Log } I$ versus $V^{1/2}$ for PPy doped PVC-PVP films

Table 1 — Theoretical values of β_{PF} , β_{SR} and experimental values β_{exp} of PPy doped PVC-PVP blend films under investigation

Composition	β_{exp} ($\times 10^{-5}$) (eV)	investigation	
		β_{PF} ($\times 10^{-5}$) (eV) (Theoretical)	β_{SR} ($\times 10^{-5}$) (eV) (Theoretical)
PVC-PVP	3.54	4.19	2.09
PVC-PVP-10% PPy	2.36	4.20	2.10
PVC-PVP-20% PPy	2.77	3.44	1.72
PVC-PVP-30% PPy	3.05	2.92	1.46
PVC-PVP-40% PPy	3.21	2.52	1.26
PVC-PVP-50% PPy	3.36	2.22	1.11

low concentrations of PPy, β_{exp} values were found to be nearer to theoretical value of β_{SR} exhibiting Schottky-Richardson type of conduction mechanism. But, the PVC-PVP film with 30% PPy shows β_{exp} value closer to theoretical value of β_{PF} suggesting Poole-Frenkel conduction mechanism dominating the Schottky-Richardson mechanism. On further increasing concentration of PPy in polyblend film the effect of Poole-Frenkel conduction mechanism seems to be reducing again.

At higher concentrations of PPy in PVC-PVP film, charge carriers injected by lowering electrode-insulator potential barrier are trapped at trapping sites which are created by polymer phase boundaries/ interface/ chain foldings *etc.* and are subsequently released, following the Poole-Frenkel mechanism²⁶. Thus, the study of electrical properties of PPy doped PVC-PVP films exhibit dopant concentration dependent electrical conductivity and conduction mechanism.

D. Optical properties

Figure 7 shows the absorbance spectra of PVC-PVP blend films doped with different weight percentage concentration of PPy. The absorption peaks that were observed at high wavelengths, 400–800 nm, for the PPy doped PVC-PVP films (Fig. 7) are related to the existence of π electrons. The shifting towards the longer wavelengths indicates the small band gaps of PPy doped PVC-PVP blend films^{27,28}. The shifts observed in absorbance spectra of doped films towards the longer wavelengths can be attributed to the existence of π -delocalization along the polymer chain²⁹. The source of π -delocalization in the doped samples is found to be related to the structure of PPy. One can see from Figure 7 that the PVC-PVP blend film doped with 50% weight concentration of PPy exhibits a distinct and

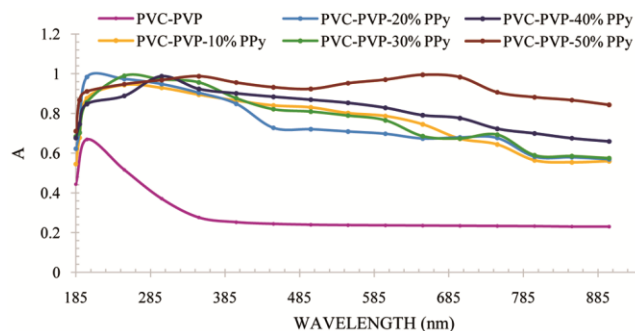


Fig. 7 — Absorbance spectra of PVC-PVP and PPy doped PVC-PVP blend films

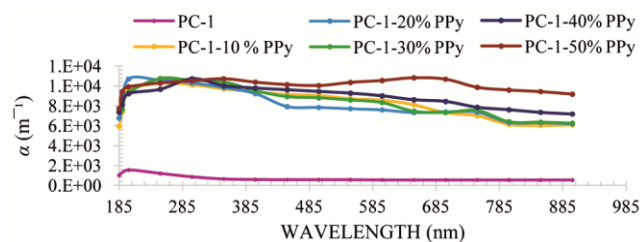


Fig. 8 — Graph of absorption coefficient versus wavelength for PVC-PVP and PPy doped PVC-PVP blend films

intense peak at around 670 nm, which reveals its suitability for photonics and optoelectronics applications.³⁰

Moreover, the absorbance spectra of PVC-PVP-PPy films show broadening of the absorption edge which increases with increasing weight percentage concentration of PPy. This behaviour is generally interpreted to be triggered by the formation of extended systems of conjugate bonds *i.e.* a possibility of formation of carbon clusters. The absorption bands in the investigated range of wavelength are associated to π - π^* transitions³¹. Furthermore, the absorbance spectra showed that PPy doped PVC-PVP blend films strongly absorbed UV rays as well as visible light.

Figure 8 represents the variation of absorption coefficient with photon energy for the undoped and PPy doped PVC-PVP films. It is seen from the spectra that, upon doping PVC-PVP films with PPy, the absorption edge is shifted towards lower photon energy sides. The absorption edge is a region in which an electron is excited, from a lower energy state to a higher energy state, by an incident photon. The absorption coefficient of PPy doped PVC-PVP blend films is observed to increase with increase in concentration of PPy. However, PVC-PVP film with 50% weight concentration of PPy exhibited highest value of absorption coefficient (Fig. 8) in the measured wavelength region.

The optical band gap (E_{opt}) in an amorphous material is generally determined by the Tauc relation given as:^{32,33}

$$(\alpha hf)^{\frac{1}{n}} = B(hf - E_{opt}) \quad \dots (10)$$

Figure 9 shows the plots of $(\alpha hf)^{\frac{1}{n}}$ versus hf which are also called the Mott plots. The linear portion of the plot when extrapolated to the energy axis corresponds to the threshold of optical absorption. From this optical band gap energy (E_{opt}) is calculated. The quantity n determines the type of electronic transitions causing optical absorption which can take values $n = 1/2, 3/2, 2$ and 3 for direct allowed, direct forbidden, indirect allowed and indirect forbidden transitions respectively. In the case of PVC-PVP and PPy doped PVC-PVP blend films the linear portion of $(\alpha hf)^{\frac{1}{n}}$ versus hf graphs was best fitted with $n = 1/2$ (Fig. 9) indicating the transition to be of direct type.

The values of E_{opt} for PVC-PVP film and PPy doped PVC-PVP films are tabulated in Table 2. On doping PVC-PVP film with PPy results in modification in Mott plot *i.e.* change in the optical band gap of the polyblend films. The optical band gap E_{opt} for PVC-PVP blend film was 4.1 eV which reduced to 2.1 for PVC-PVP blend film doped with 40% weight concentration of PPy. The reduction in optical band gap values of doped films reveals that doping PVC-PVP blend film with conducting polymer PPy can modify the electronic structure of the host PVC-PVP matrix; in particular, the energy states between the valence and conduction bands.

The lower values of E_{opt} for higher weight percentage concentration of PPy may be attributed to creation of localized states in the band gap as a result of compositional disorder³³ The decrease in optical band gap can be correlated with increase in electrical conductivity of the films as also seen in Fig. 3.

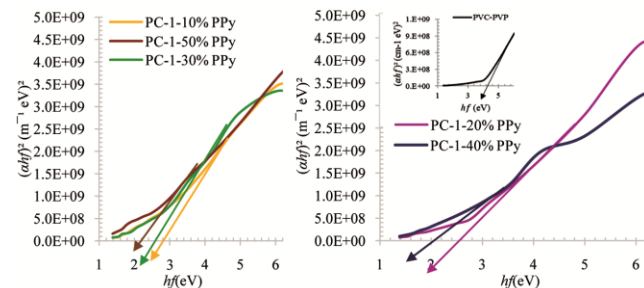


Fig. 9 — Graph of $(\alpha hf)^2$ versus hf for PPy doped PVC-PVP blend films (inset for PVC-PVP)

The absorption coefficient of films under investigation shows an exponential dependence on the photon energy which follows Urbach’s formula (equation 11). The shape of the fundamental absorption edge in the exponential (Urbach) region gives information on the disorder effects. The lack of crystalline long-range order in amorphous materials is associated with a tailing of density of states. At lower values of the absorption coefficient ($1 \text{ cm}^{-1} < \alpha < 10^4 \text{ cm}^{-1}$), the extent of exponential tail of the absorption edge characterized by the Urbach energy (E_u) is given by Eq. 11^{34,35}

$$\alpha (hf) = \beta \exp\left(\frac{hf}{E_u}\right) \quad \dots (11)$$

E_u is estimated by plotting $\ln \alpha$ versus hf and fitting the linear portion of the curve with a straight line Fig. 10. The reciprocal of slopes of this linear portion give values of Urbach energy (E_u).

The values of E_u for PVC-PVP blend film and PPy doped PVC-PVP blend films are tabulated in Table 2. The value of E_u for undoped PVC-PVP blend film was 0.433, while it increased to 0.548 for PVC-PVP blend film doped with 50% weight concentration of PPy implying an increase in band tails’ width. This increase of Urbach energy can be indirectly attributed to the increase of amorphous nature within PPy doped PVC-PVP blend films. The larger energy tails of PPy

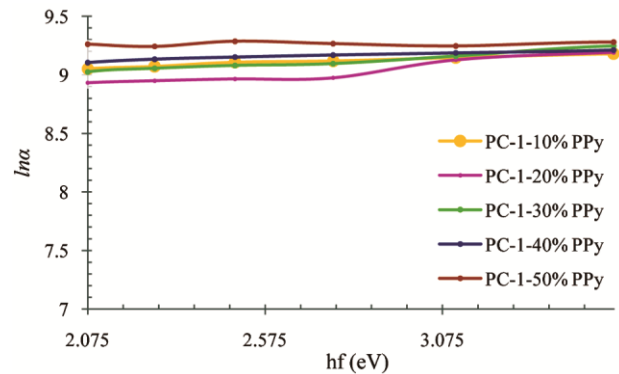


Fig. 10 — Graph of $\ln \alpha$ versus hf for PVC-PVP and PPy doped PVC-PVP blend films

Table 2 — Values of optical band gap and Urbach energy

Composition	E_{opt} (eV)	E_u (eV)
PVC-PVP	4.1	0.433
PVC-PVP-10% PPy	2.8	0.487
PVC-PVP-20% PPy	2.6	0.497
PVC-PVP- 30 % PPy	2.4	0.504
PVC-PVP-40% PPy	2.1	0.530
PVC-PVP-50% PPy	2.2	0.548

doped PVC-PVP films indicate creation of disorder and imperfection in the band structure of host PVC-PVP matrix³⁰.

4 Conclusions

In present study biocompatible blend films of PVC-PVP doped with PPy were prepared at room temperature using simple and cost-effective solution cast technique. Their structural, electrical and optical properties were studied. The analysis of XRD patterns of these films revealed their amorphous nature. The complexation taking place between the constituent polymers in the blend films was confirmed from their FTIR spectra. The study on electrical properties of these films show that PVC-PVP film doped with PPy show enhanced values of conduction of current as compared to that of PVC-PVP. The nature of electrical conduction mechanism was seen to change from Schottky-Richardson type to Pool-Frenkel type on varying the percentage weight concentration of PPy in the blend films.

The results of optical study of PPy doped PVC-PVP blend films revealed the possibility of band gap modification with suitable doping with conducting polymer, PPy. The wide bandgap PVC-PVP polymer blend film is seen to be modified into small bandgap polymer blend film by doping it with PPy, which is also an environment friendly, biocompatible polymer. PPy doped PVC-PVP blend films with tuneable optical and electrical properties can be potential material for use in photonic biosensors, solar cell and optoelectronics field.

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