

Synthesis and characterization of polyaniline-polyvinyl chloride blends doped with sulfamic acid in aqueous tetrahydrofuran

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Received 3 April 2006; accepted 17 July 2006

Abstract: The temperature dependence of direct current (dc) conductivity was studied for various samples of polyaniline-polyvinylchloride (PANI-PVC) blended films. Polyaniline was doped with different concentrations of sulfamic acid in aqueous tetrahydrofuran (THF) and the blended films were prepared by varying the amount of doped PANI relative to a fixed amount of PVC. The dc conductivity of PANI-PVC blended films was measured to determine the effect of sulfamic acid (dopant) in the temperature range (300–400K). The mechanism of conduction is explained by a two-phase model. In order to evaluate the effect of the dopant, conductivity-derived parameters such as the pre-exponential factor (σ_0) and the activation energy (ΔE) were calculated. The structural changes of polyaniline-PVC blended films were characterized by FTIR spectroscopy that explores information about the suitability of the dopant in the chemical doping process.

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Keywords: Electrical conductivity, polymer blends, doping, infrared spectra

1 Introduction

Among all conducting polymers, polyaniline (PANI) has attracted much attention because of its ease of fabrication and good stability in air. However, like most electrically

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conductive polymers, PANI was considered to be unprocessable. Polyaniline is inherently brittle and poor in processability. Efforts were conducted to improve the processability and mechanical properties of the polymer by preparing blends of conducting polymers with other conventional polymers.

The goal of combining the mechanical properties of a polymer with the electrical properties of the conducting PANI, can be achieved by blending conducting and conventional polymers. Properties such as flexibility, tensile strength, elasticity and processability can be achieved by processing conducting polymers with commercial polymers. The possibility of processing conducting polymers, in the form of blends, with commercial polymers has increased the applications and technological potential of the materials because the features required from both the conventional polymer and the conducting polymer can be combined in a synergistic manner. Blending can be accomplished in several ways, i.e. by electrochemical deposition, powder dispersion, melt blending and solution blending. Among the conducting polymers, the blends of PANI have technological importance due to their remarkable properties such as ease of synthesis, environmental stability, solubility and processability. Recently, the protonation of PANI with organic sulphonic acids was reported for the preparation of electrically conducting polymers, which is used for making conductive blends that have various applications [1].

The aim of this paper is to study the effect of doping, done with sulfamic acid in aqueous THF and to prepare its blend with polyvinyl chloride (PVC). Although, PVC has been used previously in the blending of polymers [2], our purpose for selecting PVC is that it is compatible with many different kinds of additives and can be plasticized to make it flexible for use in desirable applications. PVC has good flexibility, a smooth surface, and non-toxic qualities. Different dopants including Cu^{+1} , tellurium, etc., have been used for the doping of PANI [3, 4] but in our work PANI is doped with amidosulfonic acid (sulfamic acid) in order to increase the conductivity of the blends. In this paper, attempts were made to combine the mechanical properties of PVC with the electrical properties of conducting PANI by preparing blended films in order to increase applications [5].

2 Experimental

The polymerization of aniline to polyaniline was conducted by oxidative polymerization of aniline in the presence of an inorganic protonic acid such as HCl (1N) using ammonium peroxydisulphate as the oxidant [6]. The solution was stirred for 4-6 hr. The polymer thereby obtained was filtered and undoped by using aqueous ammonia resulting in the formation of emeraldine base (EB).

Different concentrations of sulfamic acid (dopant) in aqueous THF, including 5 %, 10 %, 15 %, 20 %, and 25 % were used to dope PANI. The sulfamic acid-doped PANI was added to the THF solvent and mechanically stirred constantly for up to 30 min in order to swell the doped PANI in the solvent. PVC solution (10 %) in THF was added to the doped PANI-THF solution and stirred vigorously. The doped PANI-PVC solution was cast in a smooth Petri dish. The THF solvent evaporated in about three hours leaving behind the

doped PANI-PVC blended films. Different doped PANI-PVC blended films were prepared by changing the amount of PANI loaded (doped with different concentrations of sulfamic acid) into a fixed amount of PVC solution (resin).

2.1 Characterization

Electrical dc conductivity was measured using a two-probe technique in the temperature range 300–400 K. Bulk samples of the doped PANI-PVC blended films were mounted between two steel electrodes inside a metallic sample holder. The voltage, 1.5V from a regular dc supply, was applied across the samples (films) and the resulting current was measured using a Keithley Electrometer (Model 617). To avoid any effect of moisture absorption, measurements were made on the annealed samples in vacuum (10^{-3} Torr) [7]. Fourier Transform-Infrared (FT-IR) spectra of doped PANI-PVC blended films were obtained using a Perkin-Elmer FT-IR Spectrometer in the range from 4000–400 cm^{-1} at a resolution of 4 cm^{-1} . The structural changes of doped PANI-PVC blended films were characterized with FT-IR spectroscopy and are shown in Fig. 8a-d.

3 Results and discussion

Figs. 1 and 2 demonstrate the variation in dc conductivity (σ_{dc}) and activation energy (ΔE) for PANI doped in different ratios relative to a fixed ratio of PVC used for the preparation of PANI-PVC blends in the temperature range 300–400K. The temperature dependence of dc conductivity (σ_{dc}) for various samples of the PANI-PVC blended films, prepared by doping with different concentrations of sulfamic acid in aqueous THF in the temperature range 300–400K, are illustrated in Figs. 3–7. The results indicate that conduction occurs in these samples through an activated process consisting of two conduction levels [8]. However, at higher dopant concentrations, such as for PANI_{20%doped}-PVC and PANI_{25%doped}-PVC, only one conduction level is observed. The dc conductivity can therefore be expressed by the relationship

$$\sigma_{dc} = \sigma_o \exp(-\Delta E/K_B T) \quad (1)$$

where ΔE is the activation energy of dc conduction, K_B is the Boltzmann constant, σ_o is the pre-exponential factor, and T is the temperature in degrees Kelvin. The calculated values of ΔE and σ_o are given in Tables 1–5 for samples doped at 5, 10, 15, 20, 25 %.

The conductivity of undoped PANI was determined to be $3.62 \times 10^{-14} \text{ohm}^{-1} \text{cm}^{-1}$. The conductivity of PANI increased as the concentration of dopant increased. However, the conductivity of the blends increased as the amount (%) of doped PANI increased in the blend. A noticeable difference in the conductivity, on the order of 10^{-2} for the PANI_{5%doped}-PVC blend, is noticed in the samples between the lowest concentration of doped PANI (the PANI_{5gm}-PVC_{100gm} blend) and the highest concentration of doped PANI (the PANI_{20gm}-PVC_{100gm} blend). The greatest increase in conductivity was observed for the highest concentration of doped PANI in the PANI-PVC blends (Table 2). Tables

1-5 illustrate that conductivity increased as the concentration of doped PANI in PANI-PVC blends increased. The increase of dc conductivity with a corresponding decrease in activation energy was determined to be associated with the shift of Fermi level in doped samples.

Some of the plots of $\ln\sigma_{dc}$ versus $1000/T$ in the temperature range 300-400 K (Figs. 3-7) are described by a single line, while in other plots, a deviation in slope is observed at a temperature about 339-349 K, above and below which the plots are described by two distinct lines of differing slopes indicative of a two-phase process. Two activation energies (ΔE_1 , ΔE_2) were observed for the 5, 10, and 15 % doped blends (Tables 1-3) corresponding to the two phases illustrated by the graphs (Figs. 3-5) plotted of $\ln\sigma_{dc}$ versus $1000/T$. Tables 1-3 show that ΔE_1 increased as the concentration of dopant increased in the PANI-PVC blends. A decrease in ΔE_2 was observed for the higher concentrations of dopant in the PANI-PVC blends which may be explained on the basis of crystallinity. The crystallinity of PANI increased as the concentration of dopant increased. The increased crystallinity of PANI is responsible for an increase in grain size. Ultimately, grain boundaries increase and thus space charges are created at the grain boundaries. At a lower temperature range, the resistance of the grain boundaries is large and therefore, conduction is low and results in a decrease in ΔE_2 . As temperature increases, the grain boundary resistance decreases, and thus conductivity increases. Therefore, at a higher temperature range, ΔE_1 increased as the conductivity of the blends increased. A single activation energy (ΔE) is observed for PANI doped with higher concentrations of sulfamic acid in PANI-PVC blends (Tables 4 and 5) which is due to the single phase illustrated by the graphs in Figs. 6 and 7. The single activation energy observed for the PANI-PVC blends doped at 20 and 25 % is less than ΔE_1 for the PANI-PVC blends doped at 5, 10, and 15 % and is greater than ΔE_2 . Figs. 3, 4, and 5 illustrate the two phases of the graphs plotted between $\ln\sigma_{dc}$ versus $1000/T$ for the 5, 10, and 15 % doped blends. However, only a single phase is observed for the PANI_{20%doped}-PVC, and the PANI_{25%doped}-PVC blends (Figs. 6 and 7) which are not linear.

One, single value of the pre-exponential factor was observed for the highly doped samples, i.e. the PANI_{20%doped}-PVC and the PANI_{25%doped}-PVC blends whereas two values of the pre-exponential factor were observed for the PANI_{5%doped}-PVC, the PANI_{10%doped}-PVC and the PANI_{15%doped}-PVC blends. Here, the values for the single σ_o measured for the PANI_{20%doped}-PVC and the PANI_{25%doped}-PVC blends were lower than σ_{o1} and were greater than σ_{o2} for the PANI_{15%doped}-PVC, the PANI_{10%doped}-PVC and the PANI_{5%doped}-PVC blends.

Using a simple two-phase model, the net σ_{dc} of PANI-PVC blends can be determined by equation 2 by considering the current flow parallel to the sample [9],

$$\sigma_{dc} = \sigma_1(x_1) + \sigma_2(x_2) \quad (2)$$

where, σ_{dc} is the conductivity of the blend, σ_1 and σ_2 are the conductivities of PVC and PANI, respectively, and x_1 and x_2 are the concentration of PVC and PANI, respectively.

Because $\sigma_2 \gg \sigma_1$, the net conductivity will increase linearly as the PANI concentra-

tion, x_2 , increases. Polarons exist in conjugated polymers and play an important role in the conductivity of conjugated polymers because they serve as hopping sites for the charge carriers. The dc conductivity can therefore, be explained in terms of hopping conduction. The conduction of the doped samples was explained in terms of the pre-exponential factor. Mott [10] suggested that the value of the pre-exponential factor, σ_o , for conduction in the localized states should be approximately $10^3 \text{ ohm}^{-1} \text{ cm}^{-1}$ which is two to three orders of magnitude smaller than for conduction in the extended states, and should be smaller still than for conduction in the localized states near the Fermi level. As reported in this present work, in some samples, two values of the pre-exponential factor, i.e. σ_{o1} and σ_{o2} , existed. The magnitude of σ_{o2} in the present work indicates that conduction is occurs by a hopping process due to the wide range of localized states near the Fermi level. However, considering the values of σ_{o1} , the possibility of conduction in extended states cannot be completely ruled out [11]. The temperature dependence of σ_{dc} can also be described by the following relationship:

$$\sigma_{RT} = \sigma_o \exp[-(T_o/T)^a] \quad (3)$$

where T_o is the characteristic temperature which represents the disorderliness in the samples, σ_o is a moderately temperature-dependent pre-exponential factor and a is the constant given by

$$a = 1/d + 1 = 1/2 \quad (4)$$

where d is the dimensionality. The value of a is equal to 1 for crystalline semiconductors. If the values of $a = 1/3$ and $a = 1/2$ are assumed, this dependence is valid for two- or one-dimensional conduction in which hopping conduction occurs. This type of co-relationship was determined to be valid for all doped conducting polymers [12].

3.1 FTIR studies

IR spectra were recorded for pure PVC, PANI_{undoped}-PVC blends and for PANI_{20%doped}-PVC blends and are illustrated in Figs. 8a, 8b, 8c, and 8d. The weak absorption band at 3080.4 cm^{-1} is assigned to aromatic C-H stretching vibrations, which shifts to 3080.9 cm^{-1} (noticed for pure PVC). The aromatic C-H stretching vibration between ($3080.4\text{-}3080.9 \text{ cm}^{-1}$) is observed for pure PVC and for lower concentrations of undoped PANI in the PANI_{2.5%undoped}-PVC blend [13]. No such peak is observed in the IR spectra for higher concentrations of undoped PANI in the blend as well as for doped PANI in the PANI-PVC blend. The broad, moderately strong band at 2876.6 cm^{-1} for the PANI_{2.5%undoped}-PVC blend is assigned to asymmetric C-H stretching vibrations in methylene groups, which shifts to 2961.7 cm^{-1} in pure PVC. When this absorption band shifts from 2876.6 cm^{-1} to 2961.7 cm^{-1} , splitting is observed in the band [14]. The small intensity band at 2723.4 cm^{-1} is assigned to symmetric C-H stretching vibrations in the octyl group of dioctylphthalate (DOP). DOP is used as a plasticizer for the preparation of the blends. This band is present only in PVC and in the undoped PANI -PVC blends. Such absorption bands are not present in the IR spectra of doped PANI-PVC blends, which

indicates that symmetric C-H stretching vibrations are present only in pure PVC and in undoped PANI-PVC blends. However, as the concentration of the dopant increased, the C-H stretching diminished. The broad, moderately strong band observed in the range of $1710.64\text{--}1752.6\text{ cm}^{-1}$ is assigned to C=O stretching. The C=O stretching is due to the presence of DOP and is observed for every sample. The weak band at 1570.4 cm^{-1} observed in the spectrum for the PANI_{20%doped}-PVC blend is characteristic of Nitrogen-Quinoid-Nitrogen[N-Q-N] stretching and is absent in pure PVC and in the PANI_{undoped}-PVC blends. The presence of this band in the PANI_{doped}-PVC blends is due to the substitution of dopant into the PANI chain. The broad bands at 1446.8 cm^{-1} – 1480.9 cm^{-1} are due to stretching of Nitrogen-Benzenoid-Nitrogen[N-B-N]. This broad band appeared only in the IR spectra for pure PVC and for the PANI_{2.5%undoped}-PVC blend. However, no such band is observed as the percentage of PANI increases in the PANI-PVC blends. The small broad band at 954.6 cm^{-1} in the IR spectrum for the PANI_{2.5%undoped}-PVC sample is assigned to C-H, in-plane bending, which is shifted to 978.7 cm^{-1} in the IR spectrum of pure PVC [15]. The characteristic peaks at $600\text{--}800\text{ cm}^{-1}$ are due to C-H, out-of-plane bending vibrations while the weak peaks at $400\text{--}500\text{ cm}^{-1}$ result from S-S stretching.

4 Conclusion

In the present research, the synthesis of PANI, its doping with different concentrations of sulfamic acid dopant, and the preparation of blends of doped PANI with PVC were accomplished. The dc conductivity of undoped PANI; doped PANI, and blends of PANI with PVC was measured in order to determine the effect of dopant on the conductivity of PANI. The blends were prepared in order to combine the mechanical properties of PVC with the electrical properties of conducting PANI doped with sulfamic acid. The conductivity of the blends increased as the concentration of highly-doped PANI in the blends increased. The conduction mechanism of the PANI-PVC blends was interpreted using a two-phase model. The calculated value of σ_{02} indicated that the conduction occurred by a hopping process due to a wide range of localized states near the Fermi level. However, considering the magnitude of σ_{01} , the possibility of conduction in extended states cannot be completely excluded. The conductivity of PANI decreased with the addition of PVC in the blend formation. These blends have good mechanical strength, which increases their utility for commercial applications. PANI-PVC blended films demonstrated the advantages of combining the mechanical properties of PVC with conductivity of doped Polyaniline.

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Table 1

PANI _{5%doped} -PVC BLENDS [w/w]	σ_{dc}	ΔE_1	ΔE_2	σ_{01}	σ_{02}	T_o	σ_{RT}
PANI _{5gm} -PVC _{100gm}	5.6×10^{-14}	1.75	2.98×10^{-1}	2.37×10^{13}	1.84×10^{-9}	3.5×10^4	0.201×10^{-12}
PANI _{10gm} -PVC _{100gm}	1.3×10^{-13}	1.58	5.17×10^{-1}	1.16×10^{10}	9.0×10^{-6}	0.078×10^4	0.166×10^{-12}
PANI _{15gm} -PVC _{100gm}	3.83×10^{-13}	0.0025	0.60×10^{-1}	4.23×10^{-13}	3.12×10^{-12}	0.145×10^4	3.94×10^{-12}
PANI _{20gm} -PVC _{100gm}	3.52×10^{-12}	0.911	0.69×10^{-1}	5.12×10^2	1.14×10^{-10}	0.081×10^4	2.420×10^{-12}

Table 2

PANI _{10%doped} -PVC BLENDS [w/w]	σ_{dc}	ΔE_1	ΔE_2	σ_{01}	σ_{02}	T_o	σ_{RT}
PANI _{5gm} -PVC _{100gm}	0.007×10^{-12}	2.62	55.5×10^{-2}	6.60×10^{24}	1.85×10^{-6}	1.33×10^5	0.059×10^{-12}
PANI _{10gm} -PVC _{100gm}	0.48×10^{-12}	1.52	9.56×10^{-2}	4.73×10^{10}	1.36×10^{-11}	0.039×10^5	0.264×10^{-12}
PANI _{15gm} -PVC _{100gm}	0.50×10^{-12}	1.48	3.61×10^{-2}	3.69×10^9	1.77×10^{-12}	0.005×10^5	0.589×10^{-12}
PANI _{20gm} -PVC _{100gm}	0.62×10^{-12}	1.24	10.8×10^{-2}	1.31×10^6	2.76×10^{-11}	0.050×10^5	1.120×10^{-12}

Table 3

PANI _{15%doped} -PVC BLENDS [w/w]	σ_{dc}	ΔE_1	ΔE_2	σ_{01}	σ_{02}	T_o	σ_{RT}
PANI _{5gm} -PVC _{100gm}	0.03×10^{-12}	2.27	4.73×10^{-2}	6.25×10^{18}	1.79×10^{-13}	0.948×10^3	0.031×10^{-12}
PANI _{10gm} -PVC _{100gm}	0.08×10^{-12}	2.17	6.02×10^{-2}	1.12×10^{18}	6.71×10^{-13}	1.53×10^3	0.084×10^{-12}
PANI _{15gm} -PVC _{100gm}	0.18×10^{-12}	1.67	14.2×10^{-2}	3.42×10^{11}	2.71×10^{-11}	8.79×10^3	0.132×10^{-12}
PANI _{20gm} -PVC _{100gm}	0.19×10^{-12}	1.03	9.64×10^{-2}	3.40×10^2	5.64×10^{-12}	3.99×10^3	0.151×10^{-12}

Table 4

PANI _{20%doped} – PVC BLENDS [w/w]	σ_{dc}	ΔE_1	σ_{01}	T_o	σ_{RT}
PANI _{5gm} -PVC _{100gm}	2.65×10^{-12}	0.866	3.55×10^1	1.85×10^5	1.237×10^{-12}
PANI _{10gm} -PVC _{100gm}	3.03×10^{-12}	0.072	1.21×10^{-10}	0.081×10^5	0.802×10^{-12}
PANI _{15gm} -PVC _{100gm}	3.9×10^{-12}	0.838	2.47×10^1	1.81×10^5	3.9×10^{-12}
PANI _{20gm} -PVC _{100gm}	7.2×10^{-12}	1.10	6.42×10^4	0.912×10^5	0.942×10^{-12}

Table 5

PANI _{25%doped} – PVC BLENDS [w/w]	σ_{dc}	ΔE_1	σ_{01}	T_o	σ_{RT}
PANI _{5gm} -PVC _{100gm}	0.76×10^{-12}	0.832	3.74×10	2.38×10^5	1.058×10^{-12}
PANI _{10gm} -PVC _{100gm}	6.3×10^{-12}	0.843	3.65×10^1	1.55×10^5	11.2×10^{-12}
PANI _{15gm} -PVC _{100gm}	11.2×10^{-12}	0.836	5.41×10^1	1.43×10^5	19.2×10^{-12}
PANI _{20gm} -PVC _{100gm}	1.74×10^{-11}	0.547	8.82×10^{-3}	1.14×10^5	4.416×10^{-12}

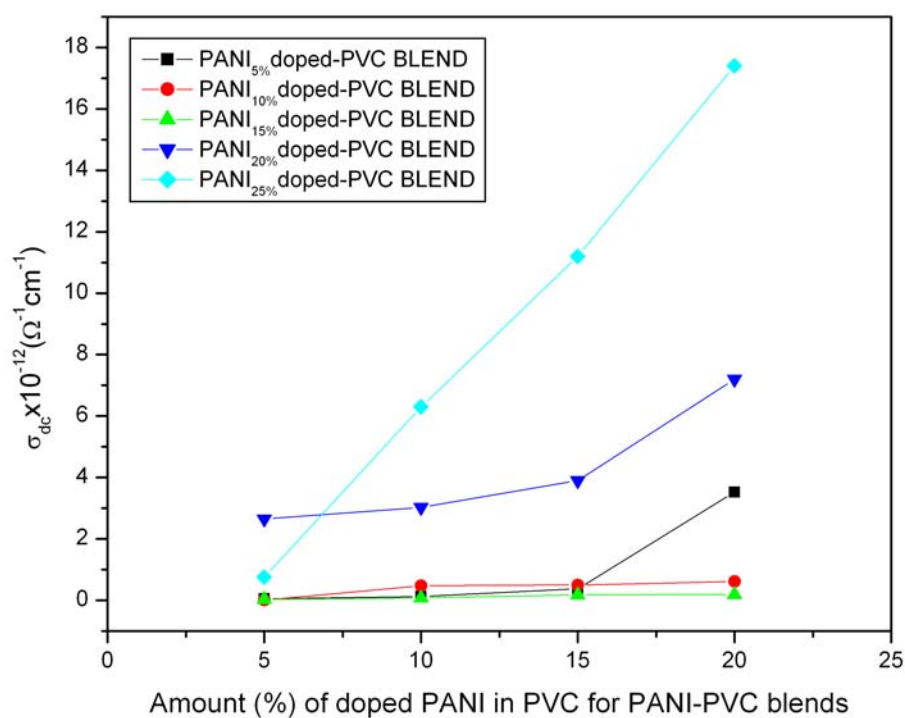


Fig. 1 Variation of dc conductivity for different ratios of doped PANI compared to a fixed ratio of PVC used in the preparation of PANI-PVC blends.

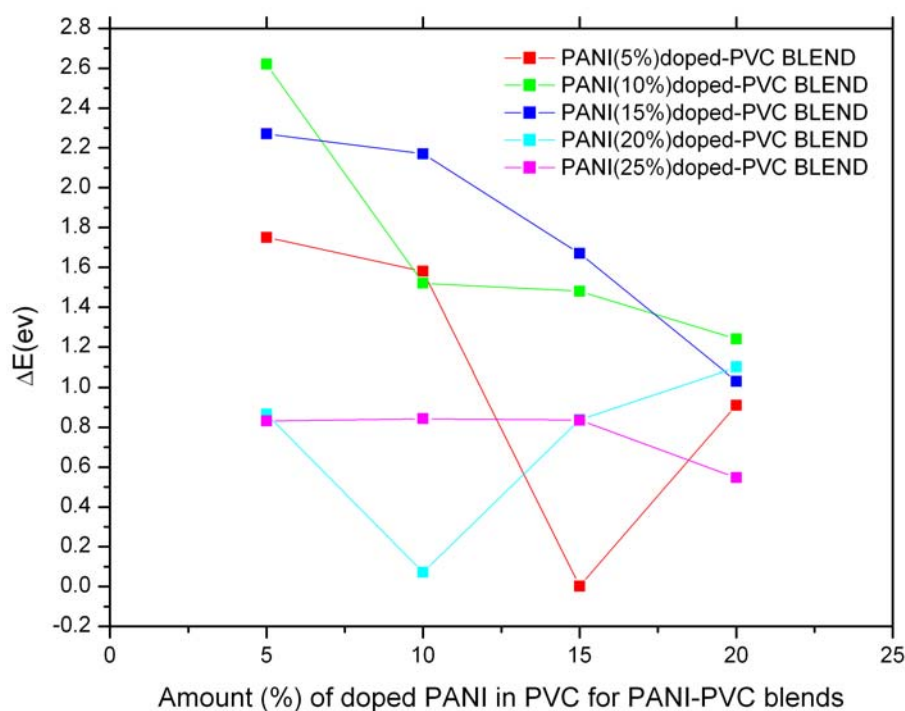


Fig. 2 Variation of ΔE for different ratios of doped PANI compared to a fixed ratio of PVC used in the preparation of PANI-PVC blends.

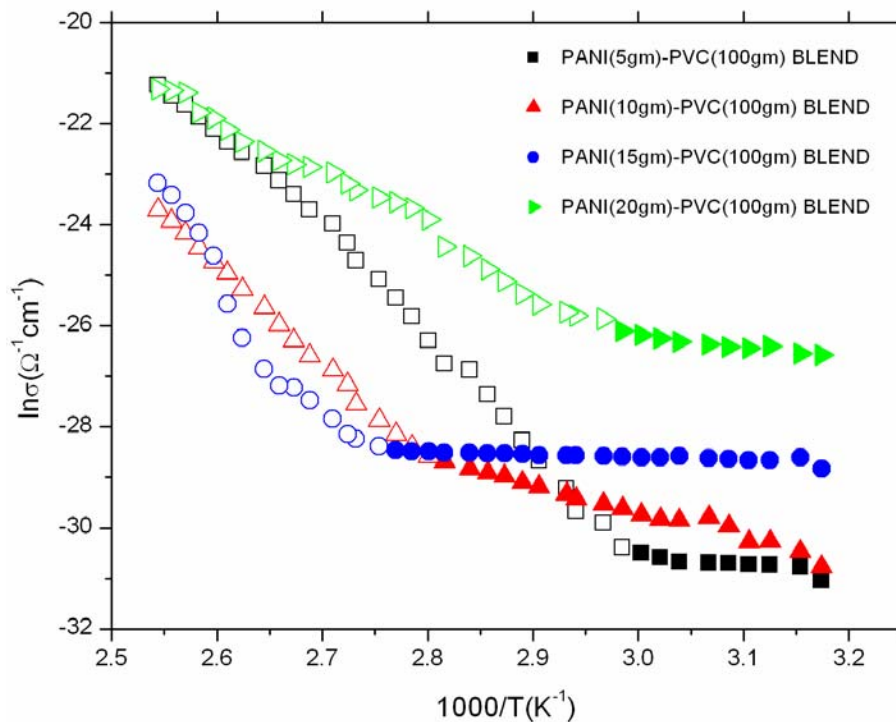


Fig. 3 Variation of dc conductivity with $1000/T$ for PANI doped with 5 % sulfamic acid in PANI-PVC blends.

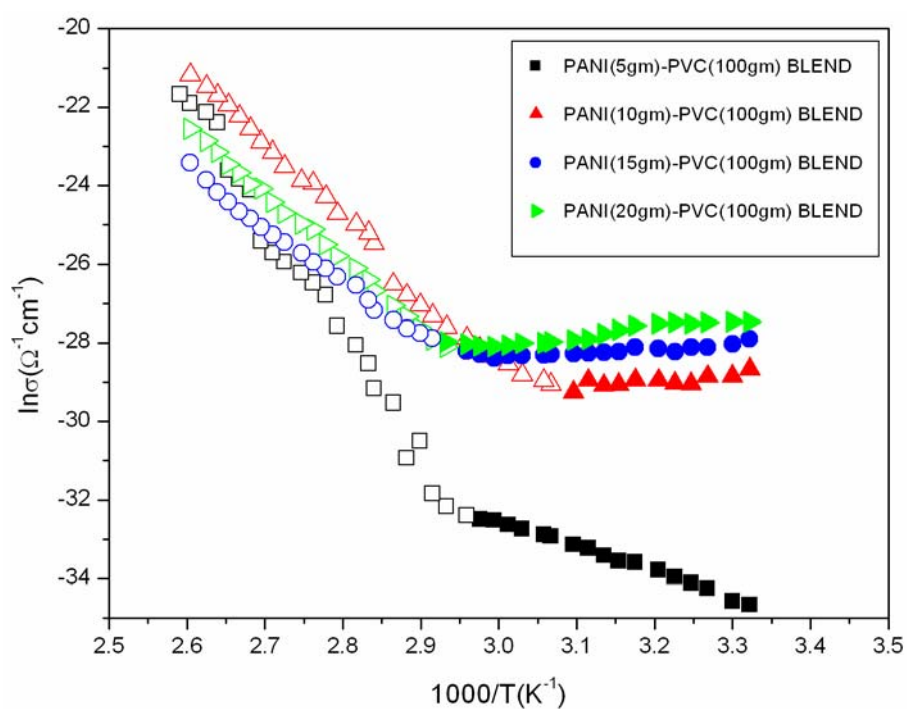


Fig. 4 Variation of dc conductivity with $1000/T$ for PANI doped with 10 % sulfamic acid in PANI-PVC blends.

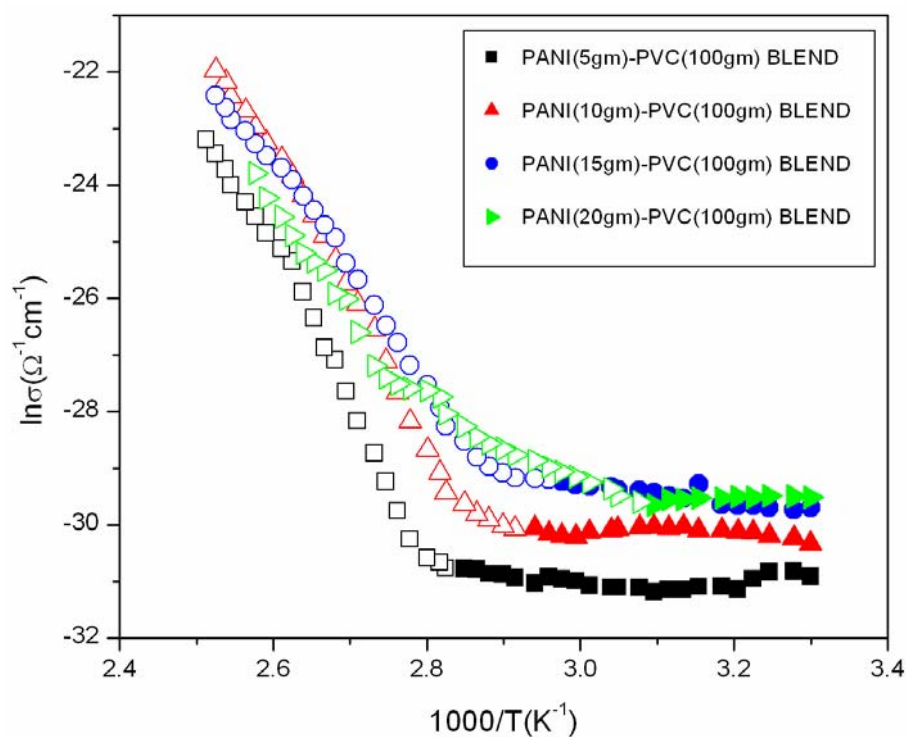


Fig. 5 Variation of dc conductivity with $1000/T$ for PANI doped with 15 % sulfamic acid in PANI-PVC blends.

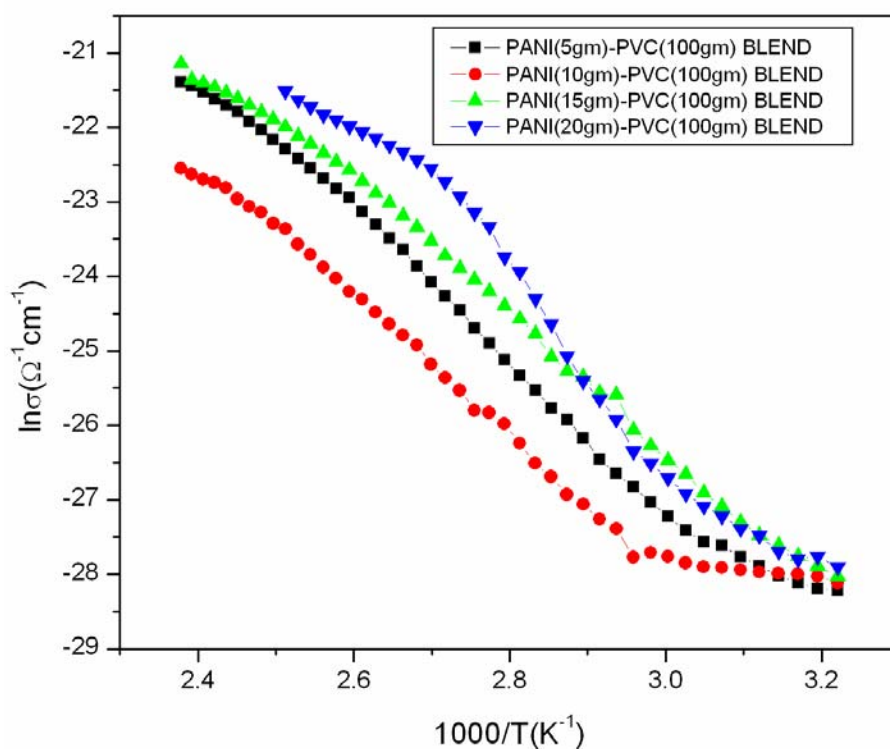


Fig. 6 Variation of dc conductivity with $1000/T$ for PANI doped with 20 % sulfamic acid in PANI-PVC blends.

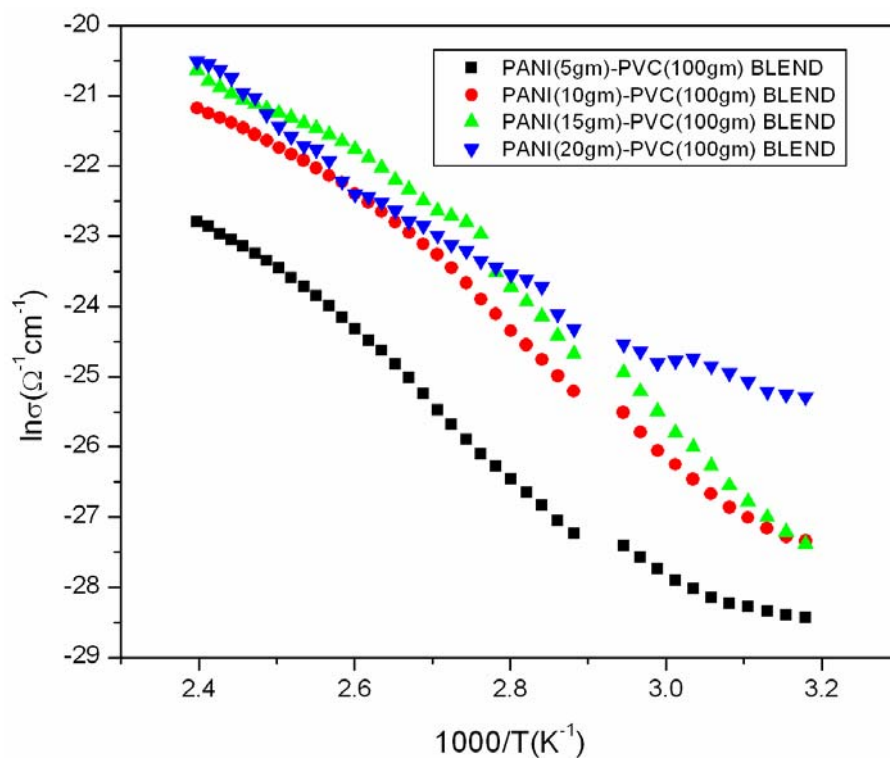


Fig. 7 Variation of dc conductivity with $1000/T$ for PANI doped with 25 % sulfamic acid in PANI-PVC blends.

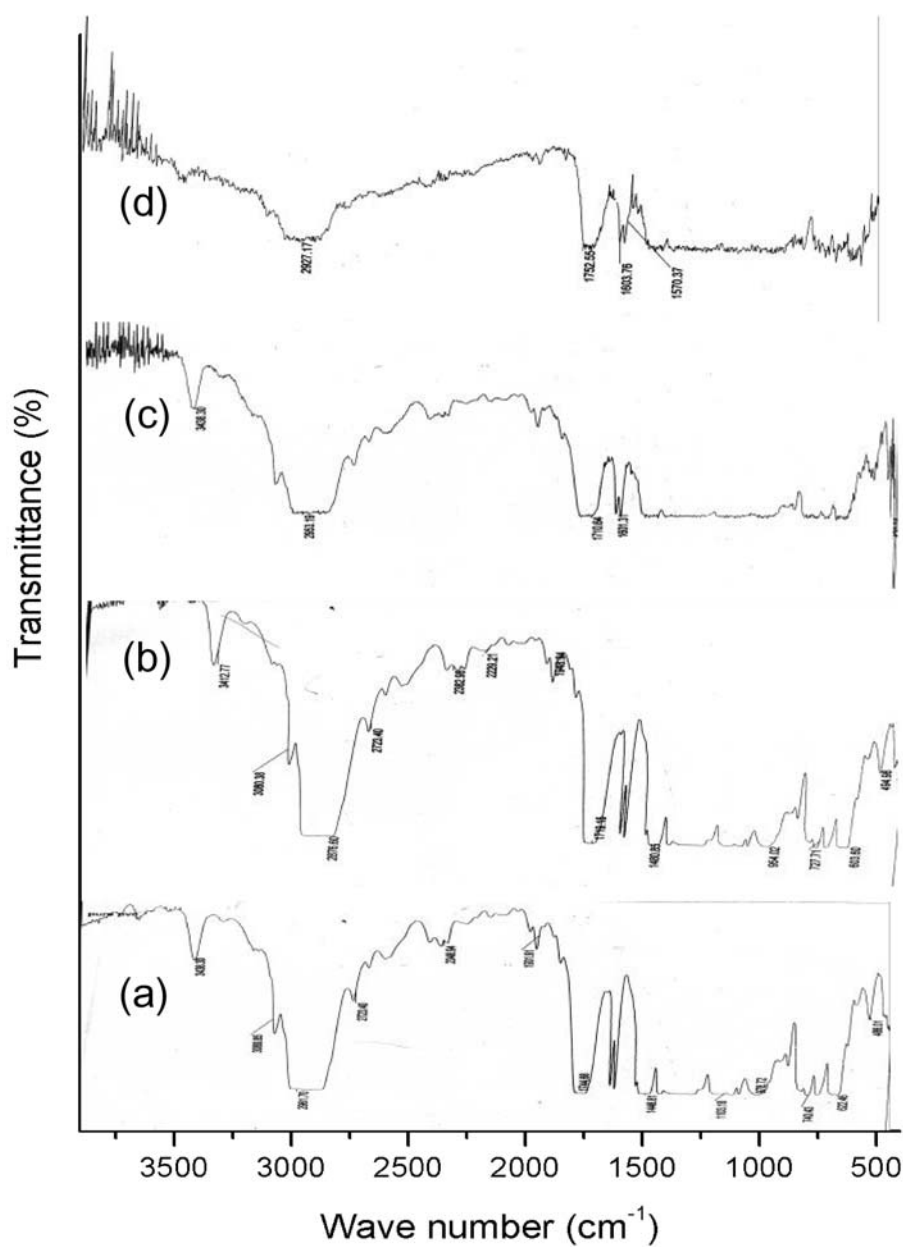


Fig. 8 FTIR spectra of (a) Pure PVC, (b) PANI_{2.5%undoped}-PVC (c) PANI_{20%undoped}-PVC, and (d) PANI_{20%doped}-PVC blends.