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Synthesis and study of electrical properties of polyaniline and polyester nanocomposite

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In this study, pure polyaniline (PANI) and PANI/polyester nanocomposites (wt % 10, 20 and 30) of thickness 10 mm have been synthesized by co-precipitation method to investigate their electrical properties. Pure PANI and its composites are then characterized using SEM, FTIR, DSC, I-V Hall and dielectric techniques. From SEM, it is observed that the particles are spherical and lying in range of micron order. To find out the nature of chemical bonds, FTIR analysis and thermal characterization (DSC) are performed. From the I-V curve, the calculated values of conductance of prepared pure PANI and PANI/PET (wt % 10, 20 and 30 nanocomposite) are obtained as 3.78×10^{-7}, 9.7933×10^{-7}, 9.584×10^{-8} and 9.882×10^{-9} ohm^{-1} respectively. From Hall measurement, electrical conductivity of pure PANI and PANI/PET (wt % 10, 20 and 30) nanocomposite is found 49.26×10^{-6}, 13.82×10^{-6}, 40.6×10^{-7} and 18.761×10^{-7} Ω^{-1}cm^{-1} respectively. Dielectric study of PANI/PET reveals downturn in the dielectric constant value with increased frequency. The electrical resistivity of the PANI/PET nanocomposite shows high anti-static efficiency.

Keywords: Dielectric constant, Electrical properties, Nanocomposite, PANI/PET composite, Polyester

Static electricity refers to the build-up of electric charge on the surface of objects. The static charges remain on an object until they either bleed off to ground or are quickly neutralized by a discharge. Discharge of static electricity can create severe hazards in those industries dealing with flammable substances, where a small electrical spark may ignite explosive mixtures[1]. Polyester fibre is broadly used for manufacturing filter fabric because of its superior strength, anti crease, good abrasion resistance and stable performance in acid and alkali medium. Polyester fibre is easy to acquire static charge on account of its hydrophobic nature. Charge density on polyester can be remodeled if the fibre surface is a fairly good conductor, the charges will be restock by flow along the fibre and charge will be vanished promptly. When polyester fibre is used in filter to separate solid particles, the flowing advancement of finely powdered materials or mechanical turbulence build-up static electricity, prevailing potential menace with assembled static charge. There are two likely clarifications why dust or lint particles which are pretended to be insulators are attracted towards a charged fabric. The dust grains may be charged themselves, few positive and few negative so that when they advance a charged fabric, the oppositely charged particles are engaged, complying basic law of electrostatics. If the particles are not charged, it is possible that electric dipoles are build up by the field from the charged fabric. These electric dipoles develop when the electrons constrained to the atoms of the particles are marginally displaced by the force of the field, giving surge to adequate build-up of counter charge adjacent to the fabric developing attractive force. In dust collectors, the electrostatic load can sprout both on filter media and on the dust cake; it is eased by low moisture levels, high temperatures, high contact velocities and small particles. Need of the electrically conductive textiles is promptly expanding in industrial uses like sensing, electrostatic discharge, corrosion protection, electromagnetic interference shielding, dust and germ free clothing, monitoring and data transfer in clothing[2-5].

The synthesis of intrinsically conducting polymers (ICPs) on PET fibre is an efficient way to improve its conductivity. Among the existing ICPs, polyaniline (PANI) has been widely studied on account of its economical cost, ease of synthesis, good electrical property, environmental stability, high electrical conductivity, simple acid-doping/base-dedoping chemistry, electrochemical activity, electrochomism and the ability to be electrically switched between its conductive and resistive states.

Conductive polymers like polyaniline are generally used to impart conductivity on conventional textile structures. This material has currently attracted much consideration due to its potential for many technological applications that could help from its

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exclusive polymeric and electronic properties[6-16]. In view of above, it is speculated to be significant if aniline can be polymerized *in situ* within a fibre structure by utilizing co-precipitation method. The present study is, therefore, aimed at synthesising and studying electrical/electronic properties of polyaniline (PANI) and PANI/PET nanocomposites.

Experimental

Polyester fibre (powder form) was used for making composite. Solutions used for formulation of PANI were aniline ($C_6H_5NH_2$), ammonium peroxidisulphate $(NH_4)_2S_2O_8$, HCl, methanol solution, acetone and NH_4OH, which were procured from Merc, India Ltd.

Synthesis of PANI

Solutions of $(NH_4)_2S_2O_8$ (25 mL) and $C_6H_5NH_2$ (10 mL) were prepared by dissolving them in double distilled water in a volumetric flask separately which was kept for 1 h at room temperature (300 K). The solution was then mixed with HCl (5 mL) by constant stirring and left for 6 h for polymerization.

After polymerization, dark green precipitate was obtained. This precipitate was treated with the NH_3 solution to get undoped polyaniline in emeraldine base. It was dried in oven at 60 °C for 1 h to obtain PANI. For though drying, it was preserved in oven at 80 °C for 2 h and converted to powder form by grinding it up to 4-6 h, finally sintered this powder in furnace up to 90 °C. The reaction mechanism of aniline and ammonium peroxidisulphate is illustrated in Fig. 1.

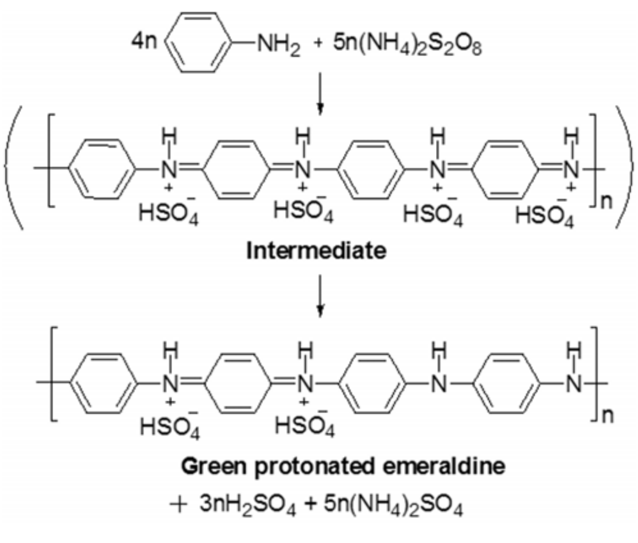

Fig. 1 — Reaction mechanism of aniline and ammonium peroxidisulphate

Synthesis of PANI/PET Nanocomposite

PANI/PET nanocomposite powder was processed by homogeneous mixing and grinding of different wt % (10, 20, and 30) of PET in PANI. From this powder, pellets of diameter 10 mm and thickness 1-3 mm were made by applying 2000-4000 kg/cm^2 pressure for 5 min using hydraulic pressure machine; finally the pellets were sintered in furnace at 110 °C.

Characterization

Above prepared samples were characterized by SEM, FTIR, DSC, I-V Hall and dielectric techniques. The surface morphological characteristics of samples were studied by scanning electron microscope (SEM) (Carl Zeiss EVO 50). Differential scanning callorimetry measurement was performed on Perkin Elmer STA 6000. In order to determine the internal chemical composition, samples architecture was characterized by Fourier transform infrared spectroscopy (FTIR). The dielectric properties were measured by Network analyzer (Model: HP 8719 ES) in the frequency range 2-7 Hz.

Results and Discussion

SEM Analysis

SEM micrographs [Figs 2 (a) and (b)] show the structure of pure PANI and PANI/PET. It can be seen that the shape and size of particles are spherical and lying in range of micron order respectively.

FTIR Analysis

In order to find out the nature of chemical bonds, FTIR spectra of pure PANI and its nanocomposite in transmission mode are recorded in the range 400–4000 cm^{-1} (Fig. 3).

The FTIR spectrum shows the characteristic peaks at around 2360, 1587, 1475, 1271, 1106, 1075, 996, 785, 690, and 560 cm^{-1}. The FTIR band at wavenumber 1,587 cm^{-1} shows the quinoid ring vibration and that at wavenumber 1475 cm^{-1} indicates benzonoid ring vibration for emerladine salt.

Three strong bands observed in 1500-1600 cm^{-1} region are mainly due to C=C in plane vibration and characteristic band at frequencies 650-1000 cm^{-1} is mainly due to C-H deformation. Characteristic band at frequencies 400-650 cm^{-1} is due to C-N bending in cyclic or aromatic compound.

DSC Analysis

Differential scanning calorimetry measurement was performed on Perkin Elmer STA 6000. Quantity of

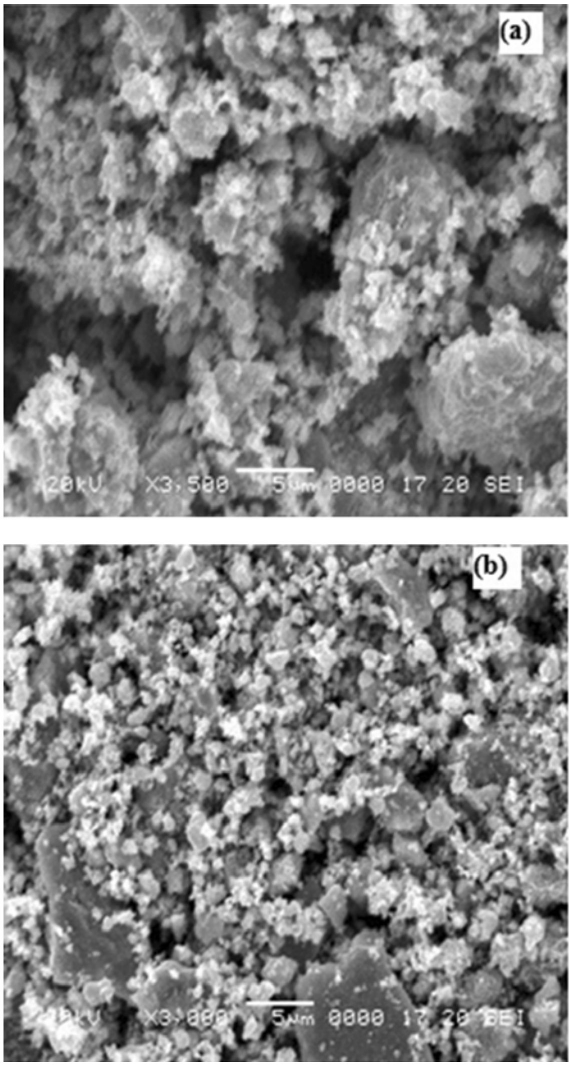

Fig. 2 — SEM images of (a) PANI and (b) PANI/PET (wt % 20)

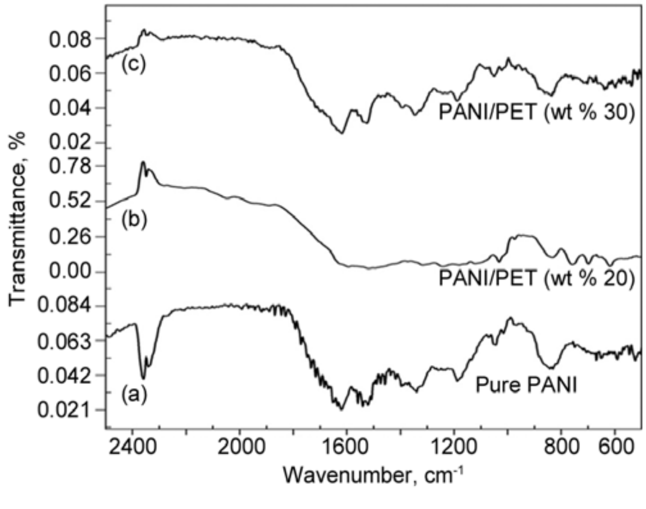

Fig. 3 — FTIR characteristics of (a) pure PANI, (b) PANI/PET (wt % 20), and (c) PANI/PET (wt % 30)

samples is varied between 5 mg and 30 mg. The samples are heated at a rate of 10° C/min from 50°C-150°C. Pure gas nitrogen is used in the experiment.

DSC analysis of pure PANI and its nanocomposite are as shown in Fig. 4. From DSC characterization no significant findings are observed.

Electrical/Electronic Characterization

I-V curves of PANI and PANI/PET nanocomposites annealed at room temperature is as depicted in Fig. 5. The calculated values of

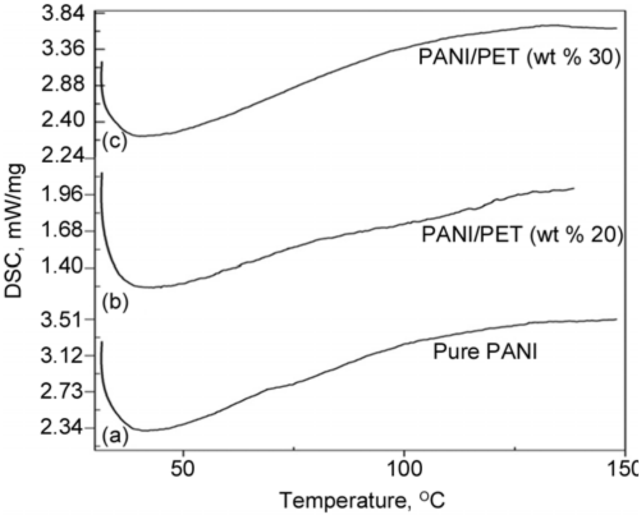

Fig. 4 — *DSC* curve for (a) pure PANI, (b) PANI/PET (wt % 20), and (c) PANI/PET (wt % 30)

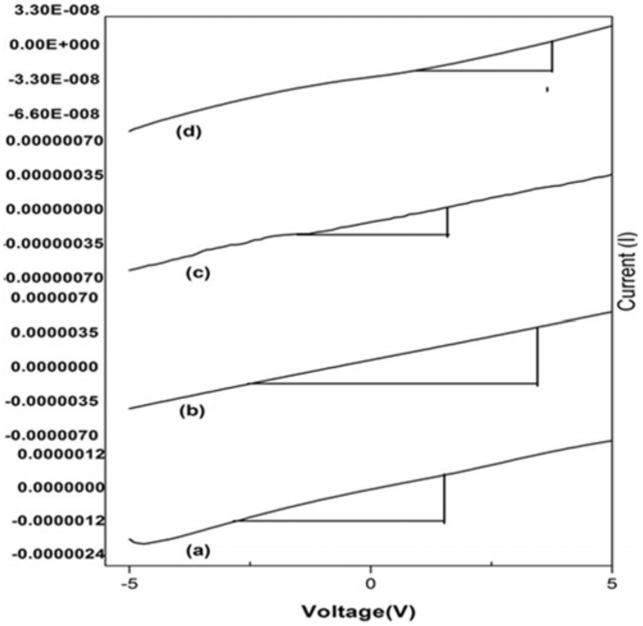

Fig. 5 — IV characteristics for (a) pure PANI, (b) PANI/PET (wt % 10), (c) PANI/PET (wt % 20), and (d) PANI/PET (wt % 30)

Table 1 ─ Electrical properties of pure PANI and PANI/PET annealed at different wt %

Sample	Bulk carrier concentration (N_b), cm^3	Mobility (μ) $cm^2 V^{-1}s^{-1}$	Resistivity (ρ), Ω-cm	Hall coefficient (R_H)	Conductivity (σ), $Ω^{-1}cm^{-1}$
Pure PANI	$1.375×10^{10}$	$8.320×10^2$	$2.032×10^4$	$5.128×10^7$	$49.26×10^{-6}$
PANI/PET (wt % 10)	$2.075×10^{11}$	$4.160×10^2$	$7.232×10^4$	$3.008×10^7$	$13.82×10^{-6}$
PANI/PET (wt % 20)	$4.191×10^{11}$	$2.795×10^1$	$5.330×10^5$	$1.489×10^7$	$18.761×10^{-7}$
PANI/PET (wt % 30)	$1.070×10^{12}$	$2.493×10^1$	$2.493×10^5$	$5.835×10^6$	$40.6×10^{-7}$

conductance of prepared nanocomposites of PANI and PANI/PET (wt % 10, 20 and 30) are found $3.78×10^{-7}$, $9.7933×10^{-7}$ ohm^{-1}, $9.584×10^{-8}$ ohm^{-1}, $9.882×10^{-9}$ ohm^{-1} respectively, which is found to decrease with increased doping % of PET in composite.

To measure the electrical properties of the samples, Keithley's Hall effect switching card, voltmeter (2182A) and constant-current source (2400) system are used. Hall measurements are carried out at room temperature (300 K) for determining electrical resistivity (ρ), carrier concentration (n) and carrier mobility (μ) by four probe method employing Van der pauw geometry.

The samples are mounted on printed circuit board (PCB) containing four point spring loaded probes. The electrical properties of samples are measured at room temperature (300 K). The electrical conductivity of pure PANI and PANI/PET nanocomposite (wt % 10, 20, 30) is found to be $49.26×10^{-6}$, $13.82 ×10^{-6}$, $18.761×10^{-7}$ and $40.6 ×10^{-7}$ $Ω^{-1}cm^{-1}$ respectively. It is evident that as wt % of PET in PANI matrix increases, the electrical conductivity decreases by four orders of magnitude (Table 1). This change of conductivity may be associated with four orders of magnitude decrement in carrier mobility (μ) from $4.160x10^2$ $cm^2V^{-1}s^{-1}$ to $2.493x10^1$ $cm^2V^{-1}s^{-1}$. Although PANI/PET nanocomposite exhibits resistivity values as low as $7.232×10^4$, $5.330×10^5$ and $2.493×10^5$ Ω-cm, they cannot be classified as conductive materials. However, they can be referred to as anti-static materials. According to the standards of the Electronic Industry Association, a volume resistivity of less than $1 ×10^4$ Ω-cm is required for a material to be classified as conductive; volume resistance values of dissipative materials must be equal to or greater than $1×10^4$ Ω-cm but less than $1×10^{11}$ Ω-cm and the volume resistivity of insulative materials must be at least $1×10^{11}$ Ω-cm [17,18].

Di-electric Measurement

The behaviours of real part of permittivity (ε') of pure PANI and PANI/PET nanocomposite as function of frequencies (2-7 Hz) are shown in Fig. 6. The electric permittivity (ε') values decrease with increasing frequency. Above certain frequency, the permittivity (ε') exhibits flat region [Figs 6(a)-(d)]. This may be due to space charge polarization.

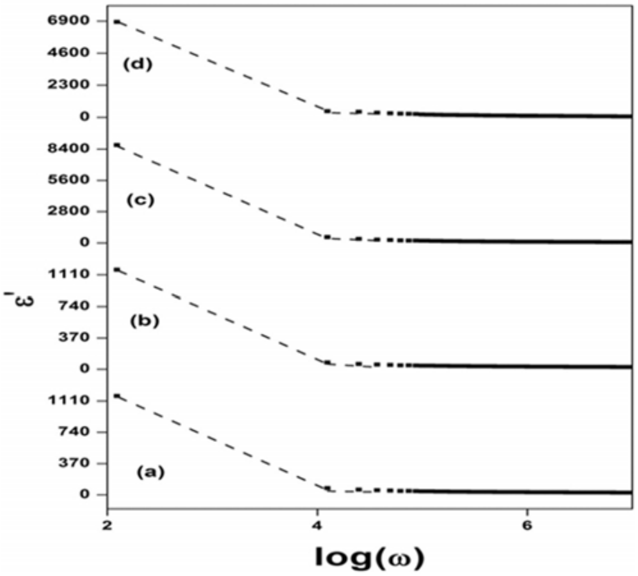

Fig. 6 ─ Dielectric constant for (a) pure PANI, (b) PANI/PET (wt % 10), (c) PANI/PET (wt % 20), and (d) PANI/PET (wt % 30)

The reason for such trend may be due to increased sample conductivity upon doping, thus behaving improved dielectric system at lower frequencies. Graphical trend (Fig. 6) between ε' and log (ω) is similar for all samples. It can be observed from Fig. 6 (b) that dielectric constant (ε') of PANI/PET (wt % 10) has maximum of 1200 F/cm, while for PANI/PET (wt % 20 and 30) [Figs 6 (c) and (d)] has increased ~ 6 times with respect to PANI/PET (wt % 10) and ~ 20 times with respect to PANI/PET (wt % 20).

From SEM, it is observed that particles are spherical and lying in range of micron order. FTIR spectrum at wavenumber 1,587 and 1475 cm^{-1} exhibits the quinoid ring vibration and benzonoid ring vibration for emerladine salt. From DSC characterization, no significant findings are observed. From I-V curve, the calculated conductance of prepared pure PANI and PANI/PET nanocomposite (wt % 10, 20, 30) is found

3.78×10^{-7}, 9.7933×10^{-7}, 9.584×10^{-8} and 9.882×10^{-9} ohm^{-1} respectively. Resistance obtained from the I-V curve is increasing with increasing wt % of PET in PANI.

From Hall study, electrical conductivities of pure PANI and PANI/PET (wt % 10, 20, 30) nanocomposite are found 49.26×10^{-6}, 13.82×10^{-6}, 40.6×10^{-7} and 18.761×10^{-7} Ω^{-1}cm^{-1} respectively. The dielectric study of PANI/PET composite affirms the increase of dielectric constant. The electrical resistivity of the PANI/PET nanocomposite exhibits high anti-static efficiency. Thus, the current study indicates an enhancement in compatibility between PANI/PET nanocomposite, hence subsequently showing anti static properties.

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