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## Electromagnetic shielding behaviour of conducting polyaniline composites

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#### Abstract

The paper reports the designing and development of conducting polyaniline composites which show a shielding effectiveness of 4-58 dB against electromagnetic interference at 101 GHz range, depending upon the loading of the conducting polyaniline in polystyrene and polymethylmethaacrylate matrix. The composites can be used for the dissipation of static charge. A static decay time of the order of 0.11-0.02 s for the conducting polyaniline composites is observed when the charge is reduced from 5000 to 500 V. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Conducting polymer; Polyaniline; Conducting composites; Antistatic behaviour; Static decay time; Shielding effectiveness

#### 1. Introduction

Conducting polymers have emerged as an important class of electronic materials because of their potential and wide applications in energy storage systems [1,2], optoelectronic devices [3,4], organic light emitting diodes [5-8], sensors for hazardous gases and toxic fumes [9,10], corrosion inhibitors for iron and mild steel [11,12], EMI shielding in radio frequency range and microwave range [13,14], super capacitors and super conductors [15,16]. The study of the conducting polymers is a subfield of the larger older field of organic electrical conductors which had already started in early 1970s, with the discovery of  $(SN)_r$ . These organic materials possess electronic conductivity comparable to metals and semiconductors. The conducting polymer, polyacetylene, discovered by Shiakawa, Heeger and MacDiarmid [17] has electronic conductivity of the order of 10<sup>5</sup> S/cm [18] whereas the conductivity of copper is  $10^6$  S/cm. With the idea that electronic conductivity can be varied with doping has revolutionized the area of research. They acquire importance over inorganic semiconductors in their application because of their high strength to weight ratio, toughness, low cost and ease of processing into film. The prospect of plastic metals has inspired much interest in

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these materials for technological applications such as antistatic coatings and electromagnetic interference shielding and in other areas where light weight, flexibility and high conductivity materials are required. Among conducting polymers, polyaniline and its analogues have been widely studied due to its ease of protonic acid doping in the emeraldine form and its environmental stability in both doped and undoped forms. Conducting polymer, polyaniline, exists in various forms (Fig. 1) and each form finds various technological applications. The fully reduced form of polyaniline is leucoemeraldine, which finds applications in Li-polyaniline battery and electrochromic devices. 50% reduced and 50% oxidized form of polyaniline is termed as emeraldine base which is the insulating form of polyaniline. It finds application in sensors for HCl gas and as corrosion protective coating on iron and mild steel. Doping of emeraldine form leads to the formation of conducting polyaniline, which is used as an electrode material in batteries, sensors, EMI shielding and electrochromic devices. The fully oxidized form of polyaniline is pernigraniline, which find applications in non-linear optics. Conductivity in polyaniline depends on the degree of protonation of the material. Unlike other phenylene based conducting polymers, polyaniline has a reactive NH group in the polymer chain flanked either side by a phenylene ring which imparts very high chemical flexibility to the system. The involvement of hetero atom in the conduction



mechanism makes polyaniline different from other conducting polymers like polypyrrole, polythiophene and

polyfuran, whose hetero atom do not contribute significantly to  $\pi$ -band formation. Electromagnetic interference is one of the unfortunate byproducts of the rapid proliferation of electronic devices.

byproducts of the rapid proliferation of electronic devices. Owing to the aesthetic appeal of plastics, they have replaced metal cabinets of electronic housings. However, plastics are transparent to electromagnetic radiations and cannot be earthed to provide electrostatic control. At present, the most effective means of controlling electromagnetic interference and electrostatic charge dissipation is to use various types of conducting composites having conducting fillers such as metal fibers, metal particulates, carbon black and carbon fiber. Carbon fiber and metal filled fiber filled polyethylene composites are reported to have poor performance for EMI shielding [19]. Similar case has been reported for metal filled composites of polypropylene [20].

The processability in polyaniline can be achieved by making conductive composites of polyaniline with conventional polymers [21–27]. The present paper reports the preparation of conductive composites of conducting polymer polyaniline with thermoplastics like polystyrene (PS) and poly(methyl methacrylate) (PMMA) wherein the composites retain the mechanical properties of the conventional polymers and the electrical conductivity of the conducting polymers.

#### 2. Experimental

The synthesis of polyaniline was carried out by chemical oxidative polymerization of 0.1 M solution of aniline in an organic protonic acid medium (pH 0.5-2.5). The acids used were *p*-toluene sulfonic acid (PTSA) and dodecylbenzene sulfonic acid (DBSA). Ammonium persulphate was

the oxidant used for the present study. The oxidant was added very slowly to the reaction vessel containing the monomer and the electrolyte kept at 0-1 °C and the stirring was carried for 4-6 h. The reaction conditions for obtaining conducting polyaniline are given in Ref. [28]. The characterization of the conducting polymer doped with PTSA and DBSA was carried out by spectroscopic techniques like UV–visible and FTIR. FTIR studies of the doped polyaniline was also carried out at different temperatures in order to see the effect of temperature on the shift of bands present in the polymer matrix.

### 2.1. Polyaniline–polystyrene and polyaniline–poly (methylmethacrylate) composites

The composites of conducting polymer polyaniline with conventional polymers PS and PMMA were made by melt blending technique in the temperature range of 180-220 °C in a brabender mixing bowl for 5 min at 100 rpm. The detail of the composite preparation is given in Ref. [29]. Different loading levels of polyaniline from 2 to 50% were blended with PS and PMMA and composites were compression molded into plates of 30 cm × 30 cm, 15 cm × 10 cm which were then used for studying the EMI behaviour, static decay time response and other electrical and mechanical properties. The mechanical properties of the composites were carried on a 5-ton Universal Testing Machine, Instron, Model 4204.

#### 2.2. Static decay time measurements

Static decay meter, model 406 D of Electro Tech System, USA was used to measure the electrostatic dissipation performance of conducting polymer composites. A 5000 V positive charge is first built up in the instrument. When the test button is depressed, the charge is transferred to the sample, between the electrodes. The time is then measured until the charge between the two electrodes is reduced to 500 V. After a gap of 5 s, the sample is charged again and the decay time is measured again. This is then repeated for three times. The sample is then neutralized and the process is repeated using a 5000 V negative charge. Three measurements are then recorded for a negative charge. The three positive values are averaged to final positive value and the three negative are averaged to final negative value.

#### 2.3. Static charge measurements

The static charge of the insulating and conducting composites was measured using Para Electronics static charge meter (SCM 1) mounted on an energy stand with a distance of 2.5 cm from energy dish whose capacitance at this distance was 25 pF. The static charge in coulombs was

calculated using the relation

$$Q = CV$$

where C is the standard capacitance of the base plate and V is the voltage indicated on the static charge meter. The static energy, E, was calculated using the relation

$$E = \frac{1}{2}CV^2$$
 (in joules)

#### 2.4. EMI measurements at 101 GHz

The electromagnetic interference measurements at 101 GHz were carried out using a phase log oscillator of 101 GHz frequency generator, Model No. 956 W4-010-101, S/N 033 using conical horned antenna, Model No. 458264-1031, S/N 012 and mm wave power receiving set up consisting of pyramidal horned antenna, Model No. 861 W/387, S/N 483. The shielding effectiveness was measured by noting the power with and without the samples by placing them near to the surface of the antenna.

#### 2.5. Thermal stability and electrical conductivity behaviour

Thermo gravimetric analysis study of the conducting polymer polyaniline was carried out using Perkin Elmer 7 TGA analyzer in the temperature range of 50–800 °C in nitrogen atmosphere at a heating rate of 20 °C/min. The electrical conductivity measurements of the conducting polymer and its composites were carried by two/four probe method. In two probe method, surface resistivity was measured by placing two copper strips on the sample at a distance of 1 cm and noting down the corresponding resistance using a multimeter MECO 801. In four probe method, two terminals of the probes were connected with the current terminals and two pins of the probes were connected with the voltage terminals and so consequently the resistivity/conductivity of the samples were determined.

#### 3. Results and discussion

The polymerization of aniline to polyaniline in the presence of organic protonic acids like PTSA and DBSA may bring certain changes in the properties of polyaniline because conduction mechanism in polyaniline involves protonation as well as ingress of counter anions to maintain charge neutrality. Protonation and electron transfer in polyaniline leads to the formation of radical cations by an internal redox reaction, which causes the reorganization of electronic structure to give two semiquinone radical cations. In the doping process, ingress of anions occurs to maintain charge neutrality in the resultant doped polyaniline matrix. This implies that nature of anions should influence the properties of the resulting polyaniline. This is the reason why polyaniline doped with inorganic dopants like  $C1^-$  and  $SO_4^{2-}$  are thermally less stable than the polyaniline doped with

organic dopants like PTSA or DBSA. It has been observed that polyaniline doped with *p*-toluene sulphonate is thermally stable up to 228 °C whereas polyaniline doped with  $Cl^-$  is thermally stable up to 140–150 °C. This is the reason of blending polyaniline doped with organic dopants like PTSA or DBSA with conventional polymers like PS and PMMA where the blending is carried out at 180–220 °C.

Thermogravimetric analysis of the undoped emeraldine base (Fig. 2, curve a) and conducting polyaniline doped with PTSA (Fig. 2, curve b) shows that whereas the undoped polyaniline sample is stable up to 433 °C, the doped polyaniline is thermally stable up to 228 °C. TGA curve of emeraldine base shows negligible weight loss up to 433 °C and from 433 °C onwards, there is a sharp weight loss indicating that the degradation of the polymer backbone has taken place. In the doped conducting polymer sample, the two transitions in the TGA curves are observed. In the first transition from 228 to 310 °C, the weight loss is approximately 38% indicating that the dopant has come out from the polymer backbone or indirectly these are the dopant moieties present in the conducting polymer matrix. The second transition from 310 °C onwards shows a sharp weight loss, indicating the degradation of the polymer backbone. Similarly, the polymer doped with DBSA was found thermally stable up to 240 °C. Thus, polyaniline doped with PTSA and DBSA was used for the blending of conducting polymer with insulating polymer like PS and PMMA.



Fig. 2. Thermogravimetric analysis curve in the temperature range of 50-800 °C in nitrogen atmosphere at a scan rate of 20 °C/min; curve (a) emeraldine base and (b) polyaniline doped with *p*-toluene sulphonate.

Table 1 Slope and residues of isothermal run of conducting polyaniline doped with DBSA

Sample	Isothermal temperature (°C)	Slope 1 (wt%/min)	Slope 2 (wt%/min)	Residue at 30 min (wt%)
PANI-doped	100	-0.030	0.010	96.20
PANI-doped	150	-0.018	-0.003	96.09
PANI-doped	200	-0.038	-0.030	95.18
PANI-doped	250	-0.062	-0.047	93.17
PANI-doped	280	-0.622	-0.470	80.75

Isothermal studies of the conducting polymer doped with PTSA and DBSA were also carried at different temperatures in order to see the thermal stability of polyaniline at different temperature with respect to time. The polymer was run under isothermal conditions at 100, 150, 200, 250 and 280 °C. The initial program was run for a temperature ramp of 100 °C/min to reach the final isothermal temperature. During the 100 °C/min temperature ramp, all the samples quickly lose 3-4wt% which is likely due to the water loss. The rate of weight loss dramatically increases above 250 °C and the values are given in Table 1. The perusal of Table 1 indicates that conducting polymer doped with DBSA can be used for blending with conventional thermoplastics at a temperature less than 250 °C.

The isothermal resistivity change of polyaniline doped with DBSA at different temperatures in the temperature range of 150–250 °C was recorded and the values are given in Table 2. The perusal of Table 2 indicates that the polyaniline doped with DBSA can be best blended with thermoplastics whose melting point lies between 150 and 220 °C. This was the reason of blending polyaniline with PS and PMMA.

#### Table 2

Isothermal resistivity of polyaniline doped with DBS vs time at different temperatures

Polymer	Temperature (°C)	Time (min)	Resistivity ( $\Omega$ cm)		
Doped polyaniline	Room temperature <sup>a</sup>	_	9,5		
1 1 5	$150 - 152^{b^{1}}$	5	10.4		
	150-152 <sup>c</sup>	15	11.6		
	150-152 <sup>d</sup>	30	15.6		
Doped polyaniline	Room temperature	_	9.5		
	200-202	5	12.6		
	200-202	15	17.8		
	200-202	30	28.6		
Doped polyaniline	Room temperature	_	9.5		
	250-252	5	39.1		
	250-252	15	406		
	250-252	30	1.2 k		

<sup>a</sup> Initial reading of polyaniline doped with DBS at room temperature.

<sup>b</sup> Polyaniline heated to 150 °C for 5 min and then brought back to room temperature and then resistance is measured.

<sup>c</sup> Polyaniline heated to 150 °C for 15 min and then brought back to room temperature and then resistance is measured.

<sup>d</sup> Polyaniline heated to 150 °C for 30 min and then brought back to room temperature and then resistance is measured.

The resistivity of the conducting composites of polyaniline blended with PS indicates that whereas a 10% loading of the polyaniline in PS shows a resistivity of 389 M $\Omega$ , 20% loading of the polymer shows a resistivity of 23.9 M $\Omega$ . 30 and 40% loading of polyaniline in PS matrix shows a resistivity of 672 and 89 k $\Omega$ . 50% loading of the polymer polyaniline shows a resistivity of 467  $\Omega$ . This shows that higher loading of the polymer when blended with PS gives better conductivity which is the reason of getting better EMI response at 101 GHz for 1:1 :: PANI:PS composite.

#### 3.1. FTIR analysis

The principal absorption bands observed in the FTIR spectra of polyaniline doped with PTSA is shown in Fig. 3. In the range  $1650-1400 \text{ cm}^{-1}$ , bands due to aromatic ring breathing mode, N-H deformation and C-N stretching are observed. Bands at 1581 and 1490 cm<sup>-1</sup> are the characteristic bands of nitrogen quinoid and benzenoid ring and are present in the doped polymer matrix. These bands show a blue shift on removal of the dopant from the polymer. The strong absorption band at 1141 cm<sup>-1</sup> in the doped polyaniline matrix is due to the charge delocalization in the polymer backbone [30]. The intensity of this band decreases rapidly once the polymer gets undoped. The band at  $819 \text{ cm}^{-1}$ confirms that the rings are joined in the polymer chain through para linkage. On recording the FTIR spectra of the polymer heated at 200 and 250 °C, sharp shifts in the characteristic bands of polyaniline has been observed (Fig.3, curves b and c). The peak at  $1581 \text{ cm}^{-1}$  gets shifted to 1622 and 1630  $\text{cm}^{-1}$  when the polymer sample is heated at 200 and 250 °C, respectively. This implies that at higher temperatures the polymer gets converted from benzenoid form to quinoid form. Similarly the peak at 1490 cm<sup>-</sup> observed for polyaniline at room temperature shifts to 1503 and  $1518 \text{ cm}^{-1}$  when the polyaniline is heated at 200 and 250 °C. The main characteristic strongest peak of polyaniline observed at 1141 cm<sup>-1</sup> at room temperature gets shifted to  $1161 \text{ cm}^{-1}$  with a strong intensity when the polymer is heated at 200 °C, but shows a feeble signal at 1138 cm<sup>-1</sup> when the polymer is heated at 250 °C. This shows that at 250 °C, the dopant moiety attached to polymer backbone completely comes out leading to weak signal of the characteristic band of the polymer. This aspect has also been confirmed by TGA analysis where it has been observed that the polymer is stable only up to 230 °C and beyond this temperature, the polymer loses the dopant attached to polyaniline.

#### 3.2. UV-visible spectra

UV-visible absorption spectra of polyaniline doped with PTSA, DBSA and emeraldine base is shown in Fig. 4. Undoped polyaniline in *N*-methyl pyrrolidinone (NMP) show absorption bands at 320 and 620 nm which are present due to the  $\pi - \pi^*$  transition of the benzenoid ring and exciton



Fig. 3. UV-visible absorption spectra of (a) emeraldine base in NMP (b) polyaniline doped with PTSA and (c) polyaniline doped with DBSA.

band. Polyaniline doped with PTSA in dimethyl sulphoxide (DMSO) shows absorption bands at 332, 439 and 815 nm whereas polyaniline doped with DBSA in chloroform shows bands at 351, 432 and 734 nm. The shift in the bands from 620 to 815 nm and appearance of a transition at 439 nm indicates the presence of metallic polaron band transitions in the polymer matrix. However, in case of polyaniline doped with DBSA, this band appears at 734 nm. This implies that dopant attached to the polymer matrix plays a major role and has a considerable effect on the conjugation length as well as solubilization of the polymer in the organic solvents. Polyaniline doped with PTSA has a longer conjugation length but has solubility in organic solvents like DMSO, NMP, etc. whereas polyaniline doped with

DBSA has lesser conjugation length but the polymer is soluble in organic solvents like chloroform besides DMSO and NMP. Moreover it has been observed that polyaniline doped with DBSA can be easily melt blended with thermoplastics because of the surfactant nature of the dopant which brings about processability in the polymer chain.

The mechanical properties of the conducting composites are also given in Table 3. The values in Table 3 indicates that whereas the blank PS exhibits a tensile strength of 48.73 MPa, the loading of conducting polymer polyaniline has a drastic effect on the mechanical properties of conducting composites. 10% loading of conducting polymer in PS matrix shows a tensile strength of the order of



Fig. 4. FTIR spectra of polyaniline doped with PTSA (a) at room temperature (b) at 200  $^{\circ}$ C and (c) at 250  $^{\circ}$ C.

 Table 3

 Mechanical and electrical properties of PS/PANI composite

S. no.	Composition of composite PS/PANI	Tensile strength (MPa)	Electrical resistivity (Ω cm)		
1	Blank PS	48.73	$> 10^{14}$		
2	90:10	36.43	$389 \times 10^{6}$		
3	80:20	28.73	$23.9 \times 10^{6}$		
4	70:30	23.41	$672 \times 10^{3}$		
5	60:40	20.68	$89 \times 10^{3}$		
6	50:50	18.39	467		

36.43 MPa whereas 20% loading of conducting polymer in PS matrix shows tensile strength of the order of 28.73 MPa. On loading 50% of the conducting polymer in PS matrix, tensile strength of the order of 18.39 MPa is observed. Though 1:1 conducting composite of PS and polyaniline shows a EMI shielding value of the order of 58 dB, however, the mechanical properties of the conducting composite decreases significantly. Hence alternatively, a suitable filler material has to be incorporated in the conducting composite so that there is a balance of electrical properties and mechanical properties in the conducting composite. We are working presently in the present direction.

#### 3.3. Static decay time measurements

The static decay time of the conducting composites was determined by static decay meter 406D by noting the time taken for the dissipation of static charge on going down from 5000 to 500 V. The static decay time measurements of the conducting composites of polyaniline blended with PS and PMMA are tabulated in Table 4. The results in Table 4 show that whereas blank PS did not dissipate the charge, a 2% loading of the conducting polymer polyaniline in the insulating matrix shows a static decay time of the order of 0.02 s. A 5% loading of PANI in the PS matrix shows a static decay time of the order of 0.01 s and a higher loading of the conducting polymer even shows a static decay time of the order of 0.01 s. Similarly a 2% loading of the conducting polymer polyaniline in PMMA matrix shows a static decay time of the order of 0.09 s when a positive charge is built up in the composite matrix whereas a static decay time of

 Table 4

 Static decay time of conducting polyaniline composites

0.11 s is observed when negative charge is built up in the composite material. A 5 and 10% loading of the conducting polymer in PMMA matrix shows a static decay time of the order of 0.01 s. Anything less than 2 s can be used as an antistatic material. The results indicate that conducting composites of PANI with PS and PMMA can be used effectively for the dissipation of static charge.

#### 3.4. Static charge measurements

Static charge measurements of the insulating polymer PS and PMMA and conducting composites was carried out using Para Electronics Static Charge Meter (SCM 1) which was mounted on a energy stand at a distance of 2.5 cm from the energy dish having capacitance of 25 F. It was found that on keeping the insulating PS sample in SCM 1 shows a deflection in the meter with the reading at 0.6 kV. Keeping the scale at Static Charge Energy monogram at 25 pF capacitance.

Reading on Charge Scale = 160Reading on Energy Scale = 450

Using the Multiplier table

Static potential = 0.6 kVCapacitance = 25 pF

On applying the formula Q = CV,

For PS,  $Q = 1.5 \times 10^{-8}$ C And Static Energy,  $E = 0.5CV^2 = 4.5 \times 10^{-5}$  J.

However, on keeping the conducting composite on the energy stand, no deflection was observed in SCM 1. The study indicates that the conducting composite sample with even 2% loading can be used for the antistatic applications.

#### 3.5. Shielding effectiveness (SE) at 101 GHz

EMI shielding effectiveness is defined as the attenuation of an electromagnetic wave produced by its passage through a shield. It is measured as the ratio of field strength that is incident on the barrier to the magnitude of the field strength

	1	2	3	4	5	6	7	8
Polystyrene	90	95	98	100	_	_	_	_
Polymethylmethacrylate	-	-	-	_	90	95	98	100
Conducting polyaniline	10	5	2	0	10	5	2	0
Static decay time <sup>a</sup> Sec (+)	0.01	0.01	0.02	DNC	0.01	0.01	0.09	DNC
Static decay time <sup>a</sup> Sec $(-)$	0.01	0.01	0.02	DNC	0.01	0.01	0.11	DNC

DNC: did not charge. This means that the machine cannot even get the charge across the plates.

 $^{\rm a}$  Time is measured for the charge to be reduced from 5000 to 500 V.



Fig. 5. Shielding effectiveness of conducting composites at different loading levels of polyaniline in PS matrix.

that is transmitted through the barrier and is expressed in decibels (dB) and is calculated according to

$$SE = 10 \log \frac{P_t}{P_i}$$

where  $P_{\rm t}$  is the power of the transmitted wave and  $P_{\rm i}$  is the power of the incident wave. The shielding effectiveness of the conducting composites by blending polystyrene with polyaniline doped with DBSA was measured at 101 GHz by a set up consisting of horned antenna by noting down the power output before and after placing the samples in front of the horned antenna. The values of the shielding effectiveness measured for the different loading levels of polyaniline in the PS matrix are shown in Fig. 5. The perusal of Fig. 5 indicates that 2 and 5% loading of the conducting polymer shows a shielding effectiveness of 4.23 and 8.24 dB whereas a 10% loading of the polyaniline in PS gives a shielding effectiveness of 11.32 dB. A 30% loading of the conducting polymer in host matrix gives a shielding effectiveness of 32.45 dB. 40 and 50% loading of the conducting polymer shows a shielding effectiveness of 42.79 and 58.59 dB. The study indicates that increasing the percentage of conducting polyaniline in the host polystyrene matrix, shielding effectiveness value increases. This implies that a conducting network domain is formed on increasing the polyaniline content in the PS matrix, which leads to a higher shielding effectiveness value for the blend. The above study indicates that low loading of the conducting polyaniline in the conducting composites can be used for electrostatic charge dissipation whereas higher loading of the polymer can be used for the shielding of electromagnetic interference at 101 GHz.

#### 4. Conclusions

Electrically conductive composites of polyaniline with PS and PMMA can be effectively melt blended in the temperature range from 180 to 220 °C. These conductive composites can be effectively used for the dissipation of static charge. A 2% loading of the conducting polymer in the PS matrix gives a static decay time of 0.02 s whereas in PMMA-polyaniline conductive composites a static decay time of the order of 0.9-0.11 s is observed. Conductive composites of polyaniline with PS shows a shielding effectiveness of the order of 58 dB against electromagnetic interference in the frequency range 101 GHz.

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