

# Conducting Fabric-reinforced Polyaniline Film Using *p*-Chlorophenol as Secondary Dopant for the Control of Electromagnetic Radiations

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(Received February 20, 2004)

(Accepted June 28, 2004)

**ABSTRACT:** Polyaniline (PAn) is a commercially viable conducting polymer exhibiting enough metallic conductivity to replace carbon/metal filled composites as a shield to control electromagnetic radiations. In this paper, we discuss the process to achieve a pore-free conducting fabric reinforced with PAn. The process involves the pretreatment of glass fabric before the *in situ* polymerization of aniline in the presence of either *p*-toluenesulfonic acid (PTSA) or camphor-10-sulfonic acid (CSA). The conducting E-glass fabric is coated with a conductive paste prepared from PAn-PTSA or PAn-CSA/*p*-chlorophenol (PCP) as the secondary dopant come solvent at 60°C to achieve a pore-free flexible conducting surface. The reinforced conducting fabrics are characterized by UV-Vis spectroscopy, conductivity, scanning electron microscopy (SEM), X-ray diffraction study (XRD), and thermogravimetric analysis (TGA). The electromagnetic interference shielding effectiveness (EMI SE) studies are carried out using the co-axial transmission line method in the frequency range of 0.1–1000 MHz. The pore-free conducting fabric reinforced with PAn-PTSA/PCP and PAn-CSA/PCP of 3 mm thickness offered an EMI SE of 58 and 55 dB at 1000 MHz. The study indicates that the SE increases with the increase in thickness and conductivity of the test coupon.

**KEY WORDS:** conducting glass fabric, secondary dopant, electromagnetic radiation, noncorrosive, environmental stability.

## INTRODUCTION

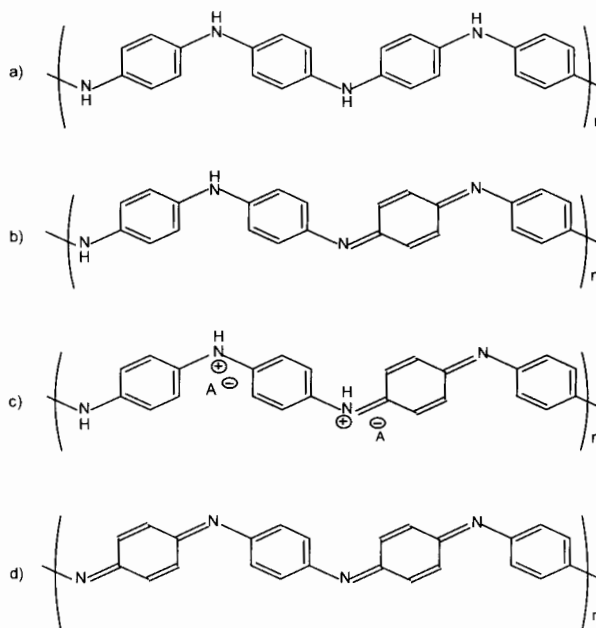
**T**HE INCREASING COMPLEXITY of electronic devices and systems in the form of higher packing density and quick response has generated pollution in the form of

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electromagnetic interference (EMI) [1]. Electromagnetic radiation is one of the unfortunate byproducts of the rapid proliferation of electronic devices. These twin problems if left unattended can cause severe damage to the communication system and the safety of operation of many devices and industrial units. The common material used for the construction of enclosures for shielding is mumetal, a high permeability alloy of 14% iron, 5% copper, 1.5% chromium, and 79.5% nickel. The other metals/materials used as a shield are brass, aluminum, silver, nickel, stainless steel, metalized plastics, and conductive carbon/graphite composites. At present the EMI is controlled by the use of metal and carbon fiber composites such as Al, Cu, Ag-epoxy matrix [2], Ni-carbon fiber/ABS [3], Ni-carbon fiber/PC/ABS [4], Ni-carbon fiber/PC/ABS /coupling agent [5], and electroless nickel carbon fiber/ABS [6,7]. These shielding materials based on metal/carbon suffer from limited mechanical flexibility, heavy weight, and corrosion.

Conducting polymers are new materials based on a carbon-carbon bond with a conjugation. They are thus flexible and free from corrosion with the advantage that their electronic conductivity can be tuned depending upon the need of its application and hence have been predicted to replace the existing metal/carbon/graphite-based material. If they replace them, the reduction in weight can be achieved and due to the noncorrosive nature can be advantageous under hostile environmental conditions. Among all known conducting polymers, polyaniline (PAn) is one of the important polymers because of its easy synthesis and environmental stability. The PAn is built up from reduced ( $-B-NH-B-NH-$ ) and oxidized ( $-B-N=Q=N-$ ) repeating units, where B denotes a benzenoid and Q denotes a quinoid ring. Thus the different ratios of amine to imine yield various structures, such as leucoemeraldine (a reduced form), emeraldine base (neutral undoped form), emeraldine salt (50% oxidized doped form), and pernigraniline (fully oxidized form). The various forms are shown in Figure 1. Conducting PAn has been



**Figure 1.** Various forms of PAn: (a) leucoemeraldine (nonconducting); (b) emeraldine base (nonconducting); (c) emeraldine salt (conducting); and (d) pernigraniline (nonconducting).

predicted to be a new material for EMI shielding [8–15]. However, no appropriate material based on PAn has so far been reported.

In this paper, we discuss the pretreatment of glass fabric prior to the grafting of PAn by the *in situ* polymerization of aniline under aqueous acidic conditions. The use of the secondary dopant, PCP to prepare a conductive paste, which can be coated on the PAn-grafted fabric to achieve pore-free reinforced PAn flexible surfaces to make it a viable material for the control of EMI is also described.

## EXPERIMENTAL PROCEDURE

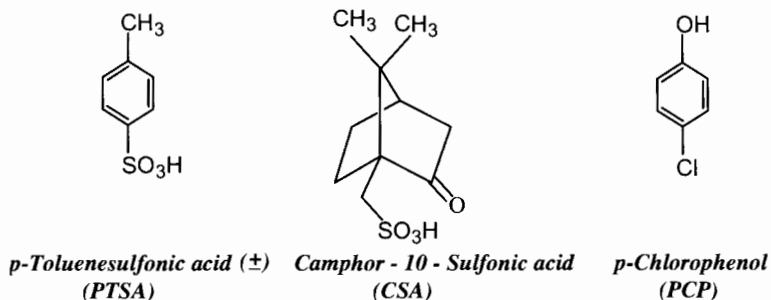
All the chemicals used are of AnalaR grade. Aniline is freshly distilled before use. Distilled water is used. Blue emeraldine base is obtained by a known synthetic procedure [16].

The E-glass fabrics are subjected to thorough cleaning using sodium carbonate solution to remove any foreign materials. These E-glass fabrics are then treated with 10% maleic anhydride solution in either ethyl methyl ketone, methanol, or acetone solution for 30–60 min and dried at room temperature. This pretreatment yields the fabric with a large number of active sites due to the carbonyl group, to act as an anchor for the PAn chain, whose formation is via the generation of free radicals. This way PAn gets anchored even in the interstices of the fabric to enhance the electronic conductivity of the grafted E-glass fabric and reduce its porosity.

The maleic anhydride-treated E-glass fabrics are immersed in a 0.1 M solution of aniline in 1 M aqueous solution of organic sulfonic acid (CSA/PTSA) at pH 1. The grafting is carried out using a double-walled tray ( $30 \times 40 \text{ cm}^2$ ) with a provision to circulate chilled water ( $\sim 2\text{--}4^\circ\text{C}$ ) to maintain the temperature. The substrates are rotated continuously on a mechanical shaker to ensure the uniformity of reaction at the solid–liquid interface. A stoichiometric amount of 0.1 M chilled aqueous solution of ammonium peroxydisulfate is added slowly to the reaction mixture containing the substrate. After an hour, the substrates are removed and rinsed thoroughly with distilled water. The grafting experiments are carried out twice to achieve good conductivity and deposition in the interstices of the fabric. At the end of the two graftings, dedoping is carried out in 1 M ammonia solution to remove all the oligomeric impurities. After thorough washing with distilled water, the dedoped fabrics are dipped in the doping acid solution of 1 M strength and agitated for 1 h to ensure uniform doping and are finally dried at  $50^\circ\text{C}$  under vacuum for 2 h.

A PAn–PTSA/PCP paste is prepared by mixing 2.5 g (7 mmol) emeraldine base powder, 2.6 g (14 mmol) PTSA, and 40 g (311 mmol) PCP. These are grounded thoroughly in a homogenizer for 1–2 h. PAn–PTSA-grafted conducting E-glass fabric reinforced with PAn–PTSA/PCP is prepared by applying this paste on the surfaces of the PAn–PTSA-grafted conducting E-glass fabric using an applicator. After applying this coating the coupons are cured for 12 h at room temperature and later dried under vacuum for 4 h at  $60^\circ\text{C}$ . The structure of the dopants PTSA, CSA, and PCP is shown in Figure 2.

Similarly, a PAn–CSA/PCP paste is prepared by mixing 2.5 g (7 mmol) emeraldine base powder, 3.2 g (14 mmol) CSA, and 60 g (467 mmol) PCP. These are grounded thoroughly in a homogenizer for 1–2 h. The PAn–CSA-grafted conducting E-glass fabric reinforced with PAn–CSA/PCP is prepared by applying this paste on the surfaces of the PAn–CSA-grafted conducting E-glass fabric using an applicator. After applying this



**Figure 2.** The structure of various dopants.

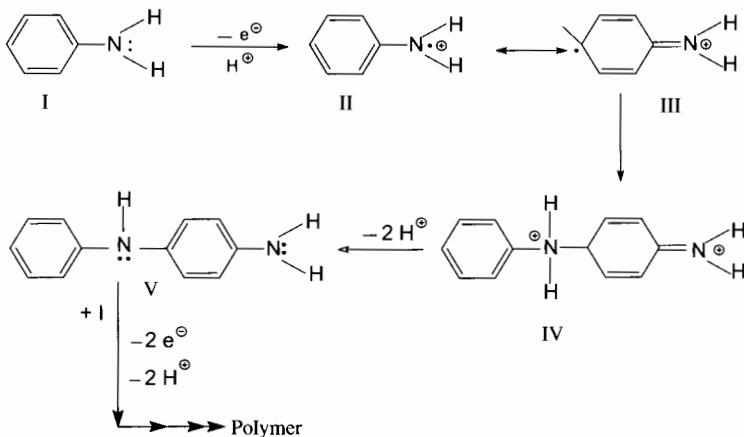
coating, the coupons are cured for 12 h at room temperature and later dried under vacuum for 4 h at 60°C.

The electronic spectra of the PAN-grafted conducting E-glass fabric reinforced with PAN/PCP is recorded on a UV-Vis-NIR Varian Cary-500 spectrophotometer. The conductivity data of the grafted samples are measured by four-probe method (Model No DFP-02). The scanning electron micrographs of these materials are recorded on a Hitachi S-3000H. The thermogravimetric analysis data are recorded on the Perkin-Elmer 7 series thermal analysis system from room temperature to 800°C at a rate of 10°C/min under an air atmosphere. The X-ray diffraction patterns are recorded using the (Cu  $K\alpha$ ,  $\lambda = 1.5418 \text{ \AA}$ ) Jeol JDX 8030 X-ray diffractometer.

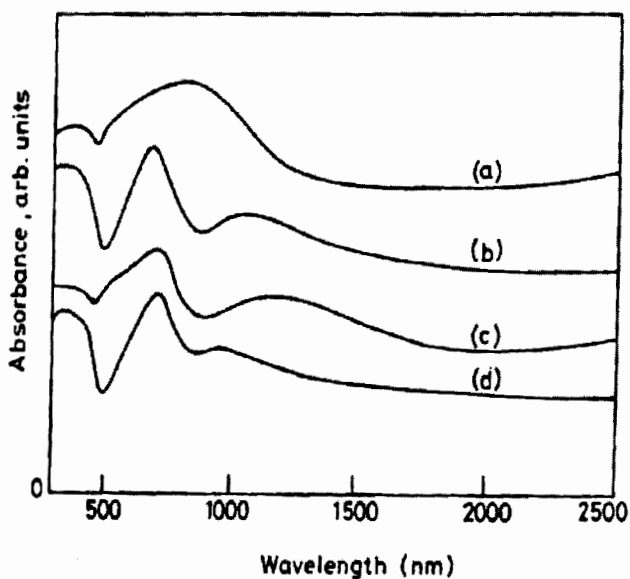
There are various methods available for the measurement of EMI SE. The coaxial transmission line method is the most preferred among them because results from various laboratories are comparable. The tests are carried out on a small torus-shaped cell. The round sample coupon of an inner diameter 43 mm and an outer diameter of 120 mm is cut and silver ink is applied on the periphery of the sample to reduce the contact resistance between the sample and the holder to 0.2  $\Omega$  cm. The measurements are made at specific frequencies using a signal generator 8642 B (Hewlett Packard) and field intensity meters (NM17/27A for 0.01–32 MHz and NM37/57A for 30–1000 MHz) with point-by-point method, with and without the specimen.

## DISCUSSION AND CONCLUSIONS

The formation of the conducting polymers occurs via the generation of a cation radical of intermediate stability, which on further coupling builds up a polymer chain. This intermediate stability of the cation radicals is utilized to carry out the *in situ* coating of the conducting polymers on the insulating surfaces like E-glass fabric using different dopants. These cations of intermediate stability get adsorbed on the surface interstices of fabrics, and these cations, on further coupling reaction, lead to the formation of stable, adherent and uniform electrically conductive coating. This conducting coating is named as grafting because it does not exhibit an electrochromic response as observed for the deposition on metal surfaces suggesting that the  $\pi$ -electrons of the aromatic nucleus are involved in the strong adsorption on the fabric and hence the transition of the structure from benzenoid to quinoid gets hampered. The mechanism of the PAN synthesis can be written as in Scheme 1.



**Scheme 1.** Mechanism of PAN synthesis.



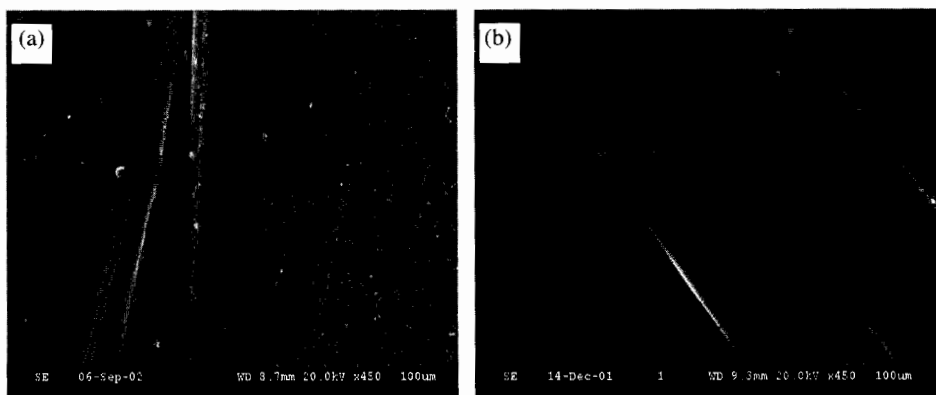
**Figure 3.** The UV-Vis-NIR absorption spectra of: (a) PAN-PTSA/PCP-reinforced conducting E-glass fabric; (b) PAN-PTSA-grafted conducting E-glass fabric; (c) PAN-CSA/PCP-reinforced conducting E-glass fabric and (d) PAN-CSA-grafted conducting E-glass fabric.

The electronic spectra of the PAN-PTSA/PCP-reinforced conducting E-glass fabric exhibits two absorption peaks at 381 and 836 nm. In the absence of a secondary dopant PAN-PTSA-grafted conducting E-glass fabric has absorption peaks at 328, 439, and 682 nm. Similarly, the electronic spectra of the PAN-CSA-grafted conducting E-glass fabric exhibits absorption peaks at 340, 416, and 698 nm and the PAN-CSA/PCP-reinforced conducting E-glass fabric exhibits absorption peaks at 353 and 828 nm and the spectrum is shown in Figure 3 and the absorption peak maxima are presented in Table 1.

The peaks observed at 328 and 340 nm for PAN-PTSA and PAN-CSA is assigned due to  $\pi-\pi^*$  transition associated with the benzenoid ring. The peak observed at 439 and 416 nm

**Table 1. Absorption peak maxima of PAN-grafted conducting E-glass fabric in the presence and absence of PCP.**

Sample	$\lambda_{\max}$ in nm	
	In the absence of PCP	In the presence of PCP
PAn-PTSA-grafted conducting E-glass fabric	328, 439, 682	381, 836
PAn-CSA-grafted conducting E-glass fabric	340, 416, 698	353, 828



**Figure 4.** Scanning electron micrograph of: (a) PAN-PTSA/PCP-reinforced conducting E-glass fabric and (b) PAN-CSA/PCP-reinforced conducting E-glass fabric.

for PAN-PTSA and PAN-CSA is due to cation radicals. The band observed at 682 and 698 nm for PAN-PTSA and PAN-CSA is due to charge carriers. The band due to the charge carrier observed at 682 nm for PAN-PTSA-grafted conducting E-glass fabric shifts to a higher wavelength  $\sim 836$  nm extending to the IR region in the presence of a secondary dopant, PCP. Similarly, the band due to the charge carriers observed at 698 nm for PAN-CSA-grafted conducting E-glass fabric shifts to a higher wavelength  $\sim 828$  nm extending to the IR region in the presence of a secondary dopant, PCP. This shows that the secondary dopant not only favors the formation of charge carriers (bipolarons), but also induces processibility in the polymer system by its plasticizing effect, which yields a closely packed polymer matrix by 'straightening out' the polymer chain from its coiled structure to the uncoiled one and becomes more expanded with a concomitant reduction in the  $\pi$ -defects caused by ring twisting as suggested by MacDiarmid and Epstein [17,18].

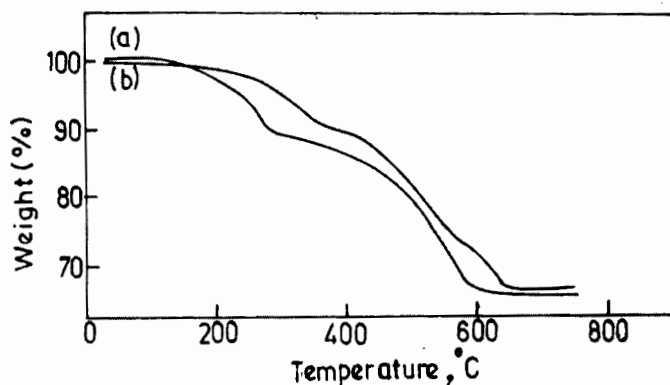
The conductivity of the PAN-grafted E-glass fabric reinforced with doped PAN/PCP is measured by the four-probe method and the data is presented in Tables 3 and 4. By this method, the conductivity of the PAN-PTSA/PCP-reinforced conducting E-glass fabric is found to be 0.6S/cm (thickness = 0.15 mm), 1S/cm (thickness = 1.4 mm) and 2S/cm (thickness = 3 mm) and that of the PAN-CSA/PCP-reinforced conducting E-glass fabric is found to be 1S/cm (thickness = 0.15 mm), 1.5S/cm (thickness = 1.4 mm) and 3S/cm (thickness = 3 mm). The thickness of the sample was increased by increasing the volume fraction of PAN.

The SEM study of the PAN-PTSA/PCP and PAN-CSA/PCP-reinforced conducting E-glass fabric indicates the fibril nature of a PAN, which is spaced at a regular interval without any morphological defect and is shown in Figure 4.

The TGA of PAN-PTSA/PCP-reinforced conducting E-glass fabric shows the first weight loss of ~2% at 100°C due to the loss of moisture. The second weight loss occurs in two steps. In the first step, a weight loss of ~9% is observed between 142 and 335°C, which is equivalent to the weight of the dopant i.e., PTSA. In the second step, a weight loss of ~16.4% is observed between 335 and 679°C, which is equivalent to the secondary dopant i.e., PCP. The final degradation of the polymer starts from 690°C and the substrate is found to be stable up to 800°C. Similarly, the TGA of PAN-CSA/PCP-reinforced conducting E-glass fabric shows the first weight loss of ~1% at 100°C is due to a loss of moisture. The second weight loss occurs in two steps. In the first step, a weight loss of ~7% is observed between 139 and 363°C, which is equivalent to the weight of the dopant i.e., CSA. In the second step a weight loss of ~13.6% is observed between 363 and 654°C, which is equivalent to the secondary dopant i.e., PCP. The final degradation of the polymer starts from ~695°C and is found to be stable up to 800°C and the curves are shown in Figure 5.

The PAN-PTSA-grafted E-glass fabric gives XRD peaks at  $2\theta = 5.7, 7.2, 8.7, 10.6, 12.1,$  and  $14.5^\circ$  and these peaks are broader indicating the amorphous nature of the cloth. The XRD patterns of PAN-PTSA/PCP-reinforced conducting E-glass fabric exhibit peaks at  $2\theta = 5.4, 6.6, 10.3,$  and  $13.2^\circ$ . Similarly, the PAN-CSA-grafted conducting E-glass fabric gives XRD peaks at  $2\theta = 5.2, 6.8, 8.6, 9.9, 12.1, 11.5, 13.5,$  and  $14.4^\circ$  and these peaks are broader indicating the amorphous nature of the cloth. The XRD patterns of PAN-CSA/PCP-reinforced conducting E-glass fabric exhibit peaks at  $2\theta = 6.3, 8.1, 9.6, 10.7,$  and  $13.2^\circ$ . As can be observed, in the presence of PCP as a secondary dopant, the XRD peaks are sharper indicating that an amorphous material has developed some crystallinity in its structure. The  $2\theta$  and  $d$  values of the PAN-grafted conducting E-glass fabrics and PAN/PCP-reinforced conducting E-glass fabrics are tabulated in Table 2 and the patterns are recorded in Figure 6.

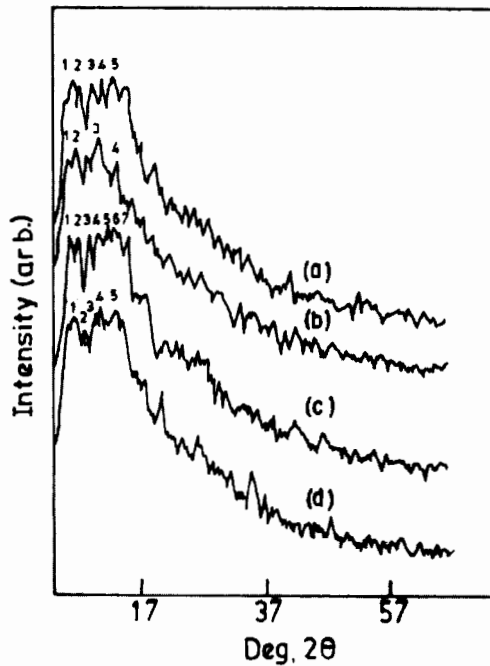
The shielding effectiveness (SE) for EMI control is largely a function of the conductivity of the material. Materials having a resistivity below  $10\ \Omega\text{cm}$  are suitable shields for electromagnetic interference (EMI) while higher resistivity materials are suitable for antistatic applications. Thus EMI SE is described as the attenuation of an electromagnetic wave produced by its passage through a shield and is measured as the ratio of the shield



**Figure 5.** The thermogravimetric analysis curves of: (a) PAN-PTSA/PCP-reinforced conducting E-glass fabric; (b) PAN-CSA/PCP-reinforced conducting E-glass fabric.

**Table 2. X-ray diffraction powder pattern ( $\text{Cu } K\alpha$ ,  $\lambda = 1.5418\text{\AA}$ ) of PAn-PCP-reinforced conducting E-glass fabric.**

PAn-PTSA-grafted conducting E-glass fabric		PAn-PTSA/PCP-reinforced conducting E-glass fabric		PAn-CSA-grafted conducting E-glass fabric		PAn-CSA/PCP-reinforced conducting E-glass fabric	
$2\theta$	$d$	$2\theta$	$d$	$2\theta$	$d$	$2\theta$	$d$
5.7	15.4	5.4	16.3	5.2	16.9	6.3	14.0
7.2	12.2	6.6	13.3	6.8	12.9	8.1	10.9
8.7	10.1	10.3	8.5	8.6	10.2	9.6	9.2
10.6	8.3	13.2	6.7	9.9	8.9	10.7	8.2
12.1	7.3	—	—	11.5	7.6	13.2	6.7
14.5	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—



**Figure 6.** The X-ray diffraction powder pattern ( $\text{Cu } K\alpha$ ,  $\lambda = 1.5418\text{\AA}$ ) of: (a) PAn-PTSA-grafted conducting E-glass fabric; (b) PAn-PTSA/PCP-reinforced conducting E-glass fabric; (c) PAn-CSA-grafted conducting E-glass fabric; and (d) PAn-CSA/PCP-reinforced conducting E-glass fabric.

strength before and after attenuation and is expressed in decibel (dB) calculated as per Equation (1) [1]

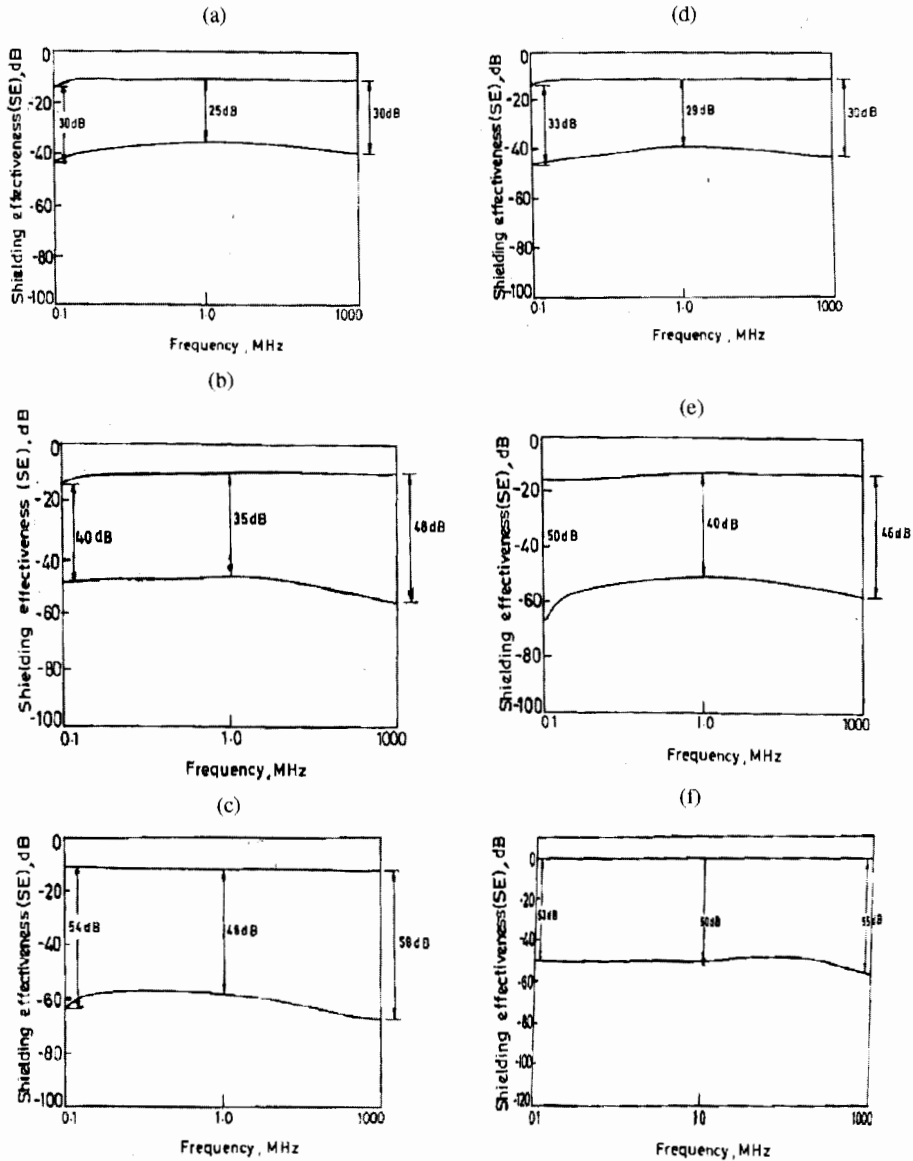
$$SE = 20 \log E_t/E_i \quad (1)$$

where  $SE$  is the shielding effectiveness,  $E$  is the electrical field strength in  $\text{Vm}^{-1}$ ,  $i$  is incident, and  $t$  is transmitted.



The EMI SE measurements are carried out using the coaxial transmission line method in the frequency range of 0.1–1000 MHz and the curves are shown in Figure 7 and the results are tabulated in Tables 3 and 4. The study also indicates that the SE of these samples has a strong dependence on:

- placement of the sample
- anisotropy of the sample



**Figure 7.** EMI SE curves of PAN/PCP-reinforced conducting E-glass fabric: (a) PAN-PTSA/PCP ( $t = 0.15$  mm); (b) PAN-PTSA/PCP ( $t = 1.4$  mm); (c) PAN-PTSA/PCP ( $t = 3$  mm); (d) PAN-CSA/PCP ( $t = 0.15$  mm); (e) PAN-CSA/PCP ( $t = 1.4$  mm); (f) PAN-CSA/PCP ( $t = 3$  mm).

**Table 3. EMI SE studies on PAN-PTSA/PCP-reinforced conducting E-glass fabric.**

Sample (Thickness, mm)	Conductivity (S/cm)	SE (dB) at				
		0.1 MHz	1 MHz	10 MHz	100 MHz	1000 MHz
PAN-PTSA/PCP-reinforced conducting E-glass fabric (0.15)	0.6	30	25	24	26	30
PAN-PTSA/PCP-reinforced conducting E-glass fabric (1.4)	1	40	35	38	42	48
PAN-PTSA/PCP-reinforced conducting E-glass fabric (3)	2	54	48	50	52	58

**Table 4. EMI SE studies on PAN-CSA/PCP-reinforced conducting E-glass fabric.**

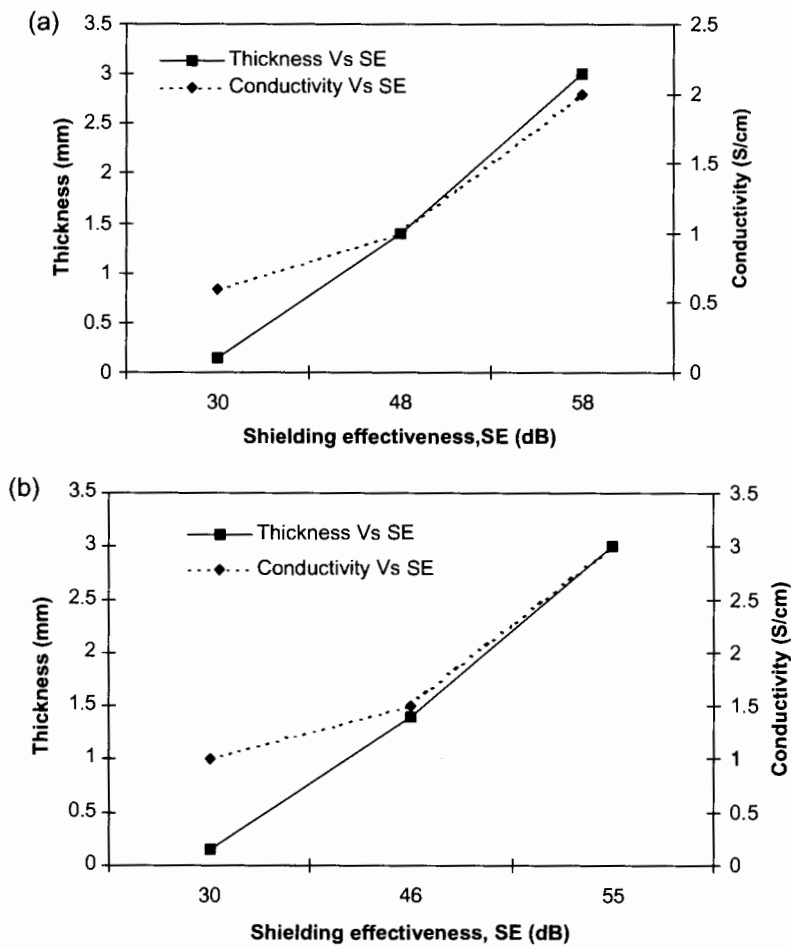
Sample (Thickness, mm)	Conductivity (S/cm)	SE (dB) at				
		0.1 MHz	1 MHz	10 MHz	100 MHz	1000 MHz
PAN-CSA/PCP-reinforced conducting E-glass fabric (0.15)	1	33	29	28	30	30
PAN-CSA/PCP-reinforced conducting E-glass fabric (1.4)	1.5	50	40	40	41	46
PAN-CSA/PCP-reinforced conducting E-glass fabric (3)	3	53	50	50	50	55

- thickness of the coating
- level of doping and type of dopant
- orientation of the dopant
- magnetic properties

Tables 3 and 4 show that increasing the sample thickness and conductivity enhances the shielding effectiveness of the material. This study indicates that a 3 mm thick sample of PAN-PTSA/PCP-reinforced conducting E-glass fabric of conductivity 2S/cm offers shielding effectiveness of 54 dB at 0.1 MHz and 58 dB at 1000 MHz and PAN-CSA/PCP-reinforced conducting E-glass fabric offers shielding effectiveness of 53 dB at 0.1 MHz and 55 dB at 1000 MHz. The reason for better shielding effectiveness of PAN-CSA/PCP is ascribed to the fibrillar structure, which is pore-free as can be seen in the scanning electron micrographs. This PAN-secondary dopant solution coated over the PAN-grafted fabric increases the thickness of the conducting PAN and its conductivity and gets deposited in the interstices of the grafted fabric to make it pore-free.

The effect of SE on the thickness and conductivity of the PAN-reinforced conducting E-glass fabric is shown in Figure 8(a) and (b) and the results show that increase in SE with the thickness and conductivity.

The shielding effectiveness of PAN-grafted conducting E-glass fabric can be enhanced by coating it with a conducting PAN paste using PCP as the secondary dopant and PTSA and CSA as the primary dopants. The advantage of using these PAN-grafted surfaces is that they withstand any level of acidic fumes and high humidity levels without any degradation.



**Figure 8.** The effect of SE on the thickness and conductivity of (a) PAN-PTSA/PCP and (b) PAN-CSA/PCP-reinforced conducting E-glass fabric.

### ACKNOWLEDGMENTS

The authors wish to thank Dr D. C. Pandey, Scientist EMI/EMC, Electronics and Radar Development Establishment (LRDE), Bangalore (India) for his help in carrying out the study on EMI Shielding Effectiveness. Two of us (S. G and K. K. S), wish to thank the Council of Scientific and Industrial Research (CSIR), New Delhi for the award of a Senior Research Fellowship.

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