

**SYNTHESIS, CHARACTERIZATION AND EVALUATION OF
CONDUCTING POLYMER BLENDS AND COMPOSITES FOR
MICROWAVE SHIELDING AND ANTISTATIC APPLICATION**

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MICROWAVE SHIELDING AND ANTISTATIC APPLICATION**

by

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Centre for Polymer Science and Engineering

Submitted

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CERTIFICATE

This is to certify that the thesis entitled “**Synthesis, Characterization and Evaluation of Conducting Polymer Blends and Composites for Microwave Shielding and Antistatic Application**” being submitted by **Mr. Parveen Saini** to the Indian Institute of Technology, Delhi, for the award of the degree of **Doctor of Philosophy** is a record of bonafide research carried out by him. **Mr. Parveen Saini** has worked under our supervision and has fulfilled the requirements for the submission of this thesis, which to our knowledge has reached the requisite standard. The results contained in this thesis are original and have not been submitted, in part or full, to any other University and Institute for the award of any other degree or diploma.

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ABSTRACT

The electromagnetic interference (EMI) which is an offshoot of rapidly growing field of electronics and instrumentation is becoming an alarming issue. It's a novel kind of pollution which interferes with the normal functioning of electronic components and may lead to complete breakdown of appliance's performance. This in-turn increased the demand for counter-measures to ensure undisturbed functioning of the appliances even in the presence of external electromagnetic noises. In this regard, intrinsically conducting polymers (ICPs) have emerged as promising material for shielding electromagnetic (EM) radiations primarily because of their relatively high conductivity (σ) and permittivity (ϵ) values and non-transparency to microwaves. These materials offer unique combination of properties, like tunable conductivity, low density, corrosion resistance, facile processing and good EM attributes. In addition, the selective incorporation of dielectric/magnetic fillers within conducting polymer matrix is expected to further boost the absorption loss and hence EMI shielding effectiveness (SE_T).

In the above regard, an attempt has been made in the present thesis to design and develop processable conducting polymers and their filled composites for microwave shielding and antistatic applications. In addition, conducting fabrics with controlled electrical and electromagnetic attributes were also prepared to display effective antistatic action and moderate shielding properties. Further, conducting polymer blends were also prepared by solution-blending of DBSA doped PANI-MWCNT with polystyrene (PS) matrix and melt-blending of PANI-DBSA (PIDB/PDB) with low density polyethylene (LDPE). These compositions were thoroughly characterized for structural (XRD, FTIR, UV-visible, XPS), thermal (TGA & DSC), electrical (EPR and four probe conductivity)

and morphological (SEM, TEM and HRTEM) attributes. The evaluation of static decay time and microwave shielding performance in the frequency range 8.2-12.4 GHz (X-band) and 12.4-18.0 GHz (Ku or P-band) was also done.

The thesis is divided into seven chapters. Chapter 1 throws light on the basic concepts of π -conjugated polymers with special address to polyaniline (PANI). This chapter also reviews the various routes for the synthesis of conducting polymers, concept of doping, conduction mechanism, different charge transport models and their applications especially in the field of microwave shielding. The basics of electromagnetic shielding theory, role of conducting polymers and different fillers for suppression of electromagnetic disturbances, their microwave shielding performance, different methods for the measurement of shielding effectiveness, dielectric and magnetic attributes, as well as theoretical correlations between shielding and electromagnetic attributes have also been covered in this chapter.

Chapter 2 describes methods used for the synthesis of conducting polymers, their composites, blends & fabrics and also illustrates different techniques and underlying principles for their detailed characterization.

Chapter-3 deals with the synthesis of polyaniline (PANI) doped with different protonic acids such as 4-dodecyl benzene sulfonic acid (DBSA), d-camphor-10-sulfonic acid (CSA), lignin sulfonic acid (LSA), cardanol azophenyl sulfonic acid (CDSA), acrylic acid (AA), poly(acrylic acid)[PAA] or polystyrene sulfonic acid (PSS), by in-situ emulsion polymerization or ex-situ doping processes, so as to evaluate the effect of various dopants on the electrical, spectral, morphological, structural, dielectric, and microwave shielding properties of doped PANI. It was observed that AA doped

emeraldine base (EB) samples prepared by ex-situ (indirect) route show total shielding effectiveness (SE_T) value of -18.5 dB. However, the in-situ (direct) doped PANI samples using polymeric dopants like PSS or PAA gives maximum SE_T values of -9.9 and -22.5 dB respectively. The emulsion polymerization of aniline in presence of “counter-anion surfactant dopants“ like CDSA, LSA, CSA and DBSA, results in further improvement of microwave shielding efficiency, with maximum SE_T values of -23.6, -38.3, -44.2 and -55.0 dB for PCD, PLS, PCS and PCD respectively. These results demonstrate superiority of DBSA over other dopants. Furthermore, DBSA doped PANI with good thermal stability ($>235^\circ\text{C}$) and compatibility with LDPE was melt-processed to form blends that shows antistatic characteristics and conforms to antistatic criteria i.e. static charge decay time of < 2.0 sec.

Chapter 4, describes the synthesis of highly conducting and microwave shielding composites by incorporation of carbon based conducting fillers [e.g. graphite powder (GP)/colloidal graphite (CG)/multi-wall carbon nanotubes (MWCNTs) etc.] within polyaniline (PANI) matrix. These composites were found to possess good processability, improved thermal stability and controlled electromagnetic attributes such as electrical conductivity and dielectric properties. The electrical conductivity data reflected that conducting fillers act as electrical link or bridge between metallic islands of the doped PANI, resulting in rapid enhancement of electrical conductivity. The high conductivity and presence of mobile charge carriers resulted in strong interaction with the electric field of the incident radiation, leading to high shielding effectiveness (SE_T). The microwave shielding effectiveness values revealed that among various fillers, the MWCNTs gives the same SE_T value (-39.6 dB) at the much lower loading levels than for GP (-39.7 dB) or

CG (-34.2 dB). However it was also observed that the reflection loss was maximum for MWCNT (-11.7 dB) loaded composites, than for GP (-8.3 dB) or CG (-8.6 dB) filled PANI, suggesting that PANI-MWCNT composite materials possess highest microwave conductivity value. The above results displayed the superiority of using high aspect ratio fillers like MWCNT than utilizing lower aspect ratio inclusions e.g. GP or CG.

In the chapter 5, emphasis has been given on the designing of organic-inorganic hybrid microwave absorbing materials with moderate conductivity and tunable dielectric/magnetic attributes so as to specifically enhance absorption loss. In this connection, different compositions were prepared by incorporation dielectric (cubic or tetragonal BaTiO₃) or magnetic (γ -Fe₂O₃ or Fe₃O₄) nano-particulate fillers within PANI matrix by in-situ emulsion polymerization of aniline using sulfonic acids like DBSA which also acts as dopant as well as surfactant. The microwave shielding measurements have shown that total shielding enhances by proper incorporation of electrically insulating/semiconducting dielectric (titanates) or magnetic (ferrites) fillers. The successive addition of cubic BaTiO₃, γ -Fe₂O₃ and MWCNT into PANI matrix leads to systematic improvement of total shielding effectiveness (SE_T) value from -23.6 dB to -57.7 dB. In contrast, the composites loaded with nanoparticles of tetragonal BaTiO₃ or Fe₃O₄ gives maximum attenuation (or SE_T value) of -71.5 and -82.5 dB respectively with improvement of absorption and concomitant reduction of reflection loss. The lowering of reflection loss was due to reduction of conductivity whereas enhanced absorption was a direct consequence of improved dielectric/magnetic losses. These results demonstrate superiority of Fe₃O₄ loaded composites among other materials. Nevertheless, the radiation

blocking ability of the order of -60 to -80 dB, justifies the utility of these materials for making futuristic microwave absorbers.

In the Chapter 6, an attempt has been made to prepare conducting fabrics based on polyaniline (PANI) or polypyrrole (PPY) by in-situ polymerization and dip coating process. This approach combines the mechanical properties and flexibility of fabrics with conductivity of doped π -conjugated polymers. Most importantly, the direct coating of conducting polymers on the surface of textiles via in-situ polymerization route eliminated the need of otherwise necessary thermal or solution processability. Furthermore, the in-situ coating of PANI over cotton fabric in the presence of dielectric (BaTiO_3) or magnetic (Fe_3O_4) nanoparticles resulted in conducting fabrics with additional dielectric/magnetic properties. In addition conducting polymer based blends were also prepared by solution blending of previously prepared PANI-MWCNT nanofiller with polystyrene (PS) matrix.

The above conducting fabrics and blends have been evaluated for their antistatic response and microwave shielding capability. These conducting fabrics exhibit ultrafast charge rates with static decay time of the order of ~ 0.2 to 0.01 sec, which clearly qualifies the antistatic criteria of 10% cut-off time of less than 2.0 sec. The good antistatic response of these textiles arises due to coated fiber that forms conducting domains having high aspect ratio accruing from the long lengths of individual fibers. In contrast, the LDPE and PS conducting blends display low percolation thresholds (0.5 to 1.5 wt %) and static dissipation time of order of 0.01 to 1.5 sec.

The microwave measurements have shown that conducting fabrics possesses finite shielding effectiveness values which can be further enhanced by forming multilayered structures and/or by incorporation of dielectric (titanate)/magnetic (ferrite) nanoparticles

within coated matrix. Therefore, the SE_T value of single layer of PSC and PAC were -1.5 and -1.7 dB respectively, which enhances to -5.2 and -5.7 dB for a four layered shield. The addition of $BaTiO_3$ and Fe_3O_4 nanoparticles resulted in further enhancement of SE_T to -5.6 dB (PSCBF) and -7.2 dB (PACBF). Similarly, the in-situ coated fabrics based on PPY- $FeCl_3$ (PPY-F) and PANI-HCl (PANI-F) exhibit SE_T value of -11.7 dB and -9.6 dB respectively. Further a multilayered shield consisting of 5 layers of PPY-F even shows SE_T value of -45.7 dB, which conforms to specified range of -30 to -40 dB for various techno-commercial applications. When, PANI-DBSA was used to makes coated fabrics (CPDB), the SE_T value of -15.2 dB was observed. The incorporation of $BaTiO_3$ and Fe_3O_4 particles resulted in enhancement of SE_T to -16.2 dB (CPBT11) and -19.4 dB (CPFF12). Furthermore, it has been observed that addition of dielectric or magnetic particles resulted in dominance of absorption (SE_A) compared to reflection loss (SE_R).

The solution blending of PANI-MWCNT filler with PS host resulted in attainment of absorption dominated ($SE_A \gg SE_R$) SE_T value of -45.7 dB for a 2.0 mm thick shield. Nevertheless, the radiation blocking ability of the order of -30 to -40 dB, justifies the utility of these materials for making commercial microwave shields.

The summary of thesis and main conclusions are given in the chapter 7. Suggestions for future work are also made.

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