# Hybrid Polyaniline/Nanomagnetic Particles Composites: High Performance Materials for EMI Shielding

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**ABSTRACT:** In this work, two types of hybrid composite materials were elaborated. The first based on polyaniline (PANI) doped Camphor Sulfonic acid (CSA), Carbon-Coated Cobalt (CCo), and FeNi nanoparticles dispersed in polyurethane. A value of $10^4$ S/m of conductivity and a 90 dB of shielding effectiveness in multilayer structure were obtained over the 8–18 GHz frequency band. The second type, based on PANI doped para-toluene sulfonic acid (PTSA), dispersed in epoxy resin with FeNi nanoparticles. A thick material with moderate conductivity and high attenuation of electromagnetic waves was obtained. It was found that a PANI-PTSA/FeNi/epoxy resin composite with thicknesses of 9.7 and 6.5 mm had, respectively, reflection loss values of −22 dB at 9.52 GHz, and −20.7 dB at 14.7 GHz. The electromagnetic properties of the elaborated structure hybrid materials can be optimized to increase the electromagnetic reflection–absorption properties. Thus, the obtained structure can be used in shielding and radar absorbing materials applications. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

## INTRODUCTION

Over the past few decades, as the use of electronic devices working in the microwave frequency band has rapidly grown, the electromagnetic interferences (EMI) have drastically increased. Thus, large perturbations were produced in electronic systems of civil and military telecommunication. In the civil domain, the electromagnetic interferences can have dramatic consequences by disrupting and reducing the life time and the efficiency of electronic systems fitted to planes or automobiles. To avoid these problems, it is necessary to protect electronic equipments against the aggression of electromagnetic waves. One way is to confine the electronic circuits in a shielding package that permits to reduce the impact of the electromagnetic coupling with incident waves.

Extensive studies have been carried out to develop new and high efficient shielding materials that can reduce the impact of the EMI and increase the lifetime of electronic devices.[1–3] Various materials have been used such as conductive metal powders, ferrites, carbon products, chiral materials, synthetic organic fibers, and conjugated polymers.[4–9] Typically, metallic materials have been widely used for EMI shielding, since they can effectively reflect the incident electromagnetic wave.[10,11] However,

they have some drawbacks including a susceptibility to corrosion and a complex and uneconomic processing. In addition, their high density is incompatible with aeronautic applications and ground transportations where the weight gains are sought. In addition, metals almost reflect totally the incident radiations and cannot be used in applications where the generated waves in turn generate disturbances or are used for the detection of aircrafts.[12,13]

The interaction between an electromagnetic wave and a material can be modeled by three phenomena: reflection, absorption, and multiple reflections inside the material. These phenomena depend on the physical properties of the material, like the conductivity, the dielectric permittivity, the magnetic permeability, and the thickness. Extensive studies have been carried out during the last decade with the aim to control and modulate these physical parameters in order to control the reflection or the absorption of the electromagnetic wave.[4,14–16]

In this context, intrinsically conductive polymers (ICPs) have inspired much interest due to their lightweight, good processability, and the possibility to modulate easily their conductivity.[17] In particular, Polyaniline (PANI) has a special status among other conducting polymers due to various advantages: it

presents a good thermal and chemical stability, low cost and its conductivity can be reversibly controlled either electrochemically (oxidation/reduction process) or chemically (protonation/deprotonation process). However, PANI presents poor mechanical properties, so it can be blended with thermoplastic or thermosetting matrix to improve its elasticity.[18].Then the resulting material combines the electronic properties of PANI and the mechanical properties of the matrix and a very low percolation threshold can be obtained. Due to theses unique properties, PANI-based composites had been used to make new materials with wide field of applications as electromagnetic shielding,[19–21] gas sensors,[22] rechargeable organic batteries,[23,24] corrosion protection, and photovoltaic cells.[25–27]

Our previous work[28] demonstrated that a multilayered material is more efficient than a mono layer for electromagnetic shielding and that an optimized multilayered PANI composite can show a shielding effectiveness superior to 40 dB or 80 dB over the 50 MHz–18 GHz frequency band, depending on the application. These composites materials were highly conductive, and then the high SE is due to the reflection phenomena.

In this work, to improve the shielding effectiveness (SE) and reflection loss ($R_L$), composites with multilayered structures were elaborated. Magnetic nanoparticles were added to PANI composites to increase their electromagnetic performances and to study their effect on the reflection and the absorption of electromagnetic waves. Thus, two types of PANI-based composites were prepared. The first base on PANI with Polyurethane (Pu), where carbon coated cobalt (CCo) and FeNi nanoparticles were dispersed. The second is based on PANI and epoxy resin matrix, in which, FeNi nanoparticles were dispersed. The morphology of the samples was investigated by Scanning electron microscopy (SEM). The influence of the fillers content on the electromagnetic properties of composites has been investigated. Microwave shielding and microwave absorption of the composites at different thicknesses were investigated in X and Ku band using a Vector Network Analyzer (VNA). The results show that with the first process of transformation of PANI, very high SE are obtained with thin films, principally by the reflection of the electromagnetic waves, while the second process provides thicker materials with magnetic properties and moderate conductivities, so they are able to absorb electromagnetic waves in the microwave band.

### EXPERIMENTAL

#### Materials

Polyurethane (Elastollan 117) was given by BASF, Commercial medapoxy (DGEBA/DGEBF) was obtained from the Algerian Granitex company. Polyaniline Emeraldine Base (PANI-EB) with a molecular weight of 65000, Camphorsulfonic acid (CSA), Dichloroacetic acid (DCAA), Carbon-coated cobalt (CCo) with a particle size of about 50 nm and a conductivity of about $1,6.10^7$ S m$^{-1}$, (FeNi) Alloy ( iron: 55% and nickel: 45%) with a particle size of 100 nm, Tetrahydrofuran (THF), BF$_3$ – complex, Hexahydrophtalic anhydride (HHPA) and Polyaniline doped para-toluene sulfonic acid (PANI-PTSA) with a conductivity value of 600 S m$^{-1}$, were purchased from Sigma-Aldrich.

**Table I.** Composition, Conductivities, and Thicknesses of PANI/Polyurethane/CCo Composites

| Composition (%) | Composite multilayer 1 | | Composite multilayer 2 | |
| --- | --- | --- | --- | --- |
| | Layer 1 | Layer 2 | Layer 1 | Layer 2 |
| PANI-CSA | 20 | 20 | 20 | 3 |
| Pu | 60 | 70 | 75 | 97 |
| CCo | 20 | 10 | 05 | 00 |
| σ (S/m) | 1400 | 1200 | 870 | 5 |
| Thick (μm) | 470 | 520 | 640 | 210 |

#### Composites preparation

**PANI-CSA/Polyurethane/CCo and FeNi Composites: Codissolution Method.** PANI-EB was doped with CSA and dissolved in DCAA which was used as solvent and secondary doping agent. The PANI-EB was slowly added to CSA-DCCA solution during 10 min under regular stirring to avoid the formation of aggregates. The resulting solution was then stirred at 65°C during 3 days. The mass of each component is calculated to obtain a fully protonated PANI. During the protonation, the solution color changes from blue to green, and the conductivity increases considerably. In these conditions, the maximum obtained conductivity is about $10^4$ S m$^{-1}$.[29] Pu was separately dissolved in DCAA for 03 h using a magnetic stirrer. Then it was added to the PANI-CSA solution and was stirred for 24 h to obtain a homogeneous mixture. CCo and FeNi, particles were independently dispersed in DCAA using an ultrasonic bath at room temperature for 4 h, then mixed with the PANI/Pu solution and stirred for 24 h at room temperature. The resulting solution was heated at 80°C under vacuum until the complete evaporation of the solvent. Finally, free standing films were obtained with a thickness varying from 100 to 650 μm. The codissolution method provided thin materials with high conductivity. Then, multilayered structures were fabricated using different compositions for each layer as it is resumed in Tables I and II.

In the Table I, composites were prepared with PANI-CSA/CCo/Pu. Multilayer 1 have a 0.99 mm of total thickness and the same amount of PANI-CSA in both Layers 1 and 2; however, in the composite multilayer 2, the PANI-CSA amount was 20 and 3% in each layer.

In the Table II, all composites of PANI-CSA/FeNi/Pu were made with the same amount of 3% of PANI-CSA in all layer composites. Composite multilayer 1 and composites multilayer 2 have total thicknesses of 0.97 and 0.93 mm and conductivities of $10^{-3}$ S m$^{-1}$ and $10^{-2}$ S m$^{-1}$, respectively.

**PANI/FeNi/Epoxy Resin Composites: Dispersion Method.** Conducting PANI-doped PTSA and FeNi were dispersed separately using THF as solvent at room temperature using an ultrasonic. The epoxy resin matrix was added to the above mentioned colloidal solutions to prepare composites with different loading PANI and FeNi under continuous magnetic stirring and heating at 65°C to improve the dispersion's quality and to remove solvent residues. The weight contents of PANI and FeNi in composites

**Table II.** Composition, Conductivities, and Thicknesses of PANI/Polyurethane/FeNi Composites

| Composition (%) | Composite multilayer 1 | | | Composite multilayer 2 | | |
|---|---|---|---|---|---|---|
| | Layer 1 | Layer 2 | Layer 3 | Layer 1 | Layer 2 | Layer 3 |
| PANI-CSA | 3 | 3 | 3 | 3 | 3 | 3 |
| Pu | 87 | 82 | 77 | 92 | 67 | 57 |
| FeNi | 10 | 15 | 20 | 05 | 30 | 40 |
| σ (S/m) | $10^{-3}$ | $10^{-3}$ | $10^{-3}$ | $10^{-3}$ | $10^{-2}$ | $10^{-2}$ |
| Thick (µm) | 340 | 330 | 300 | 260 | 280 | 390 |

were: 1, 5, 10, 15, and 20 wt %. After the evaporation of the solvent, the curing system was added. The mixtures were poured immediately in appropriate moulds, to obtain a specimen for different analysis and were subjected to cross linking steps. This method provides thick materials with moderate conductivity and their composition characteristics are presented in Table III. In this table, the single layer composites have an conductivity of $10^{-2}$ S m$^{-1}$ and thicknesses of 1.5, 9.7, and 6.5 mm, respectively, however, composites made in Multilayers 1 and 2 have a conductivity of $10^{-3}$ S m$^{-1}$ and a total thickness of 3 mm.

**Characterization**

The morphological analysis were carried out using a scanning electron microscope type Hitachi S-4300 SE/N operating between 5 kV and 20 kV and 35 µA to 50 µA. To avoid the effects of electric charge on the surface and to facilitate the flow of electrons, samples were coated with a thin layer of gold. The static conductivity measurements were carried out on the films using the Van Der Pauw method.[30] Four probes were placed at the corners of the films (square shaped with thickness in order of 650 µm and a length of 13 mm. The method of measurement permits to verify if the samples are homogeneous and isotropic. A current (Keithley 220 current source) is sent through the sample and eight voltages are measured (DMM Keihtley 196) following the procedure described in the Keithley application note.[31] The conductivity is extracted using a standard algorithm.[32] The measurement range varies from $10^{-2}$ to $10^{6}$ S m$^{-1}$. Scattering parameters ($S_{11}$, $S_{21}$) of composites at different thicknesses and different amounts of fillers were carried out using a Vector Network Analyzer (VNA) type Agilent technologies, E8362B working from 10 MHz to 20 GHz. In this frequency domain, two sets of wave-guides were used: a WR-90

for the X band and a WR-62 for the Ku band. The wave-guides were linked to the VNA with two cables in APC7 standards. The sample was clamped between two flanges of the input and output wave guides through four holes. The calibration of the VNA was made according to the TRL (Through-Reflect-Line) method following the procedure given by the manufacturer. Microwave absorption properties were measured using the same instrument but the rear face of the sample was terminated by a short—circuit that is a perfect conductor. All measurements were repeated several times to ensure the repeatability of data.

**RESULTS AND DISCUSSION**

**Morphology**

Figure 1 shows the SEM morphology of composites. It was revealed that, PANI-doped CSA forms fibers structure as it is shown in micrograph (a). It is homogeneously distributed in the Pu matrix (images b). However, in micrographs (c), and (d), a homogeneous dispersion of crystalline particles is observed presenting the nanoparticles of CCo and FeNi dispersed in PANI/Pu matrix, respectively. Micrographs (e) and (f), show the FeNi particles and the epoxy resin composites. FeNi forms agglomerates due to interactions between particles. Consequently, it is poorly dispersed in the matrix. The white regions characterized the fillers particles. In addition, the PANI, CCo, and FeNi nanoparticles were consistently dispersed in the Pu matrix, indicating a homogeneous mixture of phases.[33,34]

**Shielding Effectiveness and Reflection Loss**

The electromagnetic shielding effectiveness (SE) of materials is defined as the ratio between transmitted powers ($P_t$) through the material to the incident power ($P_i$) of an electromagnetic wave.

$$SE = -20 \log |S_{21}| \tag{1}$$

**Table III.** Composition, Conductivities, and Thicknesses of PANI/FeNi/Epoxy Resin Composites

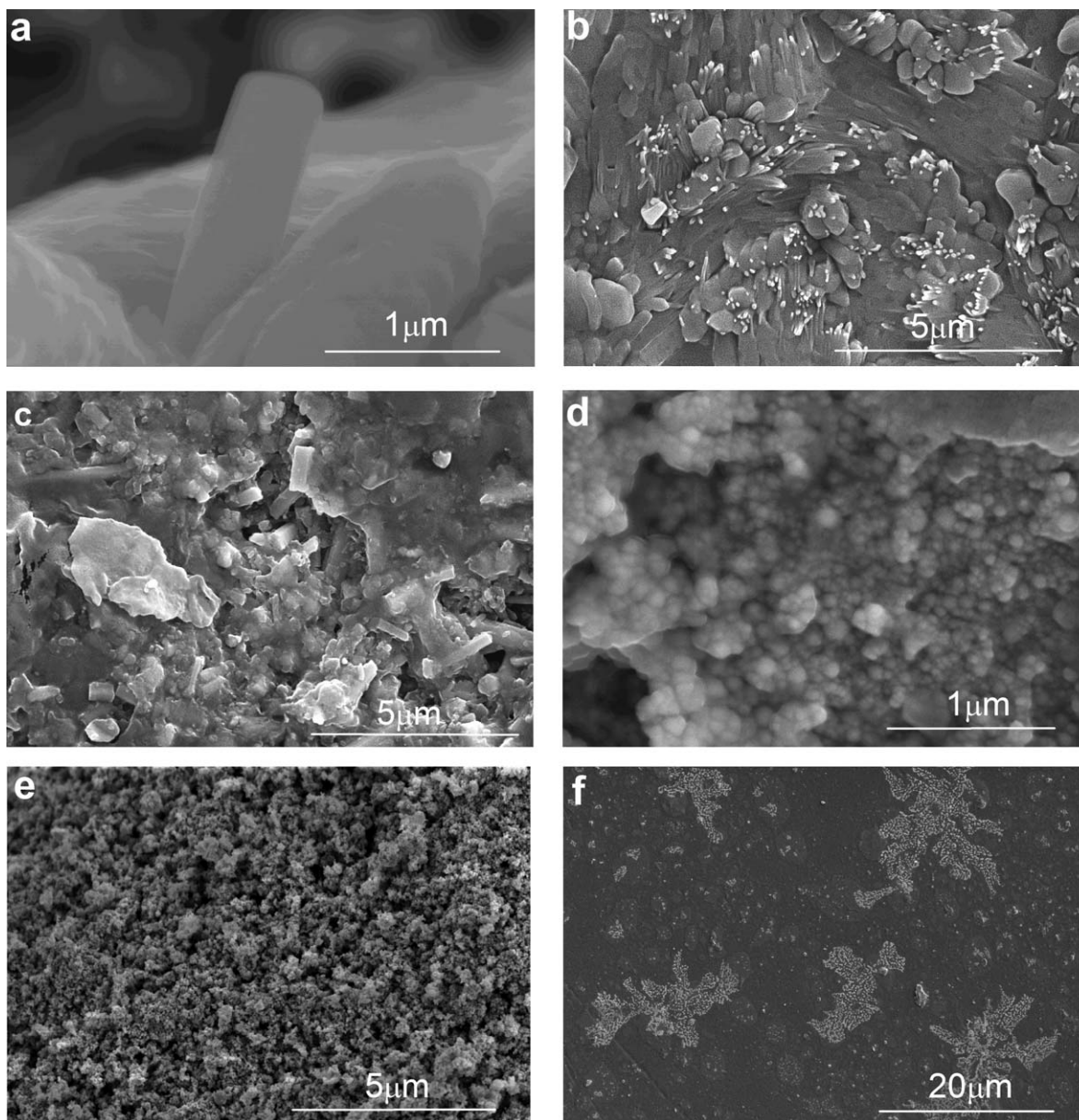| Composition (%) | Single layer composite | | Multilayer composite 1 | | Multilayer composite 2 | |
|---|---|---|---|---|---|---|
| | Composite 1 | Composite 2 | Layer 1 | Layer 2 | Layer 1 | Layer 2 |
| PANI-PTSA | 10 | 15 | 10 | 00 | 00 | 00 |
| ER | 75 | 80 | 90 | 85 | 90 | 85 |
| FeNi | 15 | 05 | 00 | 15 | 10 | 15 |
| σ. (S/m) | $10^{-2}$ | $10^{-2}$ | $10^{-3}$ | $10^{-3}$ | $10^{-3}$ | $10^{-3}$ |
| Thick. (mm) | 1.5 | 9.7 & 6.5 | 1.5 | 1.5 | 1.5 | 1.5 |

**Figure 1.** SEM micrograph composites of: PANI-CSA (a), PANI-CSA(3%)/Pu (b), PANI-CSA(3%)/FeNi(10%)/Pu (c), PANI-CSA(20%)/ CCo/Pu(10%) (d), FeNi powders (e), and FeNi (20%)/epoxy resin (f).

Experimentally, SE is directly given by the network analyzer by the parameter $S_{21}$.[6] In order to increase the electromagnetic properties; multilayered structures were studied using a theoretical model of shielding effectiveness based on transmitted wave matrix in the far field regime.[35] This model shows that the reflection and transmission coefficients depend on the intrinsic physical properties of each layer. The use of PANI composites makes possible to tune the electronic properties of the composites from insulating to conducting states and to control the thickness. Then, it is possible to optimize each layer of the structure to obtain values of the SE compatible with current standards and to control the reflection and the absorption coefficients.

The reflection loss was obtained from the measurement of the $S_{11}$ coefficient given by the network analyzer:

$$R_L = 20 \log |S_{11}| \qquad (2)$$

The variation of the reflection loss in function of frequency presents dips indicating the occurrences of absorption or minimal reflection of the electromagnetic wave. The intensity and frequency at $R_L$ minimum are depending on the electronic properties and thickness of samples.[36]

**PANI-CSA/Polyurethane/CCo and FeNi Composites.** Multilayered shielding structures were prepared, as shown in Tables I and II. The optimization of these structures was done in function of
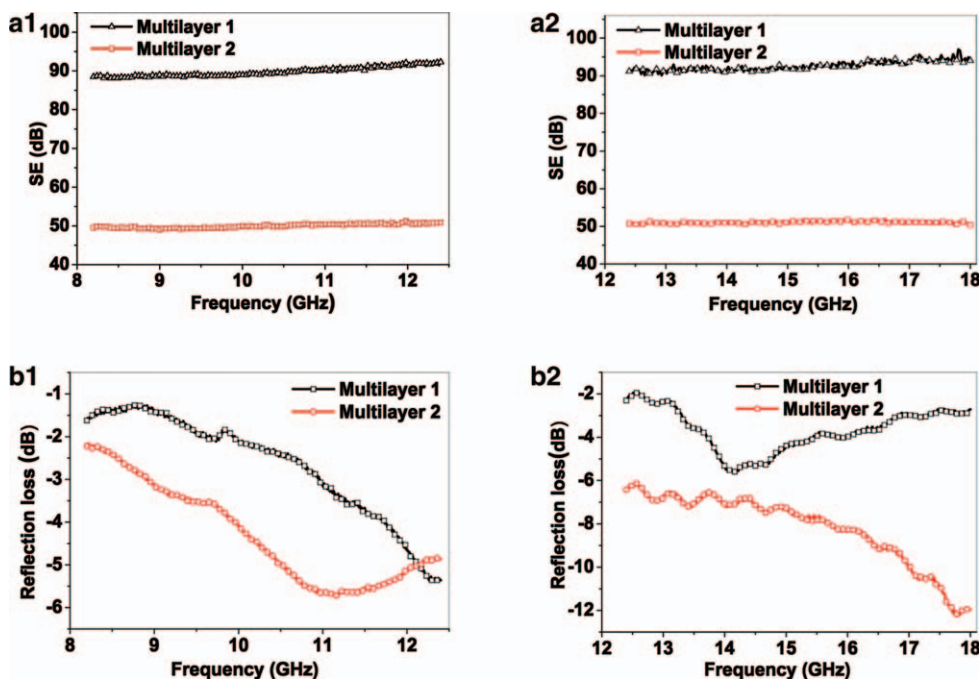
**Figure 2.** SE of PANI-CSA/Pu/CCo composite Multilayers 1 and 2 (a) and reflection loss of PANI-CSA/FeNi/Pu composite Multilayers 1 and 2 (b) in X and Ku bands. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

conductivity, dielectric permittivity and material thickness. These parameters are directly linked to the composition of the composite materials. Thus, the mass fraction of PANI, Pu, CCo, and the FeNi nanoparticles were precisely controlled in composites. The final goal of the optimization is to make light and thinner materials that meet the shielding standards (military

standard SE > 80 dB, civil standard SE > 40 dB) with a large reflection loss coefficient. Figure 2(a) shows the shielding effectiveness of the optimized multilayered materials. It is worth noticing that the multilayer materials 1 and 2 present a low thickness (<1 mm), and a shielding effectiveness superior to 80 and 40 dB in the X and Ku bands. Besides, the measured SE for
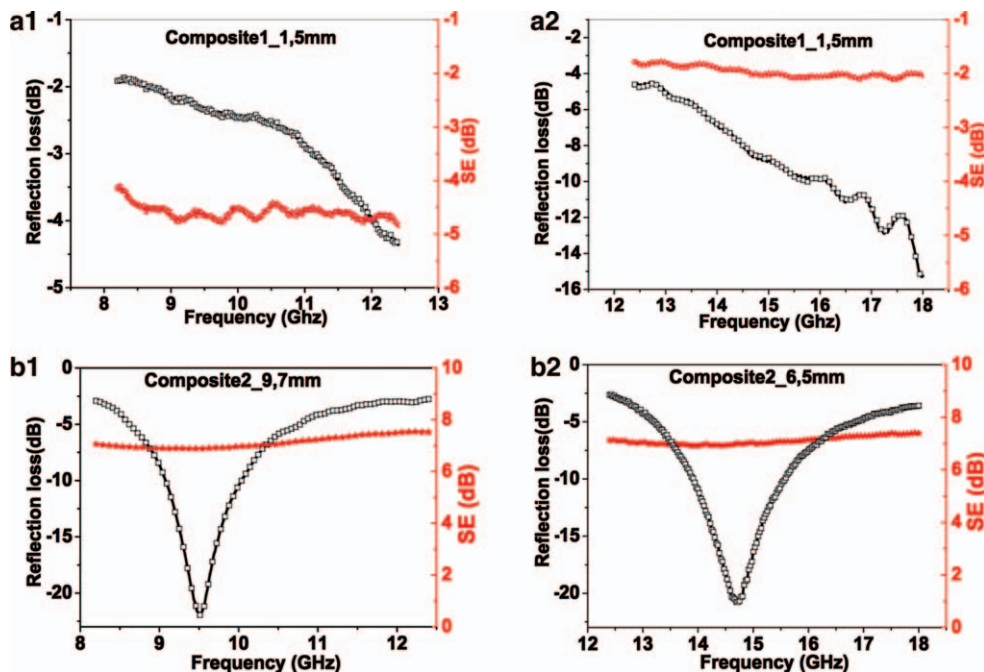


**Figure 3.** SE and reflection loss of PANI/FeNi/epoxy resin composites single layer at different thicknesses of: composites 1 (a) and composites 2 (b) in X and Ku bands. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
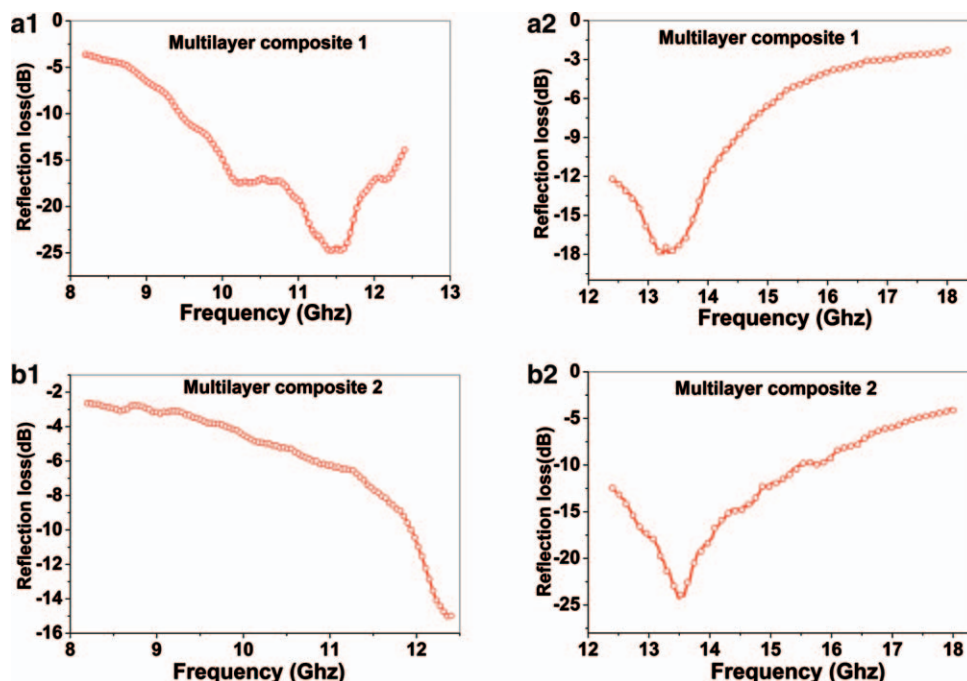
**Figure 4.** Reflection loss of PANI/FeNi/epoxy resin composite multilayered structures of: Composite 1 (a) and Composite 2 (b) in X and Ku bands. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the multilayer 1 (about 90 dB) corresponds to the limit of the system of measurement, thus the real SE of the Multilayer 1 can be estimated higher than 90 dB.[37] Figure 2(b) shows the reflection loss of two optimized multilayer structures of the PANI-CSA/FeNi/Pu composites Multilayers 1 and 2. We obtained reflection loss values of −6 dB and −12.2 dB at 14.2 GHz and 17.8 GHz in Ku band

**PANI/FeNi/Epoxy Resin/Composites.** The PANI/FeNi/ER composites frequency dependence of reflection loss and shielding efficiency in X and Ku bands at different thickness are shown in Figures 3 and 4. As expected, the reflection loss is dependent on the thickness and on the nature of the absorber. The structure based on PANI-PTSA(10%)/FeNi(15%)/epoxy resin composite, with a thickness of 1.5 mm, showed a $R_L$ of −15.3 dB and a SE of 4.5 dB at 18 GHz.

However, for the same thickness in multilayer structure of PANI-PTSA (10%)/FeNi(15%) composite, a value of −24 dB at 11.5 GHz and −17.7 dB at 13.4 GHz were observed. Moreover, a 1.5 mm of a composite 10% FeNi/15%FeNi multilayer indicate a value of −15 dB at 12.4 GHz and −26 dB at 13.5 GHz.[38] At higher thicknesses ($\lambda$/4 in X and Ku band) corresponding to 9.7 and 6.5 mm, respectively, of PANI-PTSA/FeNi/epoxy resin composite samples, reflection loss values of −22 dB at 9.52 GHz, and −20.7 dB at 14.7GHz were observed with a SE of around 7 dB. Microwave absorption is caused by the interaction between electric dipole or magnetic dipole in the material and the electromagnetic field. At a matched thicknesses and a determinate value of the conductivity, the material with high permittivity and permeability has high microwave absorption. These results obviously demonstrate that the intensity and the frequency of the microwave energy absorption for the composite also depend on the rate of the polyaniline and nonmagnetic

content in the matrix.[39] Thus, microwave absorption properties of composite are improved by the dielectric and magnetic losses when the weight fraction of the nanofillers increases.

## CONCLUSIONS

Electromagnetic composites based on polyaniline-doped CSA and PTSA dispersed in polyurethane and epoxy resin matrix with the presence of magnetic nanocharges were successfully prepared by two different methods: Codissolution and dispersion. The SEM observations showed a fiber structure of PANI, and a homogenous dispersion of magnetic charges of CCo and FeNi in the matrix. The codissolution method provides thin materials with high conductivity. A shielding effectiveness superior of 40 and 80 dB in the X and Ku band was obtained using a three layers structure, this value is mostly due to reflections. In addition, we obtained a reflection loss value of −12.2 dB at 17.8 GHz with an optimized multilayered composite. The dispersion method provides thicker materials with moderate conductivity. It was found that values of −24 dB at 11.5 GHz and −17.7 dB at 13.4 GHz were observed in multilayer composite with thicknesses of 1.5 mm. For a thickness of 9.7 mm of PANI-PTSA/FeNi/epoxy resin composite, reflection loss values are −22 dB at 9.52 GHz, and −20.7 dB at 14.7 GHz. These results encourage further development of multilayered shielding and absorbers for broadband applications.

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