



# Electrospun PVC/Fe<sub>3</sub>O<sub>4</sub> composite nanofibers for microwave absorption applications

Ovidiu Chiscan, Ioan Dumitru, Petronel Postolache, Vasile Tura, Alexandru Stancu\*

Department of Physics, Alexandru Ioan Cuza University, Iasi, 700506, Romania

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## ABSTRACT

In this paper we present a study on the microwave frequency properties of PVC/Fe<sub>3</sub>O<sub>4</sub> composite nanofibers, with different amounts of iron oxide nanoparticles incorporate in PVC matrix. Using electrospinning where obtained composite nanofibers with diameters ranging between 100 nm and 600 nm. The transmission loss of PVC/Fe<sub>3</sub>O<sub>4</sub> composites measured in the microwave frequency range of X-band were found to be below – 15 dB demonstrating that these materials can be used as protection material of electromagnetic radiation. The applied magnetic field significantly modifies the measured scattering parameters in our samples case in a rather large domain of values for the field.

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## 1. Introduction

Composite nanofibers consisting of magnetic nanoparticles embedded into a polymer matrix have been under intensive investigation in the last years because of their magnetic-field dependent physical properties. A large number of applications like magnetic cell separation [1], magnetic resonance imaging contrast agents [2], magnetic filters [3], magnetic sensors [4] and low frequency magnetic shielding [5,6] were developed based on their properties.

Among the polymeric nanofiber preparation techniques, electrospinning proved to be a versatile and effective method to prepare fibers with diameters ranging from a few nanometers to one micrometer or more [7,8]. This technique was used over the last decade to prepare nanofibers from a wide range of natural or synthetic polymers [9–12].

The polymeric nanofibers containing magnetic nanoparticles combine the typical properties of a polymeric nanofiber material, i.e. high surface/volume ratio, good mechanical flexibility and high electrical resistivity, with the high magnetic susceptibility of the magnetic nanoparticles, becoming attractive materials for high-frequency electronic applications.

In this paper the influence of magnetic nanoparticle concentration on the high-frequency electromagnetic fields absorption properties of PVC nanofibers containing Fe<sub>3</sub>O<sub>4</sub> nanoparticles was investigated.

## 2. Experimental

The chemical reagents used in this study were: emulsion powder poly(vinyl chloride) – PVC (Vynycl 124) K value 69 supplied by

Polycid, tetrahydrofuran – THF, N,N-dymethylformamide – DMF, particles of iron (II,III) oxide 98% (metals basis) supplied by Alfa Aesar.

At the beginning, a solution of PVC (7.5 g) dissolved in a THF/DMF (36 ml/9 ml) solvent mixture was prepared and shared in three equal parts. Then, each solution part was added with Fe<sub>3</sub>O<sub>4</sub> nanoparticles (size between 20 and 30 nm) in different amounts (sample A – 0.5 g, sample B – 2.5 g, sample C – 5.0 g) and homogenized for 90 min using a mechanical stirrer. These PVC/Fe<sub>3</sub>O<sub>4</sub> solutions were used to prepare composite nanofibers by means of a homemade electrospinning device containing a syringe pump able to provide flow rates between 1.0 and 30 μl/min. The syringe equipped with a 0.7 mm diameter blunt needle was placed vertically above a nanofibers collector. The needle and the collector were connected to a high voltage power supply (0–30 KV). The morphology of the obtained nanofibers was investigated by scanning electron microscopy (SEM), Vega 2 Tescan. The microwave transmission/reflection (T/R) parameters were measured in 8–12 GHz frequency domain using a waveguide fixture and an E8361A PNA Network Analyzer.

## 3. Results and discussion

Fig. 1 shows SEM images of composite nanofibers prepared using the same quantity of PVC matrix and three different amounts of Fe<sub>3</sub>O<sub>4</sub> nanoparticles. These SEM images show that the resulted composite nanofibers consist of Fe<sub>3</sub>O<sub>4</sub> nanoparticles incorporated into PVC nanofibers with diameter range between 100 and 600 nm. It can be seen also that increasing the concentration of Fe<sub>3</sub>O<sub>4</sub> nanoparticles inside the nanofibers, the distance between the enclosed nanoparticles decreases and they tend to agglomerate. The volume of agglomerations increases with nanoparticles concentration. From histograms showed in Fig. 1 it can be observed that for sample (A) (with minimum load of magnetite nanoparticles) composite nanofibers diameter range from 100 to 600 nm. Increasing Fe<sub>3</sub>O<sub>4</sub> loading in PVC matrix we can observe that

\* Corresponding author.

E-mail address: [alstancu@uaic.ro](mailto:alstancu@uaic.ro) (A. Stancu).

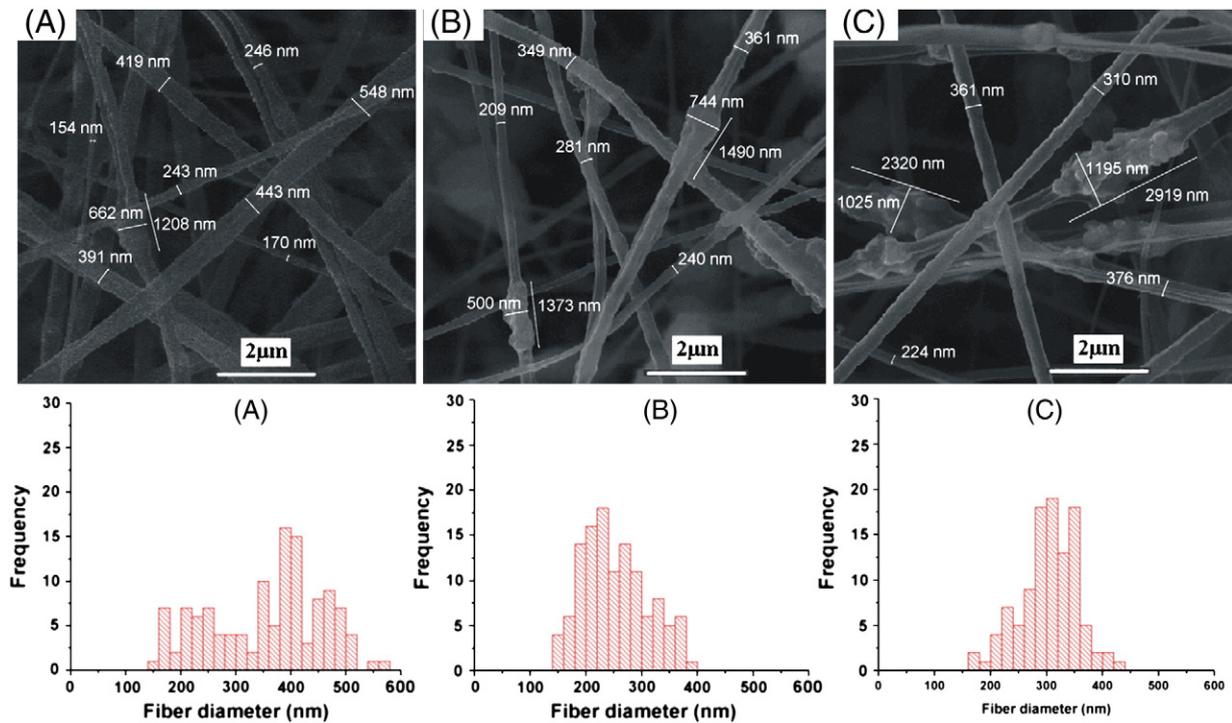


Fig. 1. SEM image and histograms of composite nanofibers with different amount of  $\text{Fe}_3\text{O}_4$  particles: (A) – 0.5 g, (B) – 2.5 g, (C) – 5 g.

the nanofibers diameter tends to a narrower range between 200–350 nm for sample (B) and 250–350 nm for sample (C). It seems that the presence of the particles has a stabilizing effect for the fibers diameter which has to be understood within the theory of the electrospinning process.

Ferromagnetic resonance (FMR) spectra were recorded and transmission/reflection coefficients of the prepared composite nanofibers were measured in order to investigate the behavior of these materials in high frequency electromagnetic fields. The FMR curves were recorded using a rectangular cavity working in TE<sub>102</sub> excitation mode at 9.98 GHz ac magnetic field frequency, sweeping the static magnetic field up to 9000 Oe. From the FMR spectra presented in Fig. 2 it can be seen a shift of the resonant absorption field accompanied by a

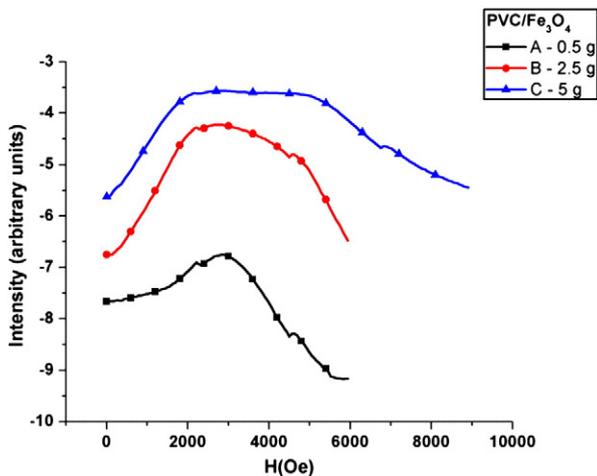


Fig. 2. Resonance spectra of composite nanofibers containing different amounts of  $\text{Fe}_3\text{O}_4$  nanoparticles: (A) – 0.5 g, (B) – 2.5 g, (C) – 5.0 g.

broadening of the resonance curve as the amount of  $\text{Fe}_3\text{O}_4$  nanoparticles dispersed into the PVC matrix increases. This broadening extends the frequency range in which these materials can be used as microwave absorbers.

The reflection/transmission coefficients of the investigated materials placed into a rectangular waveguide fixture were measured in the X band frequency domain for various sample thicknesses. The material samples under test fully fit the waveguide cross section. The samples thickness was changed by increasing the number of nanofiber membrane layers. A layer of as deposited nanofibers membrane had a fixed thickness of 0.8 mm.

By increasing the number of layers a decreasing of transmission coefficient ( $S_{12}$ ) values accompanied by an increase of reflection coefficient ( $S_{11}$ ) was measured (Fig. 3). This means that by varying the nanofibers sample thickness the ratio between reflected and absorbed energy power from the incident electromagnetic wave is modified. We can also see that, for the same number of material layers, with increase of particles concentration embedded in PVC matrix, the values of transmission coefficients decrease, being for sample C around –9.5 dB. Certainly, the most concentration sample has the lowest transmission coefficient value.

In order to investigate the effect of a uniform magnetic field on the transmission/reflection properties of the materials prepared in our work the waveguide containing the samples was placed between the poles of an electromagnet. The thickness of each sample was 4.5 mm. The measured values of the transmission/reflection coefficients for a set of magnetic field values (0–8200 Oe) are presented in Fig. 4. In case of sample A containing 0.5 g of  $\text{Fe}_3\text{O}_4$  nanoparticles embedded in PVC matrix, the transmission/reflection coefficient varies around 0.5 dB when the magnetic field increases from 0 to 8200 Oe. Increasing the amount of  $\text{Fe}_3\text{O}_4$  nanoparticles determine a linear variation of transmission/reflection coefficients (2 dB for sample B and 6 dB for sample C) in the same interval of applied magnetic field. The lowest value of the transmission coefficient was observed in the case of sample C at 3300 Oe and 12 GHz. As expected, the samples with higher particle

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