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Zinc oxide-graphene based composite layers for electromagnetic interference shielding in the GHz frequency range

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ABSTRACT

We report on preliminary results regarding the applicability of nanostructured composite layers for electromagnetic interference shielding in the frequency range of 10–20 GHz. The layers, based on commercially available graphene nanoplatelets and ZnO nanopowder grown using a hydrothermal procedure, were found to induce quite effective attenuation of electromagnetic radiation in the frequency range 10–20 GHz of around −30 dBs, this depending on the ZnO nanopowder/graphene nanoplatelets ratio and the frequency employed.

1. Introduction

Recently, there have been an increased number of applications of electromagnetic waves in the Ku-band (12.4–18 GHz) for radar, military aircraft and satellite communication [1]. As a result, much attention has been devoted to develop material acting as electromagnetic interference (EMI) shields, EMI being a well-known problem in the operation of electronic devices [2]. These shielding materials are able to disable an electromagnetic wave to penetrate into a certain space through an absorption and/or reflection process. Conventionally, conductive metallic panes and meshes are used for isolating spaces or devices from radiation [3]. However, they have many disadvantages such as heaviness, lack of flexibility and high costs of processing. In addition, some metals have an intrinsic cut-off frequency, normally below or not far from the low-GHz range, which restricts their use for GHz-shielding applications.

The present needs are thus to find broad-band shields, able to neutralize electromagnetic radiation up to the GHz frequency range, a requirement arising from the fast development of electronics, operating at enhanced data transfer speeds that require higher frequencies [4]. Furthermore, the miniaturization of such components demands high performance and lightweight manufacturing materials. Nanocomposite polymeric materials offer several advantages over traditional metals and ceramics used for EMI shielding since they can be easily shaped into a wide variety of morphologies and are substantially lighter. Since polymers are electromagnetically transparent, the incorporation of suitable conductive nanoparticles is required for effective EMI shielding, while, the tailoring of their properties offers the possibility of tuning both the effectiveness and the frequency range of applications. Regarding the type of nanoparticles that can be used, three main alternatives exist, metals, metal oxide and carbonaceous. Reports have shown that the respective electromagnetic interference shielding is influenced by the enhancement of the electrical conductivity after their addition [5]. The incorporation of metal particles or nanowires from Ag [6,7] or Cu [7,8] have been reported, which however suffer from corrosion, and in addition, it is difficult to obtain their good dispersion and efficient incorporation into polymers. Carbonaceous particles in contrast, have gained

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increasing attention since they are chemically resistant, lightweight and more compatible with polymers. Graphite, carbon black, carbon fibers, carbon nanofibers and graphene have already been investigated for use as shielding materials in the high frequency range [9–13]. Finally, composites containing metal oxide nanostructures [14–18] are also potential candidates for effective EMI shielding, a prospect supported by the possibility of having metal oxide nanostructures of various morphologies with excellent properties using simple, low cost and environmental friendly chemical growth techniques to control their characteristics through the deposition parameters [19,20].

Graphene based materials, engineered via simple, low cost preparation techniques were recently studied in our laboratory, showing highly effective electromagnetic interference shielding in the GHz frequency range [12]. However, these graphene based formulations are extremely black, which is not the most desired color for presentable applications. On the other hand, as already mentioned in the literature, pure and doped ZnO is a good candidate for these applications, which has also the advantage of white color. In this work, we present preliminary results regarding ZnO nanopowder/graphene nanoplatelets based composite layers suitable for electromagnetic interference shielding in the 10–20 GHz frequency range, a composite material that never tested before for such an application. For this purpose, commercially available graphene platelets were employed, while, ZnO nanopowder was developed using a simple, low cost and environmental friendly hydrothermal procedure [21–24].

2. Experimental

The electromagnetic interference shielding layers, with thickness of around 900 nm, were deposited on 16 cm by 16 cm foam board by brushing paint-like dispersions in deionized water. Commercially available graphene nanoplatelets were used, provided by EMFUTUR Technologies Ltd. Spain, with 5 μm width, 5 nm thickness and a bulk density of 0.03 to 0.1 g/cm^3. The carbon content of the graphene nanoplatelets was >99.5 wt%, the oxygen content < 0.1%, while, a residual acid content of <0.5 wt% existed. The synthesis of ZnO nanopowder was performed using zinc acetate ($Zn(CH_3COO)_2$) as zinc precursor through the hydrothermal procedure. In the initial stage of the preparation, two approaches were employed so that the effect of oxygen source was studied, using H_2O (50 ml) in the first case and a ratio of 2-propanol:H_2O (50 ml:0.2 ml) in the second one, with the addition of 0.4 g $Zn(CH_3COO)_2$ in both cases. The solution was placed in Pyrex glass bottles with autoclavable screw caps and heated at 95 °C for a set period varied between 24 and 72 h. After the end of each induction period, the excess of solution was removed from the bottle and the as-grown powder was dried in a laboratory oven for 24 h at 95 °C. The characteristics of the ZnO powder were investigated using X-ray diffraction (XRD) in a Siemens D5000 Diffractometer with Cu K_α ($\lambda = 1.54056$ Å) for 2-theta = 30.00–70.00°, step size 0.05° and step time 5 min/°. Moreover, their morphology and content were examined using scanning electron microscopy (SEM) and Energy Dispersive Spectroscopy (EDS), with a JEOL JSM 6362LV electron microscope and an INCA X-act dry cooling detector (Oxford) attached to the SEM, the respective operating parameters being: HV mode, operating voltage 20 kV, magnification 20,000, collection time corresponding to minimum 500,000 counts, in order to have a good accuracy.

During the preparation of the paint like dispersions, all compositions were slowly stirred for 2 h in order to remove the trapped air bubbles and to ensure reasonable macroscopic homogeneity. The as prepared mixtures were spread on the foam board substrates using a brush follow by natural air drying. The deposition procedure was repeated for several times until a material of the required thickness was prepared. Samples were visually inspected and characterized by SEM and EDS.

Finally, the transmission measurements of the prepared samples were performed in air, using a Hewlett-Packard 8722 ES vector network analyzer and four sets of microwave standard-gain horn antennas covering the range 10–20 GHz. Prior to every measurement, an absorbing chamber was created using typical microwave absorbers (ECCOSORB AN-77, Emerson & Cuming Microwave Products, Inc., Randolph, MA) over all surfaces except the top, and each sample was placed in the middle of each set of horn antennas.

3. Results and discussion

Since a large quantity of ZnO nanopowder with suitable characteristics was required, the initial trials were focused on the optimization of the growth. Two approaches were chosen (based on a sufficient amount of powder prepared) and employed: (a) a solution of 50 ml H_2O and 0.4 g $Zn(CH_3COO)_2$ for synthesis period of 72 h and (b) a solution of 50 ml 2-propanol and 0.2 ml H_2O, keeping the amount of zinc precursor and growth period constant. The structural and morphological properties of the as grown ZnO nanopowder were then studied in XRD and SEM. As found out, the second approach was more promising since it resulted in homogeneous and uniform spherical grains with a diameter 100–200 nm, as shown in Fig. 1. Moreover, the respective nanopowder had better crystallinity, as one can observe in Fig. 2, which presents XRD patterns of the as-prepared powder at 95 °C using a solution based on a ratio of 2-propanol:H_2O. The diffraction peaks are consistent with the wurtzite ZnO hexagonal P6(3)mc structure (according to JCPDS card file No. 36-1451), the pattern indicating all the characteristic peaks of ZnO, while the determined lattice parameters values of $\alpha = 3.2504$ Å and $c = 5.2055$ Å indicate a very high crystalline quality of the chemically grown ZnO. Similar XRD patterns were also found for all nanopowder samples checked. In contrast, in the case of the pure water solution, the XRD pattern of the as grown nanopowder was consistent with ε-$Zn(OH)_2$ [19]. Therefore, it seems that the presence of 2-propanol compared with solely H_2O favored the precipitation of ZnO powder, a behavior similar to that observed in the presence of NaOH [20]. Following these results, a solution made from 50 ml 2-propanol and 0.2 ml H_2O with 0.4 g $Zn(CH_3COO)_2$ for 72 h at 95 °C was repeated for ten times for the growth of 5 g ZnO nanopowder, which was further used for the preparation of the composites.

Regarding the preparation of the composite layers to be tested as electromagnetic shields, the initial idea was to generate an inorganic cement using as binder $ZnCl_2$. However, this approach was unsuccessful, since, although the final layers had good mechanical properties, they were absorbing water from the atmosphere, a behavior causing a rapid degradation of both the samples and their functionality. Therefore, only one layer employing $ZnCl_2$ was finally tested, while the rest of the samples were prepared without any binder, since they presented better stability in the environment and quite effective electromagnetic

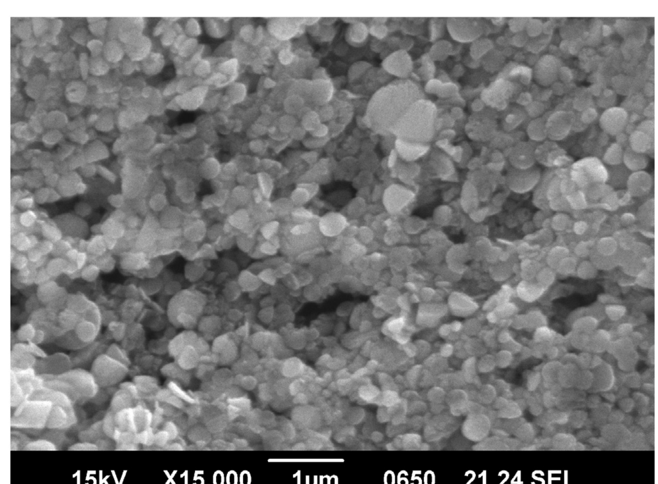

Fig. 1. SEM image of ZnO nanopowder grown using zinc acetate with 2-propanol:H_2O.

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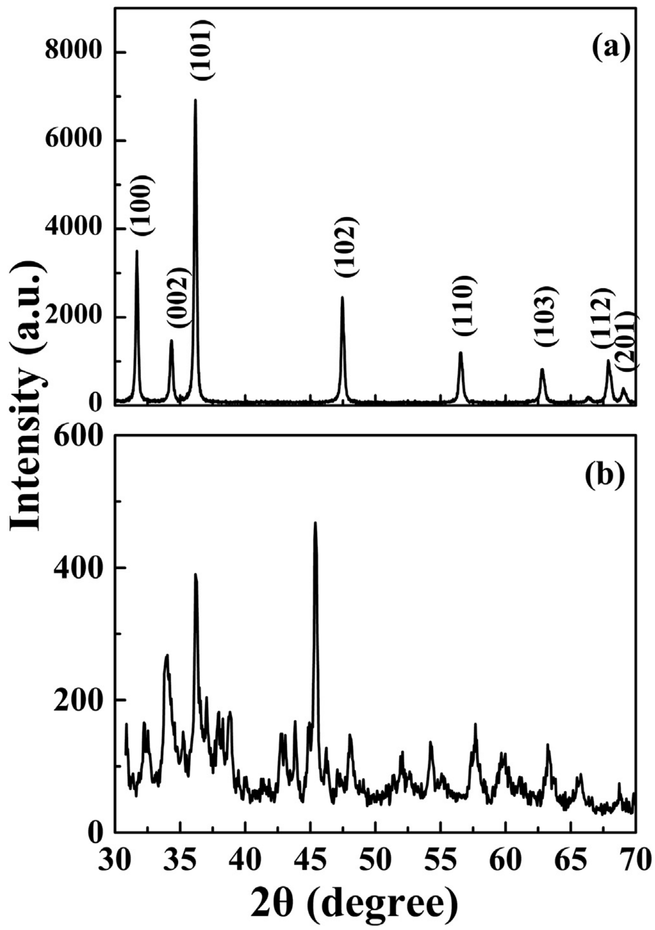

Fig. 2. XRD patterns of: (a) ZnO nanopowder grown using zinc acetate with 2-propanol:H_2O and (b) ZnO nanopowder grown with pure water.

interference shielding effect, as will be shown in the next section. The as prepared ZnO nanopowder/graphene nanoplatelets composite layers were homogeneous, with a dark grey color, but not very compact and in some cases they could flake easily if they were not handled carefully.

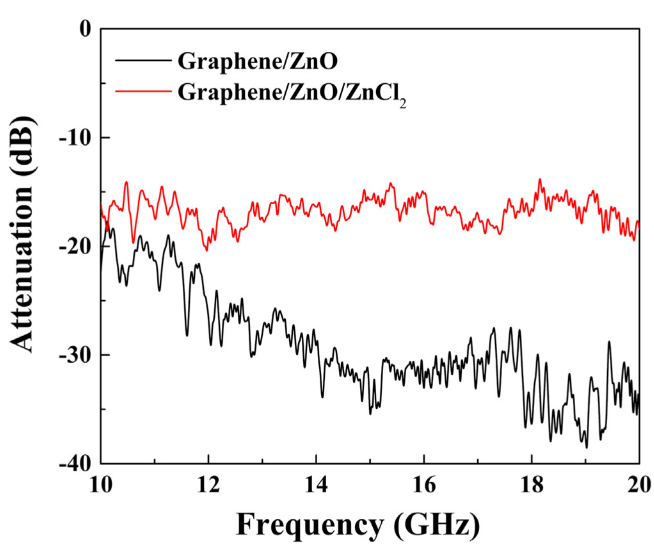

Fig. 3. Electromagnetic interference shielding of nanocomposite layers based on (a) ZnO nanopowder and graphene nanoplatelets and (b) ZnO nanopowder, graphene nanoplatelets and $ZnCl_2$, both having a ZnO nanopowder/graphene nanoplatelets ratio of 1.5.

The electromagnetic interference shielding of the two composite layers was examined and shown in Fig. 3, the layers consisting of (a) ZnO nanopowder and graphene nanoplatelets and (b) ZnO nanopowder, graphene nanoplatelets and $ZnCl_2$, both having the ZnO nanopowder/graphene nanoplatelets ratio of 1.5. As can be seen, the layer with $ZnCl_2$ exhibits an almost constant electromagnetic interference shielding effect, of about −17 dBs, for the frequency region under investigation. The respective layer without $ZnCl_2$ presents a more significant electromagnetic interference shielding effect, this increasing almost monotonously from −20 dBs at 10 GHz up to −38 dBs at 20 GHz. Although both layers present similar shielding effect at about 10–11 GHz, the layer without $ZnCl_2$ is much more effective at higher frequencies, offering attenuation >30 dBs at this frequency range, which is quite effective. Taking into account this result and the fact that the samples with $ZnCl_2$ present a rapid degradation due to the absorption of water from the atmosphere, the investigation was focused on samples consisting of ZnO nanopowder and graphene nanoplatelets only.

Various layers with different ZnO nanopowder/graphene nanoplatelets content, the respective ratio varying between 0.33 and 1.5, were prepared and studied. Fig. 4 presents SEM images of a) the commercial graphene nanoplatelets, b) the composite with the lowest ZnO nanopowder concentration and c) the composite with the highest ZnO nanopowder concentration. The images indicate that all the materials are very conductive (they are quite transparent for the electron beam) and the graphene flakes are quite flat even in the composite. Composite materials are homogeneous and ZnO has a more or less uniform distribution in the composites bulk as small agglomerations, which are more pronounced in the case of the highest ZnO nanopowder concentration. Moreover, the graphene flakes keep their general aspect in the composite materials (they don't agglomerate or crinkle very much), being decorated with ZnO nanoparticles agglomerations.

The prepared composites were also analyzed by EDX. Analysis confirms the expected composition, following the preparation conditions, consisting on Zn, O and C and traces of Ca, Al, Fe, S and Cl. The composites macroscopic homogeneity was confirmed also by mapping the elemental distribution on their surface. Fig. 5 illustrates the elemental composition of the sample with the lowest ZnO nanopowder concentration, where the color code was green for C and red for Zn.

Fig. 6 presents the variation of the electromagnetic wave attenuation as a function of frequency for four composite layers having different ZnO nanopowder/graphene nanoplatelets ratio. The points correspond to the average of various measurements obtained at the same frequency for the same sample. As can be seen, in all cases, electromagnetic interference shielding becomes more effective as the frequency increases. For ZnO nanopowder/graphene nanoplatelets ratios < 1, the rate of increasing of the electromagnetic interference shielding is more or less similar, being quite effective in all the frequency range under investigation, since the respective attenuation values are between −30 dBs (at 10 GHz) up to −38 dBs (at 20 GHz). For the case of the higher ZnO nanopowder/graphene nanoplatelets ratio, the rate of increasing of the electromagnetic interference shielding is larger, the attenuation starting from smaller values at low frequencies but becoming larger at the higher frequencies. Therefore, ZnO nanopowder/graphene nanoplatelets nanocomposite layers are promising candidates for electromagnetic interference shielding in the GHz frequency range. Moreover, the content of ZnO nanopowder in correlation with that of graphene nanoplatelets affects the shielding efficiency, especially if ZnO nanopowder is more than graphene nanoplatelets.

For a better understanding of this relation, the variation of the electromagnetic wave attenuation of the composite layers as a function of the ZnO nanopowder/graphene nanoplatelets ratio was examined for four frequencies, the respective results shown in Fig. 7. The points also correspond to the average of various measurements obtained at the same frequency for the same sample. As can be seen, the actual variation of the electromagnetic wave attenuation as a function of the ZnO

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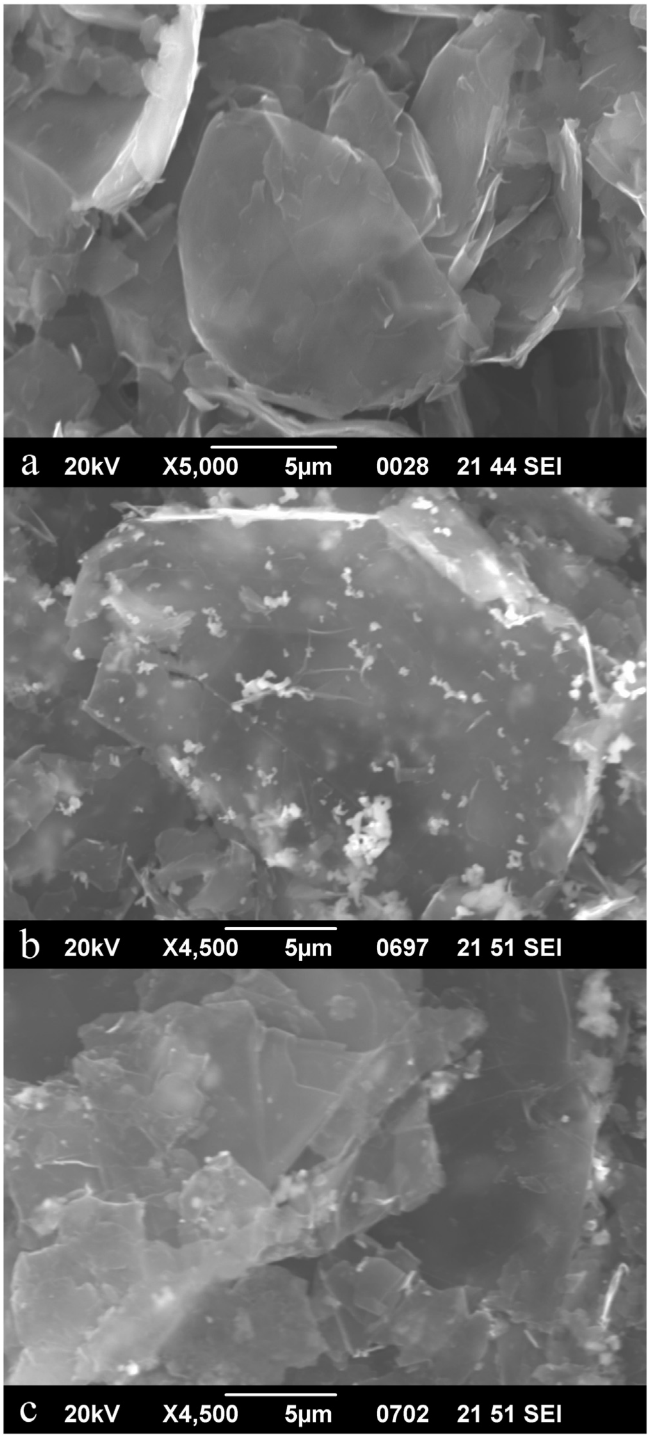

Fig. 4. SEM images of: (a) the commercial graphene flakes, (b) the composite with the lowest ZnO nanopowder/graphene nanoplatelets ratio and (c) the composite with the highest ZnO nanopowder/graphene nanoplatelets.

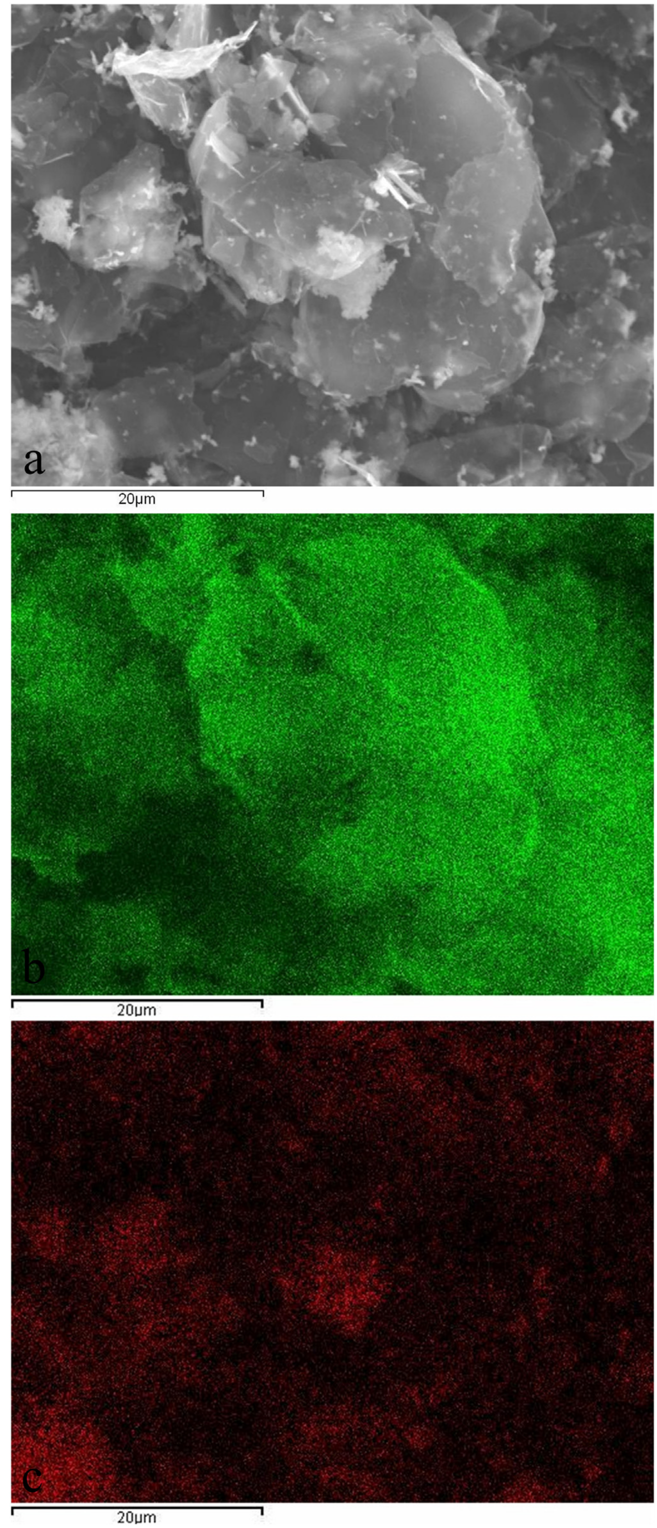

Fig. 5. Elemental composition of for the lowest ZnO nanopowder concentration: (a) SEM image, (b) distribution of C (green color presenting C), (c) distribution of Zn (red color presenting Zn). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

nanopowder/graphene nanoplatelets ratio strongly depends on the frequency. For frequencies lower than 15 GHz, an increasing of the ZnO nanopowder/graphene nanoplatelets ratio results in a decrease of the electromagnetic interference shielding effectiveness, while for frequencies around 20 GHz, the opposite appears. Moreover, at frequencies around 18 GHz, electromagnetic interference shielding is almost unaffected by the ZnO nanopowder/graphene nanoplatelets ratio. Therefore, the shielding efficiency can be tuned at particular frequencies by varying the ZnO nanopowder/graphene nanoplatelets ratio. Moreover, taking into account these observations together with those in Fig. 4, it seems that further investigation is required for better understanding of the response of the composite layers, especially regarding the relative contribution of ZnO nanopowder and graphene nanoplatelets.

Although there exist several reports on electromagnetic interference shielding in the 10–20 GHz frequency of composites based on graphene or ZnO combined with other material, no report exists for their

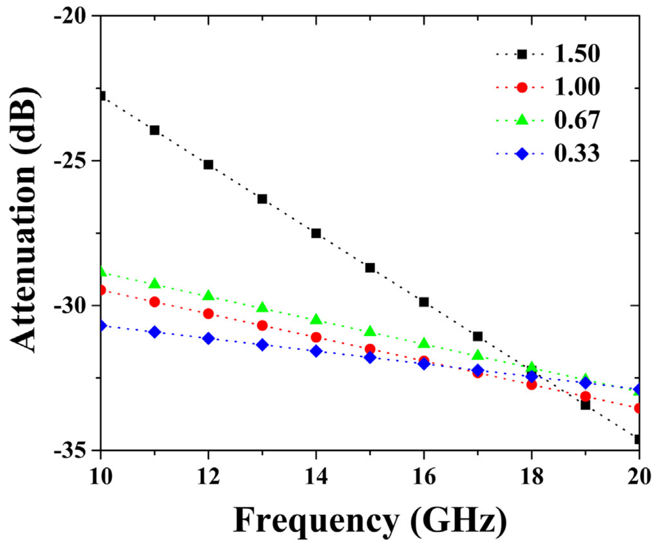

Fig. 6. Variation of electromagnetic wave attenuation as a function of frequency for different ZnO nanopowder/graphene nanoplatelets ratios.

combination. Regarding ZnO combined with carbon based materials, results have published of the electromagnetic interference shielding of zinc oxide particles coated multiwalled carbon nanotubes [25] and core/shell structured C/ZnO nanoparticles [17]. In particular, core/shell structured C/ZnO nanoparticles composites were observed to offer an electromagnetic interference shielding efficiency of about −14 dBs at 12 GHz [17], while, in the case of multiwalled carbon nanotubes coated with zinc oxide particles, this was found to be around −10 dBs [25]. Moreover, in the case of multi-wall carbon nanotubes decorated with ZnO nanocrystals, the respective efficiency was around −20 dBs [14]. Taking into account their findings, the composites studied here seem to be very promising, although their composition should be improved so that their physical properties will become more suitable for commercial products. Regarding the respective shielding mechanisms, these are still under investigation. In any case, the electromagnetic interference shielding in a materials comes from reflection due to the electrical conductivity and/or absorption by electric or magnetic dipoles of the electromagnetic radiation. Following the literature, the observed shielding could be attributed to microwave-absorption [25], as well as it can be

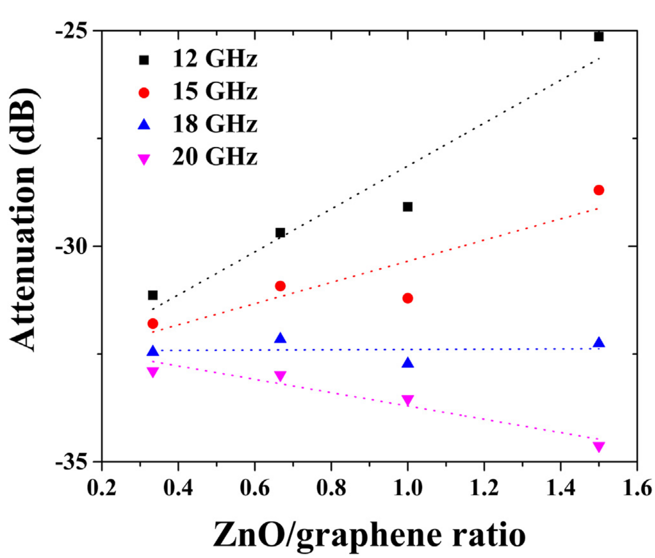

Fig. 7. Variation of electromagnetic wave attenuation of the composite layers as a function of their ZnO nanopowder/graphene nanoplatelets ratio for various frequencies.

based on orientational polarization, space charge polarization and electrical conductivity [17].

4. Conclusions

Results were presented related to the electromagnetic interference shielding performance of paint-like nanocomposite layers consisting of commercially available graphene nanoplatelets and homemade ZnO nanopowder. ZnO nanopowder was grown using a simple, low cost and environmental friendly hydrothermal procedure, employing solutions consisting of 2-propanol, H_2O and $Zn(CH_3COO)_2$ for synthesis period of 72 h and a temperature of 95 °C, resulting in spherical wurtzite ZnO grains with a diameter 100–200 nm. The graphene flakes were found to be quite flat even in the composite material, where, ZnO was presenting a uniform distribution, exhibiting agglomerations. The graphene nanoplatelets and ZnO nanopowder composite layers were inducing quite effective attenuation of electromagnetic radiation in the frequency range 10–20 GHz. The content of ZnO nanopowder was found to affect the shielding efficiency, especially if this was more than graphene nanoplatelets. Moreover, the shielding performance was depending on the electromagnetic wave frequency. Therefore, one may conclude that ZnO nanopowder/graphene nanoplatelets composites are promising candidates for electromagnetic interference shielding. In addition, these composites can be easily engineered to lower the cost and tune the color from black to lighter grey shades. Further studies will be performed in this scope.

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