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In this study, the effect of carbon nanotubes (CNT) and graphene nanoplatelets (GNP) at various weight ratios between them on the dielectric (dielectric permittivity, dielectric loss angle tangent) and microwave (reflection coefficient, attenuation coefficient, shielding effectiveness) properties of nanocomposites on the basis of natural rubber has been investigated in the wide frequency range (1–12 GHz). Some additional investigations on the morphology and microstructure of the graphene particles and CNT used have been carried out by transmission electron microscopy and selected area electron diffraction. The results achieved reveal how by using the selected combinations between CNT and GNP the microwave and dielectric properties of natural rubber nanocomposites can be tailored depending on the need for real practical application.

Keywords: rubber composites; carbon nanotubes; graphene nanoplatelets; dielectric and microwave properties

Introduction

Polymers and polymer matrix composite materials are being utilized in an increasing number of industrial applications including transportation, automotive, aerospace, defense, sporting goods, energy, and infrastructure sectors. This is due to their high durability, high strength, light weight, design and process flexibility, etc.

An especial space among this group of materials the polymer nanocomposites occupies. They are commonly defined as the combination of a polymer matrix and additives that have at least one dimension in the nanometer range. The additives can be one dimensional, such as nanotubes and fibers, two dimensional, which include layered clay minerals or graphene sheets, or three dimensional, including spherical particles.[1]

At present, nanocomposites employing carbon-based reinforcement materials are dominated by carbon nanotubes (CNTs).[2–4] However, the development of CNT-reinforced

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composites has been impeded by both their difficult dispersion in the polymer matrix and their high cost.

The discovery of graphene [5] and the subsequent development of graphene-based polymer nanocomposites are an important addition in the area of nanoscience and technology. Deservedly the Nobel Prize in Physics for 2010 was awarded to Andre Geim and Konstantin Novoselov from the University of Manchester for their work on a single free-standing atomic layer of carbon (graphene). Graphene is an allotrope of carbon, whose structure is one-atom-thick planar sheets of sp^2-bonded carbon atoms packed in a honeycomb lattice. It is the basic structural element of some carbon allotropes including graphite, charcoal, carbon nanotubes, and fullerenes.

Compared to carbon nanotubes, as well as its high aspect ratio and low density, graphene has attracted considerable attention because of its unique and outstanding mechanical, electrical, and electronic properties.[6] In addition to good thermal conductivity, remarkable mechanical stiffness, and high fracture strength, graphene has been supposed to be a semiconductor with zero gap which is quite different from conventional silicon semiconductors. In graphene, electrons shoot along with minimal resistance which may allow for low-power, faster-switching transistor and become a candidate to replace silicon in the area of microchip electronics.

All these unique properties in a single nanomaterial have made physicists, chemists, and material scientists exited about graphene's potential. The history, chemistry, preparation methods and possible applications of graphene are reviewed in.[7–9] Another new review focused on trends and frontiers in graphene-based polymer nanocomposites was published an year ago.[10] Recently, the trustees of Princeton University received a patent for graphene-elastomer nanocomposites where functionalized graphene sheets had been dispersed in vulcanized natural rubber, styrene-butadiene rubber, Ps-isoprene-Ps, and PDMS.[11] The authors conclude that graphene-rubber nanocomposites possess qualities like those of carbon nanotube composites but are much cheaper to make.

The data about graphene influence on the microwave properties of the elastomeric composites are very scarce. Chen et al. used functionalized graphene–epoxy composites as lightweight shielding materials for electromagnetic radiation.[12] De Rosa and coworkers have a wide expertise in the design of micro/nanocomposites based on carbon fibers and carbon nanotubes, for the realization of high performing radar absorbing screens, with tailored properties.[13–15] In a recent paper,[16] the authors have accomplished a Salisbury screen that consists of three layers. The first layer consists of a thick metal panel. The second layer (the spacer) is a low-loss-tangent nanocomposite based on a Bisphenol-A-based epoxy resin filled with graphene nanoplatelets (GNPs) at 0.5% and 1% wt. The third layer is the lossy sheet which function is to absorb the EM energy associated with the incident field. This lossy sheet is realized by the epoxy-based nanocomposite made with an unsaturated polyester resin filled with Ni-coated carbon fibers and multiwall CNTs. The real and imaginary parts of the complex effective permittivity within the 8–18 GHz range of the nanocomposite filled with GNPs have been shown. It has been observed that the real part of the effective permittivity is nearly constant.

There are no literature data about the dielectric and microwave properties of nanocomposites comprising a combination of CNT and graphene (GNP). Neither there are available reports on investigations on those properties carried out in a wider frequency range, first of all at frequencies lower than 8 GHz. Therefore, the aim of this work is to study the influence that the combination (CNT–GNP) has on the dielectric (dielectric permittivity, dielectric loss angle tangent) and microwave properties (absorption and reflection of the electromagnetic waves, the effectiveness of the

electromagnetic shielding) of natural rubber-based composites in a significantly greater frequency range – from 1 to 12 GHz as well as the possibilities to tailor this properties by the ratio between CNT and GNP.

Experimental

Materials

Natural rubber (SMR 10) was purchased from North Special Rubber Corporation of Hengshui, Hebei Province, China. Other ingredients such as zinc oxide (ZnO), stearic acid (SA), Mercapto benzothiazole sulphenamide (MBTS), Tetramethyl thiuram disulfide (TMTD), Dimethylbutyl-phenyl-p-phenylendiamine (Vulkanox 4020), and sulfur (S) were commercial grades and used without further purification.

Characterization of the carbon nanotubes used

Multiwalled CNT as produced by Hayzen Engineering Co., Ankara, Turkey, were used in our investigation. The material's purity is more than 95%, density – 150 kg/m^3. CNTwere with average diameter about 15 nm and length 1–10 microns (Figure 1).The pattern taken in an electron diffraction regime (insert in the right corner) shows the typical polycrystal structure of CNT.

Characterization of the graphene used

Graphene used in the investigation was also produced by Hayzen Engineering Co., Ankara, Turkey. GNP have a 'platelet' morphology, meaning they have a very thin but wide aspect. Aspect ratios for this material can range into the thousands. Each particle consists of several sheets of graphene with an overall thickness of 50 nm and average plate diameter 40 μm. The pattern taken in selected area electron diffraction (SAED) regime shows a considerable number of spot-like reflections typical for single-crystal structures (Figure 2 – insert in the right corner).

Preparation of rubber composites

Table 1 summarizes the formulation characteristics of the rubber compounds (in phr) used for the investigations.

First, mixtures of CNT with graphene (GNP) were prepared by grinding them together in a grinding machine for 1 h. The rubber compounds were prepared on an open two-roll laboratory mill (L/D 320 × 360 and friction 1.27) by incorporating the filler mixtures into a natural rubber matrix at various loadings (Table 1). The speed of the slow roll was 25 min^{-1}. The experiments were repeated for verifying the statistical significance. The ready compounds in the form of sheets stayed 24 h prior to their vulcanization.

The optimal vulcanization time was determined by the vulcanization isotherms, taken on an oscillating disk vulcameter MDR 2000 (Alpha Technologies) at 150 °C according to ISO 3417:2002.

These composites were evaluated for their dielectric (dielectric permittivity, dielectric loss angle tangent) and microwave (reflection coefficient, attenuation coefficient, shielding effectiveness) properties in the 1–12 GHz frequency range.

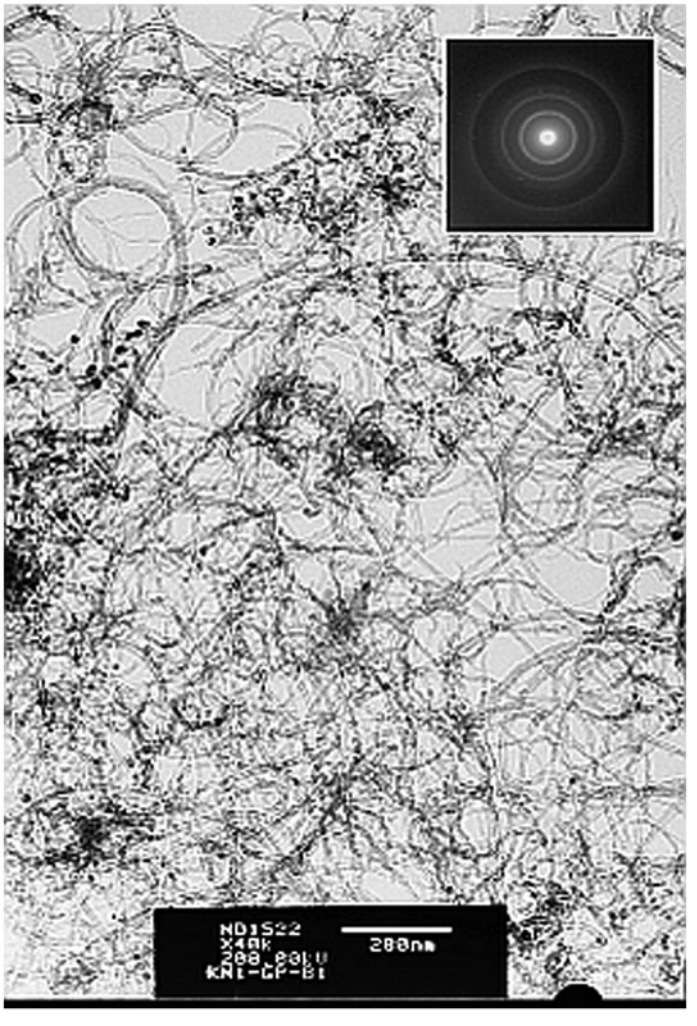

Figure 1. TEM micrographs of CNT in transmission regime and SAED regime (insert in the right corner).

Characterization and measurements

Reflection and attenuation

Measurements of reflection and attenuation were carried out using the measurement of output (adopted) power P_a in the output of a measuring line without losses, where samples of materials may be included. Because of the wide frequency measurement, a coaxial line was used. Samples of materials were shaped like disks with an external diameter $D = 20.6$ mm, equal to the outer diameter of the coaxial line and thickness $\Delta = 2$ mm. The internal diameter is depending on the relative dielectric permittivity of the material.

Part of the incident electromagnetic wave with power $P_{in.}$ on the sample was reflected from it. The rest of the wave with power P_p penetrates the material so that the attenuation L depends on the coefficient of reflection $|\Gamma|$. His module is determined by a reflect meter.

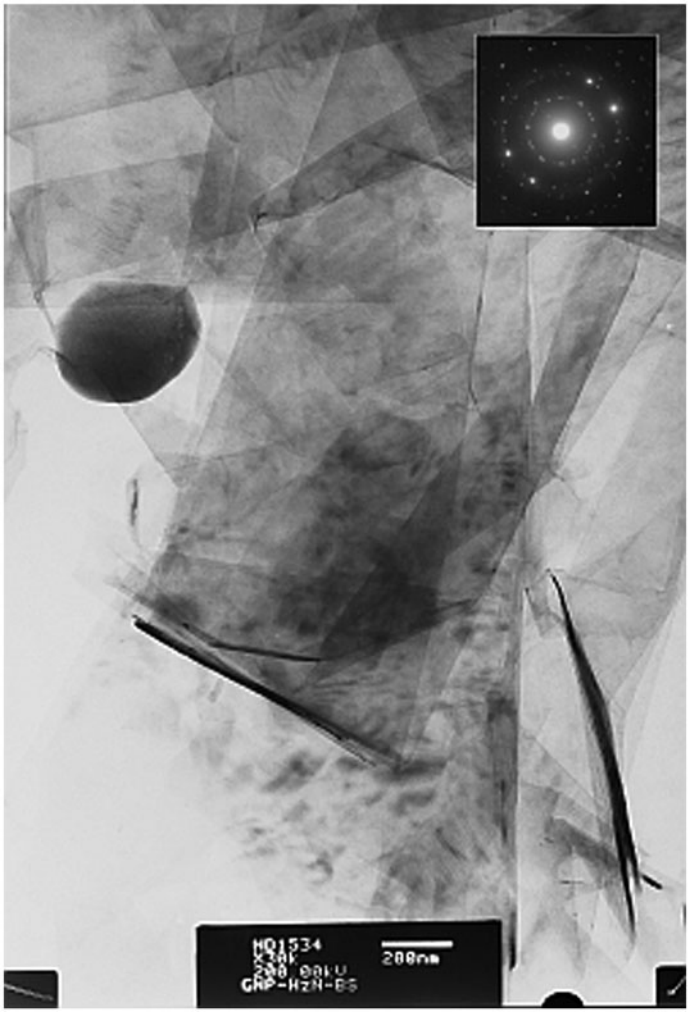

Figure 2. TEM micrographs of GNP in transmission regime and SAED regime (in the right corner).

So attenuation is determined by

$$L = 10 \log \frac{P_a}{P_p}, \text{ dB}, \tag{1}$$

where

$$P_p = P_{in.}(1 - |\Gamma|^2) \tag{2}$$

For the testing of both parameters is used, the scheme consisting of a set of generators for the whole range HP686A and G4 – 79 to 82 (1), coaxial section of the deck E2M Orion, with samples of material (2), Power meter HP432A (3), Scalar reflectance meter HP416A (4) and Reflect meter (R) including two directional couplers Narda 4222.16 and two crystal detectors Narda 4503-N (Figure 3).

Table 1. Composition of the rubber compounds studied.

	NR1	NR2	NR3	NR4	NR5	NR6
Natural rubber	100	100	100	100	100	100
Foaming agent	8	8	8	8	8	8
Stearic acid	1	1	1	1	1	1
Zinc oxide	4	4	4	4	4	4
Processing oil	10	10	10	10	10	10
CNT	0	2	4	6	8	10
GNP	10	8	6	4	2	0
MBTS[a]	2	2	2	2	2	2
TMTD[b]	1	1	1	1	1	1
Vulkanox 4020[c]	1	1	1	1	1	1
Sulphur	2	2	2	2	2	2

[a]Mercapto benzothiazole sulphenamide.
[b]Tetramethyl thiuram disulphide.
[c]Dimethylbutyl-phenyl-p-phenylendiamine.

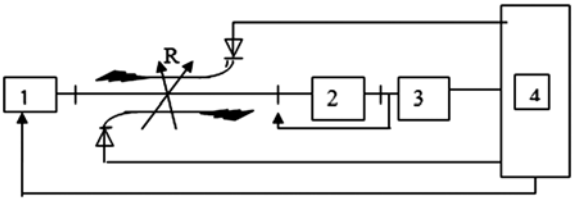

Figure 3. Scheme of the equipment for measuring the microwave properties.

Sheilding effectiveness (S.E.)

This parameter is defined as the sum of the reflection losses R, dB and attenuation L, dB in the material.[17] It can be directly measured or calculated from the measured reflectance and attenuation in the material. In the first case, as measured incident power on the sample P_{in} and adopted after the sample P_a (Figure 3), S.E. is determined by

$$\text{S.E.} = 10 \log \frac{P_0}{P_a}, \text{ dB} \tag{3}$$

In the second, if known reflection and absorption in the material, S.E. is determined, by definition, as

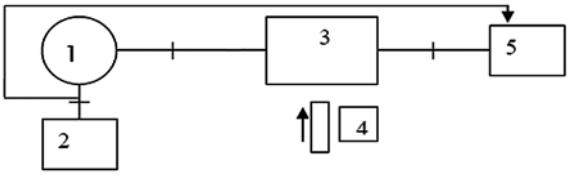

Figure 4. Scheme of the equipment for measuring the dielectric properties.

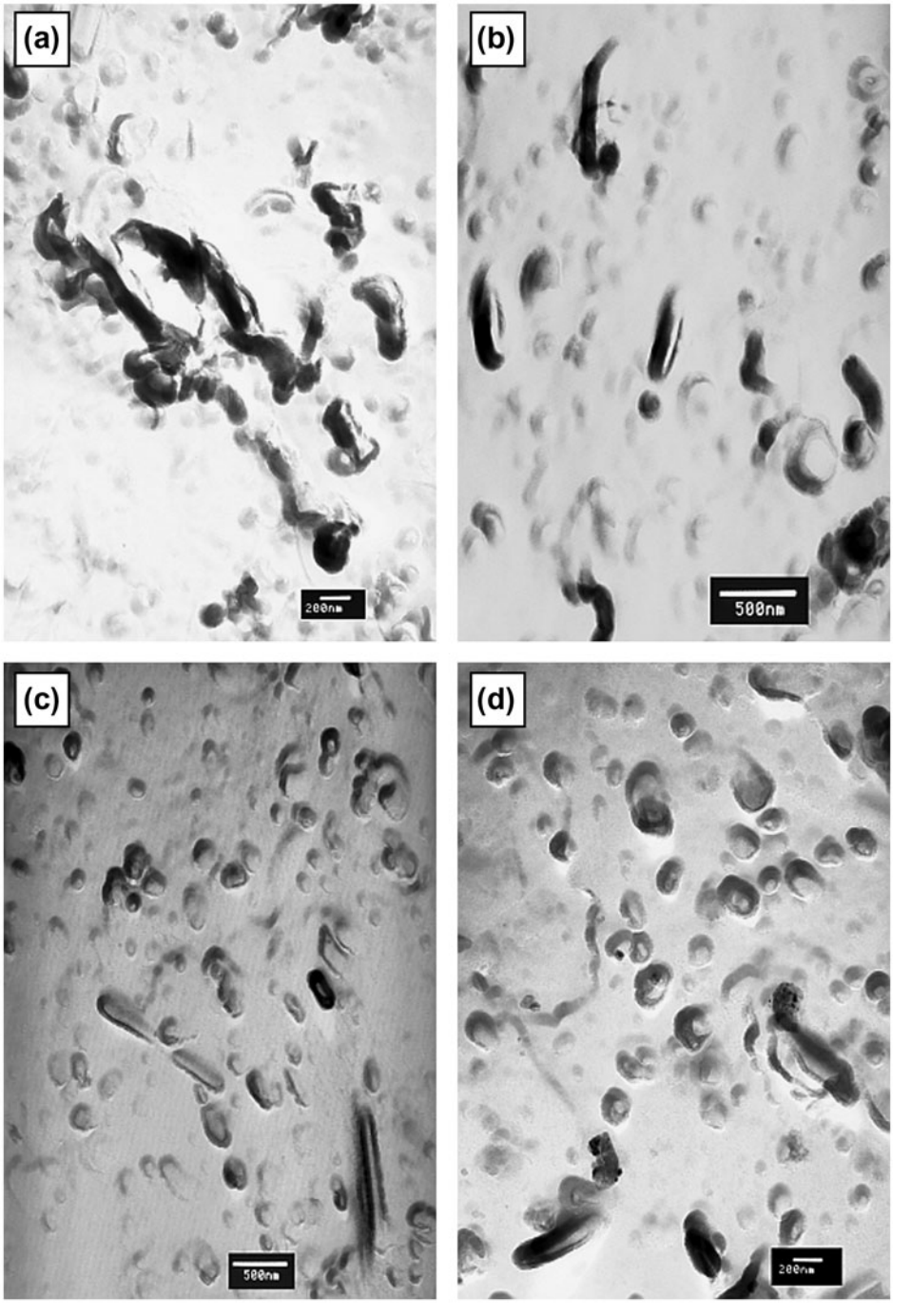

Figure 5. TEM micrographs of NR composites containing different amount in phr of graphene nanoplatelets (m) and carbon nanotubes (n): (a) – $m/n = 2/8$; (b) – $m/n = 4/6$; (c) – $m/n = 6/4$ and (d) – $m/n = 8/2$; (e) – $m/n = 10/0$; (f) – $m/n = 0/10$.

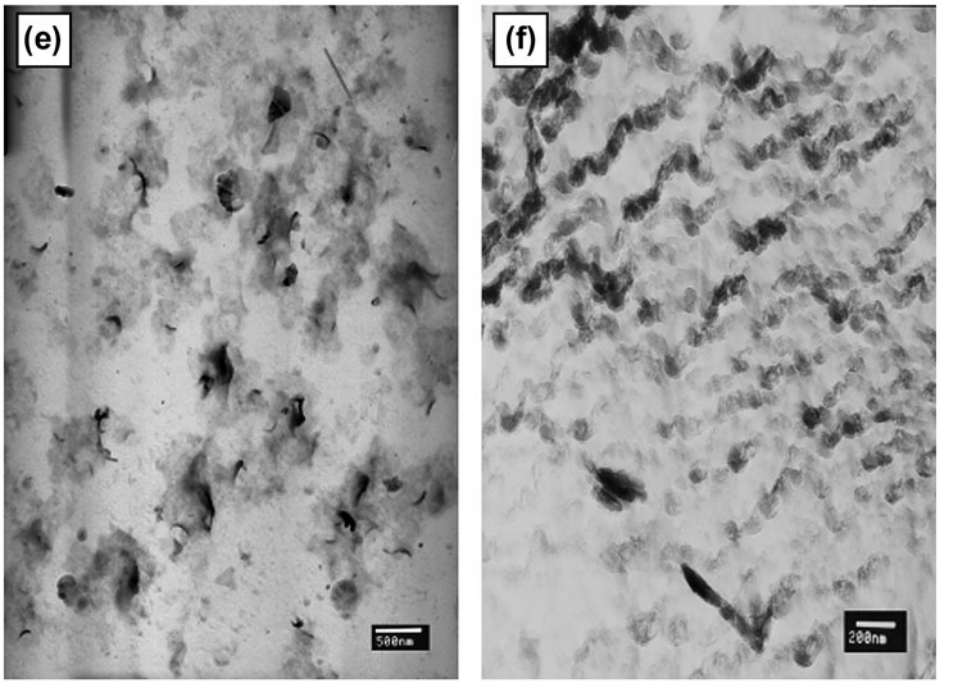

Figure 5. (*Continued*)

$$\text{S.E.} = R, \text{dB} + L, \text{dB}, \tag{4}$$

where R, dB is the attenuation due to the reflection of power at the interfaces.

Complex permittivity

The determination of complex permittivity is carried out by the resonance method, based on the cavity perturbation technique.[18]

Measuring resonance frequency of empty cavity resonator f_r and then measuring the shift in resonance frequency with the sample material f_ε. Then, the dielectric constant ε_r is calculated from the shift in resonance frequency, cavity, and the sample cross-sections S_r and S_ε, respectively

$$\varepsilon_r = 1 + \frac{S_r}{2S_\varepsilon}.\frac{f_r - f_\varepsilon}{f_r}. \tag{5}$$

The sample is in the form of a disk with a diameter of 10 mm and thickness about 2 mm. It is placed at the maximum electric field location of the cavity. Because the thickness of the sample is not equal to the height of the resonator, in the place of its inclusion obtains a dielectric with an equivalent permittivity ε_e, which is determined by (5) and instead ε_r be saved ε_e. Then, ε_r is determined by

$$\varepsilon_r = \varepsilon_e(k+1) - k, \quad (\Delta \ll 1), \tag{6}$$

where $k = l/\Delta$ and l is the distance from the disk to the top of the resonator.

Loss factor tan δ

The loss factor tan δ is calculated from quality factor of the cavity with Q_ε and without sample Q_r

$$\tan\delta = \frac{1}{4\varepsilon_r}\frac{S_r}{S_\varepsilon}\left(\frac{1}{Q_\varepsilon} - \frac{1}{Q_r}\right). \tag{7}$$

The measurement set-up uses several cavity resonators for the whole range, generators for the whole range, frequency meter, and oscilloscope. The measurement set-up used several generators for the whole range: HP686A and G4 - 79 to 82 (1), frequency meters: H 532A; FS – 54 (2), Cavity resonator (3), Sample (4), and Oscilloscope EO 213 (5) (Figure 4).

TEM micrographs

The particle size, size distribution of the fillers, and microstructure of the composites were determined using a TEM JEOL 2100 at accelerating voltage 200 kV. The specimens were prepared by grinding the samples in an agate mortar and dispersing them in ethanol by ultrasonic treatment for 6 min. A droplet of the suspension was dripped on standard carbon films on Cu grids. Additional data for crystal structure were obtained

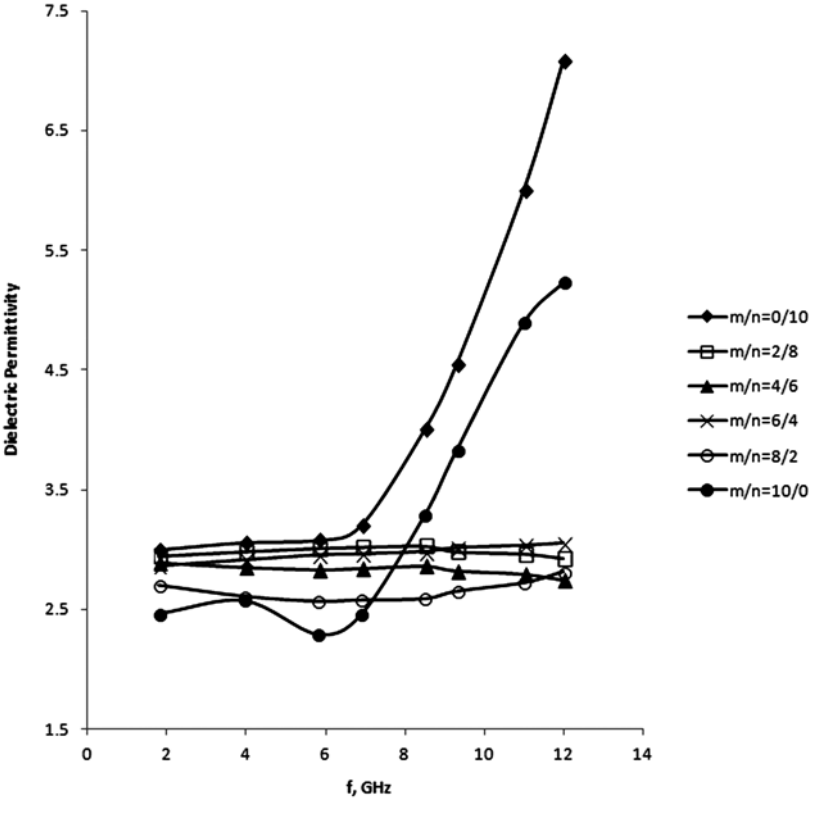

Figure 6. Effect of different ratios of graphene nanoplatelets (m) and carbon nanotubes (n) on relative dielectric permittivity of NR-based nanocomposites.

using SAED method. Standard carbon extraction replica technique was used for transmission electron microscopy (TEM) investigations of fractured at temperature of liquid nitrogen NR rubber composites samples, containing different amounts of CNTand graphene.

Results and discussion

We used TEM to investigate the CNT aggregates and graphene particles and their dispersion. Figure 5(a–f) presents TEM micrographs of NR composites containing different amount ratio CNT-GNP. Figure 5(e) corresponds to NR1 (only GNP 10 phr), and Figure 5(f) corresponds to NR6 (only CNT 10 phr).

The micrographs on Figure 5 show that the fillers particles are comparatively evenly dispersed in the elastomeric matrix, and it is characterized with adequate homogeneity that guarantees good interaction with the electromagnetic waves. In Figure 5(a) stand out clearly the CNTbecause in this composite CNT quantity is the biggest ($n = 8$) by contrast with Figure 5 (d) where the GNPs predominate ($n = 2$). However, the CNTshown on Figure 1 have undergone an additional disaggregation in the process of compounding, as a result of which the sizes of CNTs in the composites have decreased.

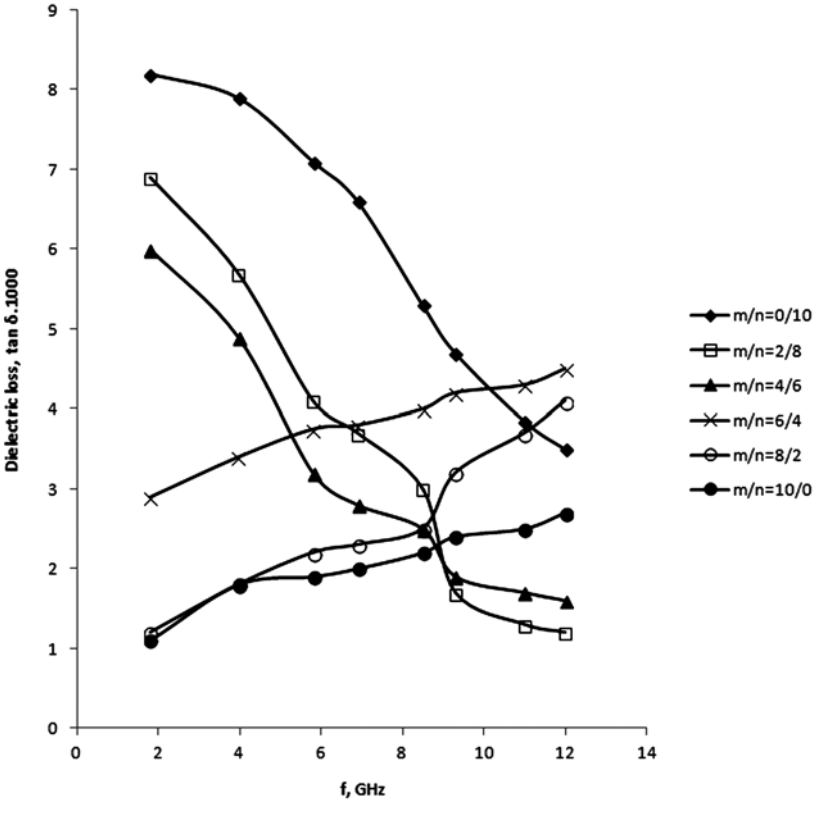

Figure 7. Effect of different ratios of graphene nanoplatelets (m) and carbon nanotubes (n) on dielectric loss angle tangent of NR-based nanocomposites.

The dielectric and microwave properties of natural rubber-based nanocomposites, containing different weight ratios of CNTand GNPs, are shown in Figures 6–10.

Complex relative permittivity: real part (relative dielectric permittivity)

The dependence on frequency and on the correlation between the two fillers at constant total amount is shown in Figure 6. There are two radically different trends depending on the ratio (in phr) between the two fillers.

The first two curves which correspond to $m/n = 2/8$ and 4/6, that is, in excess of carbon nanotubes, tend to reduce ε_r with frequency and to increase it with the increase in the amount of nanotubes. The variation is in small ranges. When reversing the ratio in favor of graphene (GNP), that is, where $m/n = 6/4$ and 8/2, ε_r increases with the frequency increase and decreases with the growth in the amount of graphene. As a general conclusion, it can be assumed that the dielectric permittivity varies in a small range – up to 0.5, which includes the measurement errors.

There is another important factor: the test material is porous (containing foaming agent), and the measured values actually exhibit the equivalent value of the dielectric permittivity with the participation of the pores created. It can be expected that ε_r of the material is greater, if it is thick. The same applies to the dielectric losses.

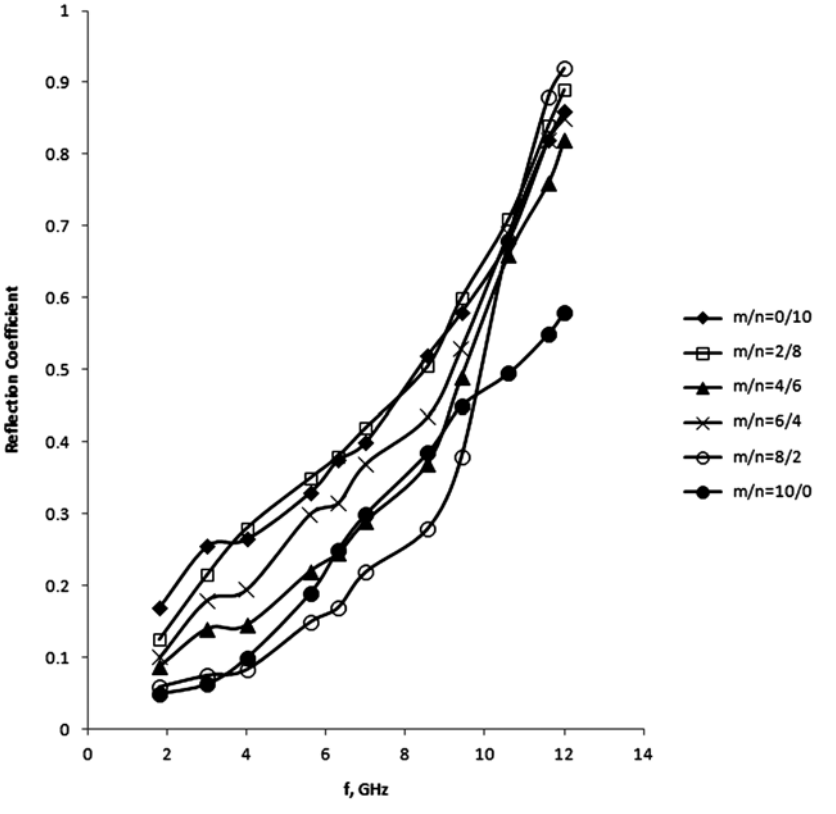

Figure 8. Effect of different ratios of graphene nanoplatelets (m) and carbon nanotubes (n) on coefficient of reflection of NR-based nanocomposites.

Complex relative permittivity: imaginary part (dielectric loss)

The nature of variation of the dielectric loss is shown in Figure 7. With the predominance of carbon nanotubes, that is, $m/n = 2/8$ and 4/6, losses decrease in the range of $(5.7–1.2) \times 10^{-3}$. When reversing the ratio in favor of graphene nanoparticles, that is, $m/n = 6/4$ and 8/2, the losses increase in a much smaller range, but they are different for the various ratios. This shows that by changing the ratio of mass proportions of the two fillers, the variation direction of the real and imaginary part of complex permittivity (depending on the particular application) can be controlled.

Coefficient of reflection

The coefficient of reflection $|\Gamma|$ (Figure 8) varies widely, from 0.125 in the beginning of the range to 0.92 for the higher frequency. The functions have an approximately constant gradient, regardless of the ratio m/n. For frequency 10.6 GHz, a change of the position of the curves is observed for $m/n = 8/2$, $|\Gamma|$ it has a greater value than any other. However, in general, nanocomposites with higher CNT content tend to have a higher value of the reflection coefficient compared to composites containing a greater amount of graphene, that is, CNT contribute to higher reflection than GNP. With the increase in the frequency over 11 GHz, the opposite trend is observed – composites with higher

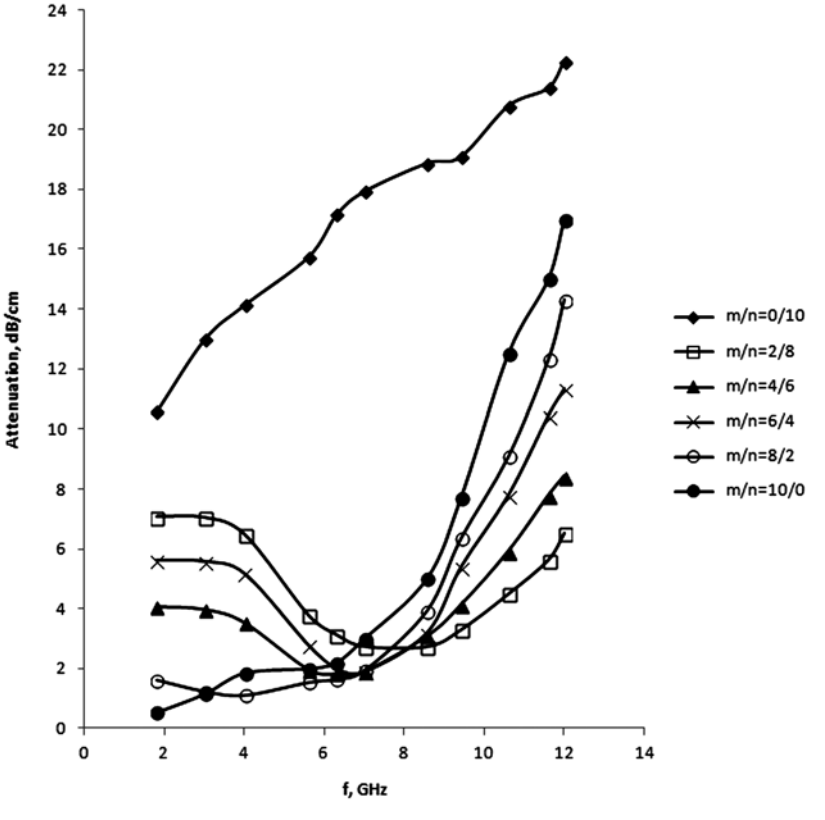

Figure 9. Effect of different ratios of graphene nanoplatelets (m) and carbon nanotubes (n) on coefficient of attenuation of NR-based nanocomposites.

content of GNP have greater reflection than those with higher content of CNT. All this shows that the reflection coefficient of the investigated composites is strongly frequency dependent.

Coefficient of attenuation

The course of the dependence of the attenuation coefficient α on frequency (Figure 9) is very interesting. One critical value is observed at 7 GHz. Up to about 7 GHz, the composite $m/n = 2/8$ ($\alpha = 7.1$ dB/cm for a frequency 4 GHz) has the greatest attenuation. In the beginning of the range, the attenuations vary widely and as the frequency comes closer to 7 GHz the differences decrease. After this frequency value, the curves are arranged in the order: $m/n = 8/2$, 6/4, 4/6, and 2/8. The attenuation coefficient increases steeply and reaches up to 14.3 dB/cm at 12 GHz. From these dependences, it is clear that attenuation is not large, varying in the range (1.63 − 14.33) dB/cm for $m/n = 8/2$, with continuous growth. For all other values, the attenuation decreases slightly to around 7 GHz and then increases in the order described. From these results, it can be concluded that up to 7 GHz the composites with higher content of CNT have higher values of the attenuation coefficient, and those with increased content of GNP – the lowest. Above 7 GHz, the trend is just the opposite.

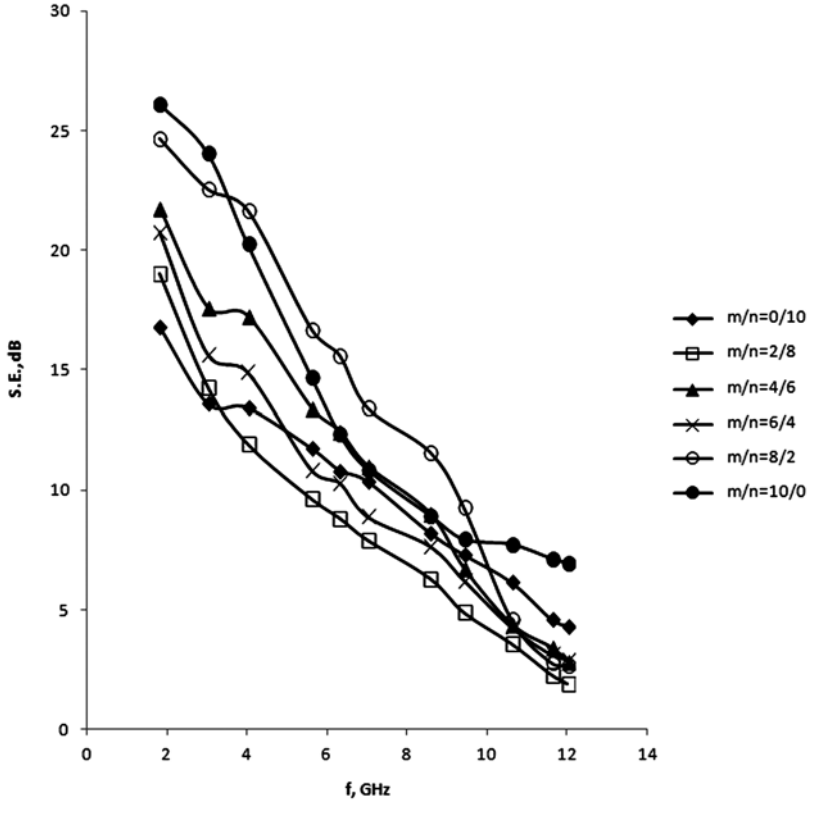

Figure 10. Effect of different ratios of graphene nanoplatelets (m) and carbon nanotubes (n) on electromagnetic interference shielding effectiveness of NR-based nanocomposites.

These results once again confirm how by using properly selected filler and filler amount the microwave and dielectric properties of the elastomer nanocomposites can be controlled.

Electromagnetic interference shielding effectiveness

The natural result of changes in the coefficients of reflection and attenuation is the dependence of the S.E. on the frequency for the same values of the parameter *m*/*n*. (Figure 10). It is seen that the composites with higher content of GNP have the highest value of electromagnetic shielding effectiveness in the investigated frequency range whereas those with increased content of CNT – the lowest.

Conclusions

The microwave and dielectric properties of nanocomposites based on natural rubber containing the combination of carbon nanotubes and graphene (CNT–GNP) have been examined in the frequency range 1–12 GHz. It has been found that by changing the ratio of mass proportions of the fillers, the variation direction of the real and imaginary part of complex permittivity (depending on the particular application) can be controlled.

As seen from the schemes of the equipment for measuring of the dielectric and microwave properties, we did not use a Network Analyzer. That presumes allowing of experimental error within the range of about 10%. The measurements were carried out repeatedly, and the results were averaged especially for these points where the tenor of dependencies was changed. For every measurement, we used at five simples of respective rubber compound. The variation of the properties for given compound was within the borders of the accuracy of used measurement method (about 10%). The used technology for preparing of the simples ensured uniform distribution of the fillers particles in the elastomeric matrix that explain insignificant difference in the obtained results.

Our previous investigations indicated that the chemical nature of elastomeric matrix had significant influence on the interaction between the electromagnetic waves and the composite.[19,20] The elastomers with highly polar functional groups or bonds – acrylonitrile–butadiene rubber (NBR), chloroprene rubber (CR), determine better microwave properties of the absorbers, and the polarity is more important factor than the elastomer's ability to crystallize. The improvement of absorbing properties of the absorbers based on polar crystallizing elastomers becomes mainly by the reason of the decrease in the passed through absorber power (the interaction between the electromagnetic waves and absorbing composite is more active). Crystallization ability of the elastomer exerts noticeable positive influence on the absorbing properties if the elastomer has no polar nature – comparison between styrene–butadiene rubber (SBR), natural rubber (NR), and butyl rubber (BR).

The reflection coefficient of the investigated composites is strongly dependent on the frequency and the ratio between the fillers. The results for attenuation coefficient show that up to 7 GHz the composites with higher content of CNT have higher values, and those with increased content of GNP – the lowest. The trend is just the opposite when the frequency is above 7 GHz.

The frequency and filler amount ratio have an effect upon the reflection and attenuation coefficients as well as upon the electromagnetic shielding effectiveness. The attenuation is not great enough to compensate the increasing reflectance. Therefore, as a whole, the shielding effectiveness decreases gradually with the increasing frequency, especially at frequencies up to 7 GHz.

The results achieved reveal how by using the selected combinations between CNT and GNP the microwave and dielectric properties of natural rubber nanocomposites can be tailored depending on the need for real practical application. The developed nanocomposites could have commercial potential for lightweight shielding materials for electromagnetic radiation.

Acknowledgments

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