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Polyethylene thermoplastic polymer composites with neodymium filler

Petru V. NOTINGHER, Cristina STANCU

University POLITEHNICA of Bucharest, 313 Splaiul Independentei, Bucharest, ROMANIA

Denis PANAITESCU

ICECHIM, 202 Splaiul Independentei, Bucharest, ROMANIA

Virgil MARINESCU

INCDIE ICPE CA, 313 Splaiul Unirii, Bucharest, ROMANIA

ABSTRACT - In this paper an experimental study regarding the structure and electrical properties of low density polyethylene composites filled with neodymium particles is presented. The analysis of the samples structure shows that the neodymium particles appear as clusters and the distance between them decreases if the filler content increases. The DC measurements show that the electrical conductivity values increase with the temperature and filler content. Using the absorption currents, dielectric response functions of composites $f(t)$ are determined and the complex permittivity values are calculated and compared with experimental values. Good correlation between calculated values and experimental ones was found for frequencies lower than 100 Hz. Variations of the complex permittivity components with the neodymium content, temperature and frequency are also presented.

Keywords – Polymer composites, dielectric response function, electrical conductivity, complex permittivity.

1. INTRODUCTION

The fast development of electronics and electrotechnics involves achieving of multifunctional materials, with special (electrical, magnetic, thermal and mechanical) properties, easy processability and low costs [1]. Among the new class of materials which meet these requirements, polymeric composite materials are remarked. These materials combine the outstanding properties and easy processability of polymers with the special properties (conductive, magnetics, thermal etc.) of fillers. Polymeric composites with high electric conductivity, magnetic permeability, remanent magnetization and coercive field were manufactured. Their operating temperatures are higher to those of polymers.

For their manufacture, polyethylene, polyvinylchloride, polycarbonate, epoxy resin, acrylonitrile-butadiene-styrene, polystyrene, nylon 6,6 etc can be used as matrix and aluminum [2] carbon and graphite [3,4], copper [5], steel [6], aluminum nitrides [7], nickel or silver particles [9], barium titanate [10] etc – as filler. Such composite materials were used for encapsulation, thin film coating, packaging for electronic circuits, electromagnetic and radio-frequency interference (EMI/RFI) shielding for electronic devices and electrostatic dissipation ESD [4,9,11].

Properties of composites materials depend, on one hand, on the matrix and filler characteristics, and on the other hand, on

the technological processes parameters and environmental action [12-14]. The influence of nature, concentration and filler dimensions on the electrical properties of polymer composites with conductive [15-18] or magnetic [14], [19-20] fillers was studied in several papers. It is shown that there is a critical concentration for that the percolation process occurs, leading to important variations of the electrical conductivity, permittivity and dielectric losses [14], [21-22].

In previous papers the authors present an experimental study regarding the electrical conductivity (measured both in dc and ac) and loss factor of composites from polyethylene with magnetic filler [14, 23].

Results of an experimental study performed on low density polyethylene samples with magnetic filler (neodymium particles of micron size) regarding their behavior in constant and time variable electric fields is presented in this paper.

Dielectric response function, variations of electric conductivity and complex permittivity components with filler content, temperature and frequency of the electric field are determined. The possibility to use these materials in electrical engineering field (electromagnetic shielding etc.) is then analyzed.

2. DIELECTRIC RESPONSE FUNCTION

If a step-like electric field E_0 is applied to a dielectric at any time t_0 and assuming that E_0 remains constant for $t \geq t_0$, the polarization $P(t)$ of the dielectric is given by the equation:

$$P(t) = \varepsilon_0 \chi(t) 1(t) E_0, \quad (1)$$

where $\chi(t)$ is the dielectric susceptibility, ε_0 is the free space permittivity and $1(t)$ indicates the unit step for the electric field E_0 . $P(t)$ and $\chi(t)$ represent step response functions in time domain [24-26].

For a time dependent excitation $E(t)$, the time dependent polarization $P(t)$ is found (using the Duhamel's Integral) by the equation:

$$P(t) = \varepsilon_0 \chi_\infty E(t) + \varepsilon_0 \int_{-\infty}^t f(t-\tau) E(\tau) d\tau, \quad (2)$$

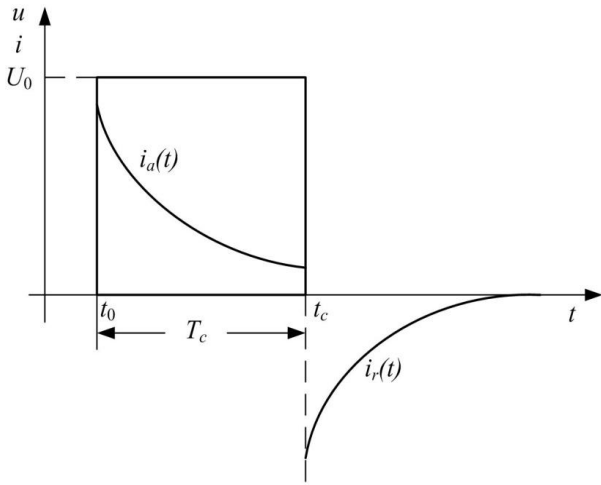


Fig. 1. Absorption (i_a) and resorption (i_r) currents.

where $f(t)$ is the so-called dielectric response function in time domain.

If a DC voltage U_0 is suddenly applied to the test object, a current $i_a(t)$ occurs through the test object (Fig. 1):

$$i_a(t) = C_0 U_0 \left[\frac{\sigma}{\varepsilon_0} + \varepsilon_\infty \delta(t) + f(t) \right] \quad (3)$$

where C_0 is the geometric capacitance of the test object and $\delta(t)$ is the delta function from the suddenly applied step voltage at $t = t_0$ [25].

If the test object is short-circuited at $t = t_c$, the resorption current $i_r(t)$ can be measured (Fig. 1). The sudden reduction of the voltage U_0 is regarded as a negative voltage step at time $t = t_c$ and, neglecting the second term in equation (3) (which is again a very short current pulse), we obtain for $t \geq t_0 + T_c$ [24]:

$$i_r(t) = -C_0 U_0 [f(t) - f(t + T_c)]. \quad (4)$$

As the resorption current is proportional to the dielectric response function $f(t)$, the equation (4) is the second basis for the measurement of the $f(t)$ dielectric. Supposing that T_c is high enough so that $f(t + T_c) \approx 0$, it results from (4):

$$f(t) \approx -\frac{i_r(t)}{C_0 U_0}. \quad (5)$$

In some papers [27-29], function $f(t)$ is considered as:

$$f(t) = \frac{A}{\left(\frac{t}{t_0}\right)^m + \left(\frac{t}{t_0}\right)^n} \quad (6)$$

where A , m and n are material constants.

Considering that an AC voltage of pulsation ω is applied to a capacitor, that the polarization processes are instantaneous, and $\underline{F}(\omega)$ is the Fourier transform of the dielectric response

function $f(t)$, respectively the complex susceptibility $\underline{\chi}(\omega)$, it results:

$$\underline{F}(\omega) = \underline{\chi}(\omega) = \int_0^{\infty} f(t) \exp(-i\omega t) dt = \chi'(\omega) - i\chi''(\omega), \quad (7)$$

$$\chi'(\omega) = \varepsilon_r'(\omega) - 1 = \int_0^{+\infty} f(t) \cos(\omega t) dt, \quad (8)$$

$$\chi''(\omega) = \varepsilon_r''(\omega) = \int_0^{+\infty} f(t) \sin(\omega t) dt, \quad (9)$$

where $\chi'(\omega)$ and $\chi''(\omega)$ represent the real and imaginary parts of complex susceptibility $\underline{\chi}(\omega)$, $\varepsilon_r'(\omega)$ and $\varepsilon_r''(\omega)$ - real, respectively imaginary part of complex permittivity $\varepsilon_r^*(\omega)$ ($\varepsilon_r^*(\omega) = \varepsilon_r'(\omega) + j\varepsilon_r''(\omega)$).

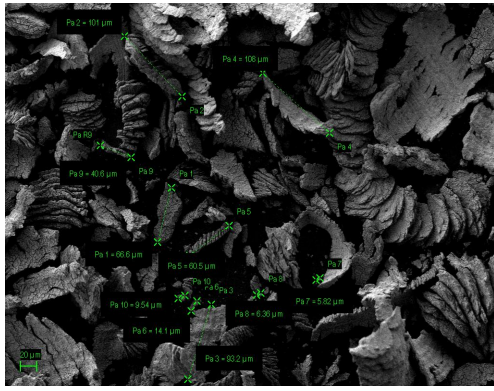
3. EXPERIMENTS

Experiments were performed on flat samples of composites prepared from low density polyethylene LDPE with a melt flow index (190 °C, 2.16 kg) of 0.3 g/10 min, a density of 0.920 g/cm³ at 23 °C and an electrical conductivity of 5·10⁻¹⁷ S/m. As filler, particles of Neodymium with the length of 5.82...106 μm and the width of 12.8 -45 μm (Fig. 2), density of 7 kg/dm³ and electrical conductivity of 1.56·10⁶ S/m were used. Maleic anhydride-grafted polyethylene (MA-PE), with a density of 0.925 g/cm³ and a melting point of 105 °C, was used as compatibility agent. A 50 cm³ mixing chamber of a Brabender Plasti-Corder LabStation was used for mixing and homogenizing Neodymium powder with the polymer matrix and the compatibility agent (5 wt % MA-PE). Metal powders (concentration of 5, 10 and 15 wt %) were slowly added (~ 2 minutes) to the mixture of PE and MA-PE and mixed at 160 °C, for 8 min (the speed of the rotors of 100 rpm).

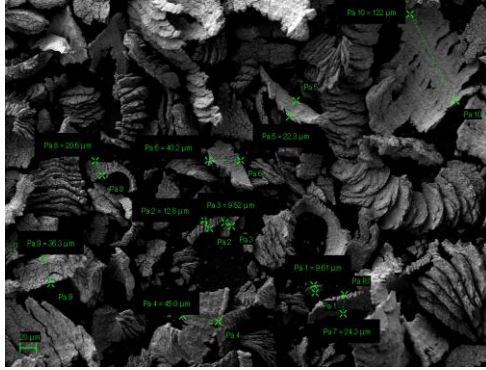
Square plates 100x100x0.5 mm³ have been realized by hot pressing at 170 °C for 5 min., with a force of 50 kN. After pressing, the samples were cooled to room temperature under a pressure of 5 bars.

The structure of samples and the dispersion of neodymium particles in polyethylene matrix were analyzed using the optical microscopy (with a NIKON TI-e microscope) and Scanning Electron Microscopy SEM (with a crossbeam (FESEM-FIB)-model AURIGA, Carl Zeiss SMT and detector type EVERHART-THORNLEY).

The absorption and resorption currents were measured on square plates (of side $a = 100$ mm) with a Keithley 6517 electrometer. The applied voltage U_0 was from 100 to 1000 V and the temperatures between 25 ÷ 70 °C. The components of the complex permittivity were measured on square plates (of side $b = 40$ mm) with a Novocontrol Impedance Analyzer. The applied voltage was 1 V and the frequency between 1 mHz and 1 MHz [24]. For each type of material (E0, E1, E2 and E3, see Table 1), 6 samples (3 with $a = 100$ mm and 3 with $b = 40$ mm) were manufactured. The measurements (currents, permittivity components) were performed 3 times on each sample and the average values were calculated.



a)



b)

Fig. 2. Neodymium particles length (a) and width (b) (Optical microscopy, 200 X).

Table 1. Samples

Sample	Nd mass concentration c_m (%)	Nd volumic concentration c_v (%)
E0	0	0
E1	5	0.688
E2	10	1.377
E3	15	2.064

Table 2. Dimensions and distances between clusters

Sample	Average clusters dimension (μm)	Average distance between clusters (μm)
E0	0	0
E1	95.92	117.81
E2	100.95	94.04
E3	103.6	67.5

4. RESULTS AND DISCUSSIONS

Structure, electrical characteristics (conductivity, complex permittivity components) and dielectric response functions were determined for all samples type. Their dependence on the filler content was analyzed.

4.1. Microscopic investigation

SEM analysis reveals a small (almost negligible) porosity in both surface and volume of the samples (P, Fig. 3). LDPE shows a lamellar structure and neodymium particles form clusters (metal “islands” [23]) of variable dimensions (Figs. 3-4). These clusters are uniform distributed in the samples and the distance d between them decreases with the increase of the filler content (Fig. 4 and Table 2). Thus, for samples E1 the distance d varies between 99.5 and 275 μm and for E3 between

47.49 and 211 μm . On the other hand, the dimensions of the clusters increase with the filler content (Fig. 4 and Table 2).

4.2. Electrical permittivity

Values of the complex permittivity components (ϵ_r' and ϵ_r'') for frequencies between 1 mHz and 1 MHz and temperatures between 30 and 80 $^\circ\text{C}$, for all type of samples (E0...E3) were determined.

4.2.1. Real part of permittivity

In Figure 5 the variations of the real part of permittivity ϵ_r' with frequency are presented. It comes out that, for all samples, ϵ_r' increases with the frequency decrease, similar variation being observed by other authors [15], [17-20], [30-31].

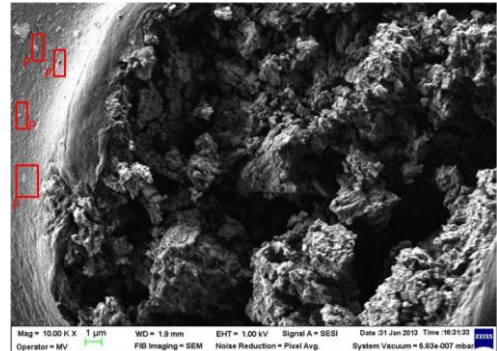
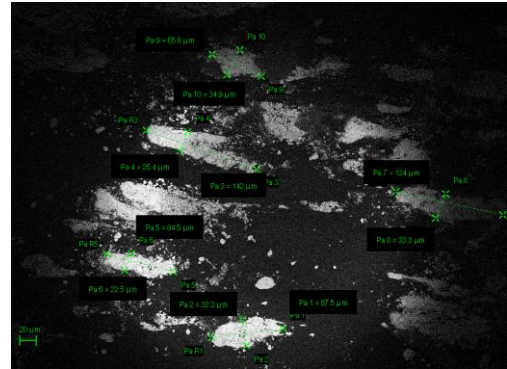
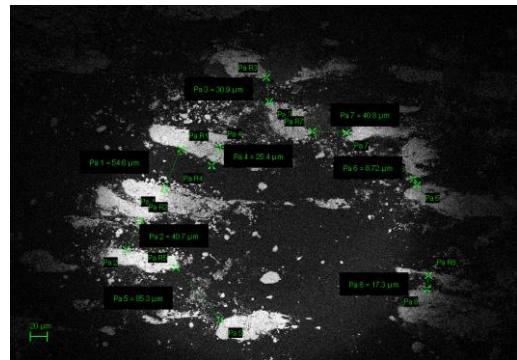


Fig. 3. Neodymium particles cluster and pores in LDPE matrix (SEM, 1000 X).



a)



b)

Fig. 4. Dimensions (a) and distances between neodymium particles clusters (b) in E3 samples (Optical Microscopy, 200X).

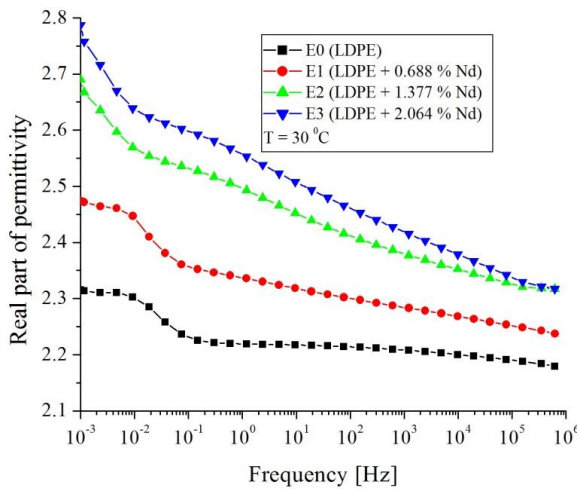


Fig. 5. Variations with frequency of the real part of complex permittivity for samples E0, E1, E2 and E3 ($T = 30\text{ }^{\circ}\text{C}$).

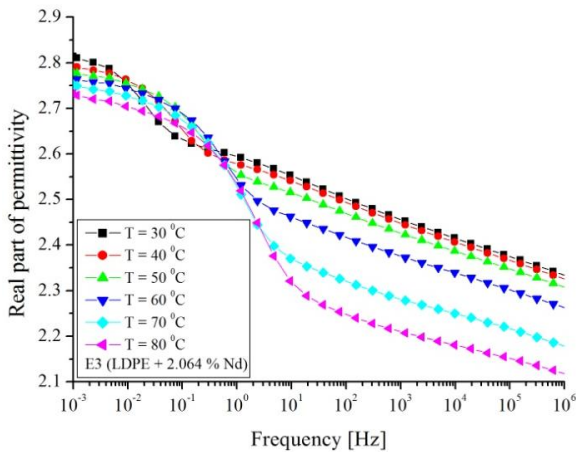


Fig. 6. Variation with frequency and temperature of the real part of complex permittivity for samples E3.

This is due, on one hand, to the space charge separation at the polyethylene/filler interfaces and to enhancement of interfacial polarization and on the other hand to the large movements (at lower frequencies) of entities with higher dipole moment and molecular weight (maleic aldehyde, longer branches in low density polyethylene, lateral hexyl groups, etc.).

Variation of ϵ_r' with frequency and temperature is presented in Figure 6. It may be seen that ϵ_r' values decrease with temperature, less for lower values of the frequency (1 mHz) and more for high frequencies (1 MHz). This is due to the increase with frequency of own energies of ions that separates at the matrix/filler interfaces and especially to polar entities which determines the orientation polarization in samples [32].

The real part of permittivity ϵ_r' increases with filler content for any frequency value (Fig. 7), in accordance with other results [12], [19-20], [33-34]). Variations of ϵ_r' with mass neodymium filler content c_m are not linear, following (with a certain approximation) Bruggeman's and Doyle's equations that are presented in [33].

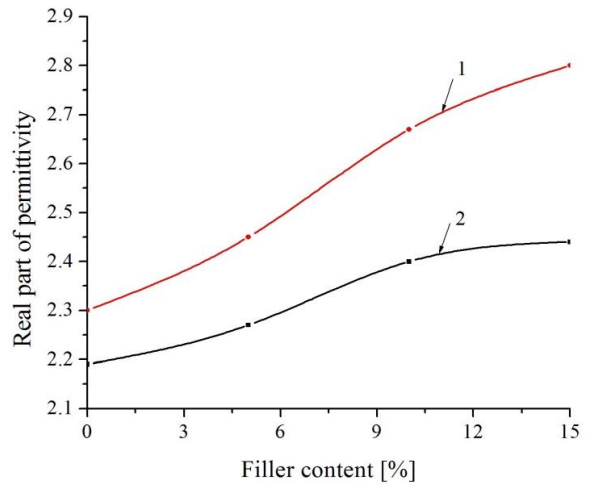


Fig. 7. Variations with mass filler content of the real part of permittivity for $f = 1\text{ mHz}$ (1) and $f = 1\text{ kHz}$ (2) ($T = 40\text{ }^{\circ}\text{C}$).

Increase of ϵ_r' with c_m is due to the increase of the polyethylene/neodymium interfaces area and to charge values that separate at interfaces. As consequence, a local increase of the electric field occurs leading to the interfacial polarization and electrical permittivity enhancement [35]. On the other hand, the increase of the filler content (over 10 %) determines a bonding of more chain ends with neodymium particles. Therefore, the mobility of polar chains reduces, which determines a small decrease of the permittivity component that correspond to orientation polarization.

4.2.2. Imaginary part of permittivity

Variations of the imaginary part of the complex permittivity ϵ_r'' with frequency of the electric field for samples E0...E3 at the temperature $T = 30\text{ }^{\circ}\text{C}$ are presented in Figure 8. It may be seen that $\epsilon_r''(f)$ curves have a first maximum (peak) at lower frequencies, respectively in the range (0.02...0.4) Hz. This maximum is probably due to α -relaxation polyethylene branches, maleic anhydride and charge carriers that form space charge (being at the origin of the interfacial polarization) [36]. Similar maximum at low frequency was obtained for polystyrene/barium ferrite composites) [20].

On the other hand, increasing the filler content, ϵ_r'' increases at its turn [37] and their maxima move to lower values of the frequency (Fig. 8). This is probably due, to the increase of the LDPE/Nd interface area and thus to space charge that separates on this interfaces and number of maleic anhydride molecules and molecular chains that are fixed on neodymium particles.

Variations of ϵ_r'' with frequency for different temperature values are presented in Figure 9 for samples E2. It may be seen that for all temperatures, $\epsilon_r''(f)$ curves have approximatively same shape but increasing the temperature both height and width grow and move to higher values of the frequency, phenomenon obtained also in [38]. This may be explained by the increase of the polar species balance with higher molecular weight in the orientation polarization process.

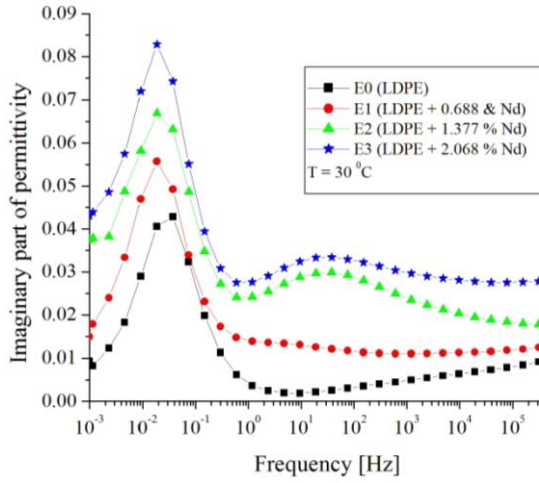


Fig. 8. Variations with frequency of imaginary part of complex permittivity for samples E0, E1, E2 and E3 ($T = 30\text{ }^{\circ}\text{C}$).

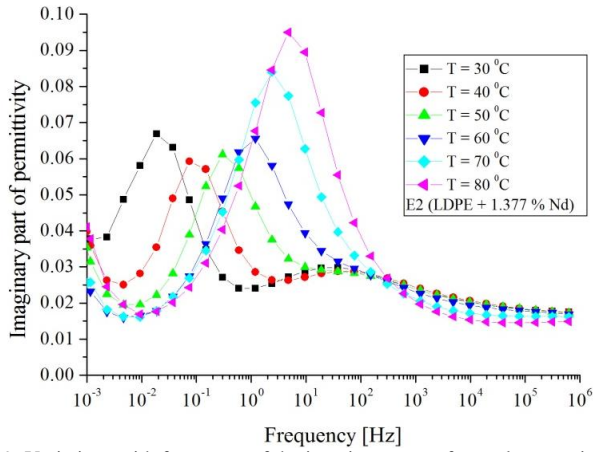


Fig. 9. Variations with frequency of the imaginary part of complex permittivity for samples E2.

4.3. Electrical conductivity

The DC conductivity values ($\sigma_{DC}(t)$) were calculated with the equation:

$$\sigma_{DC}(t) = \frac{i_a(t) - i_r(t)}{U_0} \frac{g}{S} \quad (10)$$

where $i_a(t)$ and $i_r(t)$ are the absorption, respectively resorption currents (Fig. 10), g – the sample thickness and S – the area of the electrodes' active surface of the measuring cell [4].

For all samples, the values of $\sigma_{DC}(t)$ decrease with the measurement time (Fig. 11). This is due to the decrease with time of the carrier's content (electronic and/or ionic) that corresponds to the space charge inside the samples during the manufacturing process or injection from electrodes. This charge may neutralize itself or attach on the samples defects (molecular chain ends, microcavities, polyethylene-filler interfaces etc.).

The values of the conductivity increase with the filler content. Thus, for a neodymium filler content higher than 40 %, σ_{DC} overlaps 10^4 S/m and the material has a semi-conductive behavior and the composites can be used for electromagnetic shielding [14].

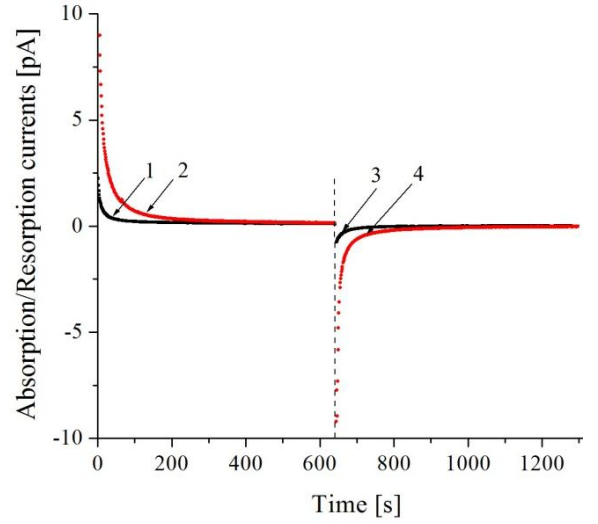


Fig. 10. Variation with time of the absorption (1, 2) and resorption currents (3, 4) in samples E0 (1, 3) and E3 (2, 4) ($T = 70\text{ }^{\circ}\text{C}$, $U_0 = 500\text{ V}$).

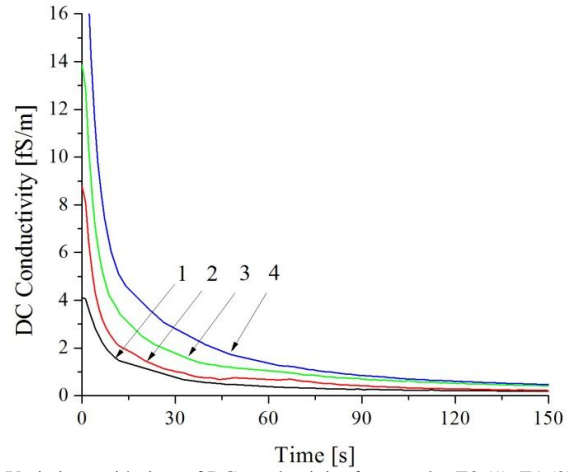


Fig. 11. Variations with time of DC conductivity for samples E0 (1), E1 (2), E2 (3) and E3 (4) ($T = 70\text{ }^{\circ}\text{C}$).

The values of the electrical conductivity of samples increase with the temperature, because of the increase of the charge carrier's mobility and eventually of their content (by releasing the charge that is fixed in less deep traps) [14].

4.4. Dielectric response function

Knowing the variations with time of resorption currents and using the equation (6) parameters A , t_0 , m and n of dielectric response function $f(t)$ for all samples type were determined. Their values depend on the samples type and temperature during measurements (Table 3).

Dielectric response function curves $f(t)$ for samples E2, at 40 and 70 °C are presented in Figure 12. It may be seen that the values of $f(t)$ increase with temperature. This is due to the increase of the own energies of dipolar particles (radicals and ends of molecular chains) and ion mobilities that form space charge inside the samples.

Using the functions $f(t)$ and the equations (8) – (9) the values of complex permittivity parts ϵ_r' and ϵ_r'' for all samples type were calculated and compared with the experimental ones (see & 4.2).

Table 3. Values of the coefficients A , m , n and t_0

Sample	Temperature (°C)	A (s ⁻¹)	m	n	t_0
E0	40	0.0000651	0.4632	0.8603	49.61
E0	50	0.82451	0.6867	0.8393	0.00006
E0	70	0.5482	0.7733	0.6233	0.00003
E1	40	0.000878	0.9650	0.1570	0.9704
E1	50	1.62551	0.8432	0.8407	0.00016
E1	70	0.0224	0.8901	0.9266	25.00
E2	40	1.74212	0.8792	0.8792	0.20
E2	70	1.01641	0.8094	0.8040	0.00028
E3	40	0.86854	0.6756	0.8738	0.00051
E3	50	0.90618	0.7380	0.8687	0.00055
E3	70	1.71882	0.9469	0.9469	0.00131

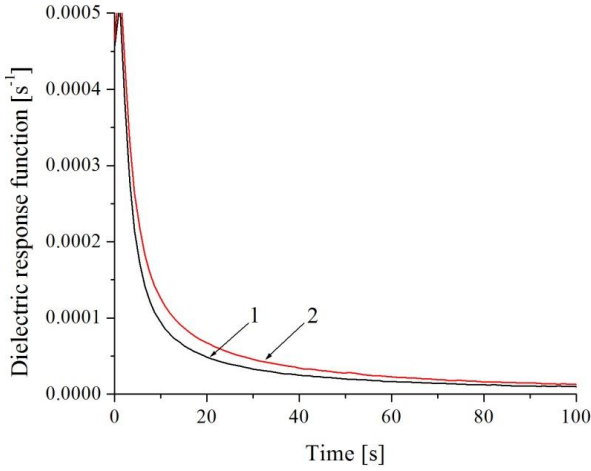


Fig. 12. Variations with time of the dielectric response functions for samples E2, at 40 °C (1) and 70 °C (2).

Variations of ϵ_r' with frequency for samples E0 and E1 at 40 °C are presented in Figures 13 and 14. It may be seen that for lower values of the frequency (below 100 Hz – for samples E0 – and below 1 Hz – for samples E1), the calculated values of ϵ_r' are not very different to those experimentally determined (under 3.5 % - for E0 and up to 1.3 % for E1). For high values of the frequency, the calculated values differ greatly to those experimentally determined, overlapping at 40 °C and 100 Hz, 60 % - for samples E1 – and 57 % - for samples E2.

In the case of imaginary part of complex permittivity ϵ_r'' , the differences between the calculated values and the experimental ones are greater (Fig. 15). Thus, for frequencies lower than 100 Hz, these differences rise to 19.5 % for samples E2, to 24 % for samples E1 and up to 50 % for samples E0. For frequencies higher than 100 Hz, the values of ϵ_r'' calculated with $f(t)$ differ by an order of magnitude to the measured ones. It results that the dielectric response function that are determined with parameters from Table 2 do not correctly characterize the behavior of the samples in electric fields with frequencies higher than 100 Hz (and especially those of high frequencies). Differences between the calculated and measured values of ϵ_r' and ϵ_r'' depend on the errors during measurements of resorption currents (below 1 pA) and to approximations made for numerical calculations of $f(t)$ (using relation (6) [27] ϵ_r' and ϵ_r'' (using the relations (8)-(9)).

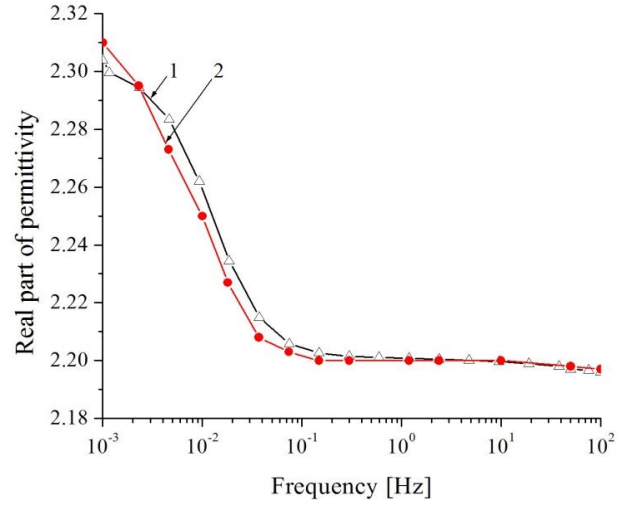


Fig. 13. Variations with frequency of the real part of complex permittivity for samples E0, measured (1) and calculated with $f(t)$ (2) ($T = 40$ °C).

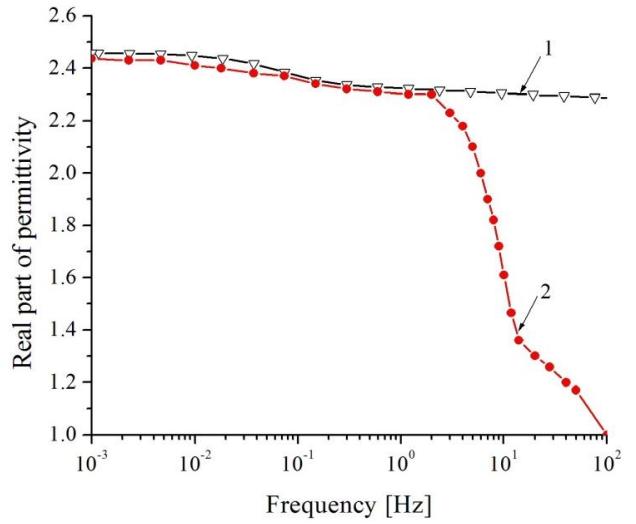


Fig. 14. Variations with frequency of the real part of complex permittivity for samples E1, measured (1) and calculated with $f(t)$ (2) ($T = 40$ °C).

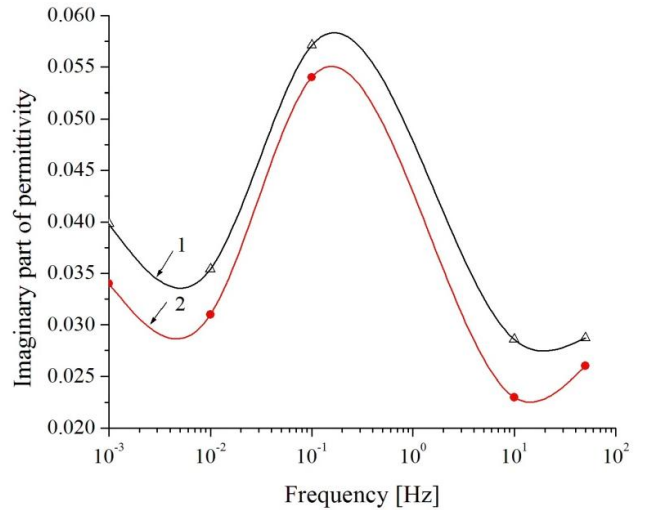


Fig. 15. Variations with frequency of the imaginary part of complex permittivity for samples E2, measured (1) and calculated with $f(t)$ (2) ($T = 40$ °C).

5. CONCLUSIONS

The presence of neodymium particles determines an inhomogeneous structure of the LDPE composites, metallic islands structure being generated.

Measurement of absorption and resorption currents allows determining the DC conductivity σ_{DC} and dielectric response function $f(t)$ of samples.

Dielectric response functions $f(t)$ determined in this paper allow to determine the complex permittivity components ϵ_r' and ϵ_r'' for frequencies lower than 100 Hz. In this case the calculated values are different to experimentally ones with less than 3.5 % for ϵ_r' and 54 % for ϵ_r'' . For frequencies higher than 1 kHz, the calculated values exceed with one magnitude order the measured ones.

The increase of temperature determines, generally, an increase of the electrical conductivity and of the imaginary part of the permittivity and a decrease of the real part of permittivity.

Neodymium filler determines important increases of the electrical conductivity values, real ϵ_r' and imaginary part ϵ_r'' of complex permittivity of samples. If the neodymium content exceeds the percolation content, the electrical conductivity rise to enough values for that these composites may be used for applications in the electromagnetic shielding domain.

An analyze of magnetic properties of polyethylene composites with neodymium filler will be presented in a future paper.

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