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E.A. LYSENKOV,[1] S.A. BILYI,[1, 2] S.D. NESIN,[2] V.V. KLEPKO [2]
[1] Petro Mohyla Black Sea National University
(10, 68-Desantnykiv Str., Mykolayiv 54003, Ukraine; e-mail: ealysenkov@ukr.net)
[2] Institute of Macromolecular Chemistry, Nat. Acad. of Sci. of Ukraine
(48, Kharkivske Road, Kyiv 02160, Ukraine)

DIELECTRIC PROPERTIES AND RELAXATION PROCESSES IN NANOCOMPOSITES BASED ON POLYLACTIC ACID AND CARBON NANOTUBES

The dielectric properties of nanocomposites based on polylactic acid with the addition of carbon nanotubes, as well as relaxation processes in them, have been studied. The dependence of the dielectric permittivity on the frequency and the filler concentration is found, the percolation threshold is determined, and the relaxation behavior of the system is analyzed using the electric modulus. The results obtained testify to the influence of the structural and interfacial effects on the dielectric characteristics of the composite, which is important for the development of new functional materials.

Keywords: polylactic acid, carbon nanotubes, dielectric permittivity, percolation, electric modulus, relaxation.

1. Introduction

In modern condensed matter physics, considerable attention is paid to the study of polymer nanocomposites that combine the properties of polymer matrices and nanofillers. Polylactic acid (PLA) is a biodegradable polymer with high polarity, which makes it a promising basis for the creation of functional nanocomposites [1]. Carbon nanotubes (CNTs), due to their unique electrical, thermal, and mechanical properties, are widely used as fillers for polymer modification [2]. The combination of PLA with CNTs allows the creation of nanocomposites with improved dielectric and relaxation characteristics, which is relevant for applications in electronics, sensorics, and power engineering [3].

The dielectric properties of such nanocomposites are determined not only by the properties of their components, but also by the interaction of the components at the micro- and nano-levels. In particular, when a certain concentration of CNTs is reached in the PLA matrix, a percolation transition is observed, which leads to a drastic increase in electrical conductivity and a change of the dielectric constant of the composite [4]. This effect is associated with the formation of a continuous conductive network of CNTs in the polymer matrix, which substantially affects the electrophysical properties of the material [5–7]. Other studies show that the dielectric permittivity can be increased, simultaneously maintaining low dielectric losses, which makes these nanocomposites suitable for applications in electronic devices and protection against electromagnetic interference [8].

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Relaxation processes in nanocomposites are associated with interfacial polarization, the movement of macromolecular segments, and the interaction of nanotubes with the polymer medium [9]. Those processes affect the dielectric losses and the frequency dependence of the dielectric permittivity, which is important for the understanding of the mechanisms of charge transfer and polarization in such systems. The study of relaxation properties makes it possible to more deeply understand the physical processes occurring in nanocomposites and optimize their properties for specific applications [10].

The features of relaxation processes in composites based on PLA and CNTs have been analyzed in detail. For example, Xu *et al.* [11] analyzed the formation of a nanofiller network, where CNTs create conductive paths in the PLA matrix, contributing to an increase of the polymer dielectric permittivity. The increase of the dielectric permittivity is associated with the polarization mechanisms, where the unique structure of CNTs interacts with the polymer matrix and affects the relaxation processes under the action of an electric field.

The improvement of the dielectric properties of those composites is explained by various polarization mechanisms. The addition of CNTs promotes interfacial polarization, when charges are accumulated at the interfaces between the conductive nanotubes and the insulating PLA matrix, thus effectively increasing the dielectric permittivity of the material [12, 13]. This interfacial polarization becomes more pronounced as the CNT concentration grows and leads to higher dielectric permittivity values. Thus, the study of the dielectric and relaxation properties of nanocomposites based on PLA and CNTs is of both fundamental and applied importance. Understanding the physical mechanisms that govern those properties will favor the development of new materials with improved characteristics for their applications in various fields of science and engineering.

2. Experimental Part

In the research, nanocomposites based on polylactic acid and carbon nanotubes are used. Polylactic acid (PLA), produced by Devil Design (Poland), was chosen as the polymer matrix. At the temperature $T = 20$ °C, PLA is a solid substance with the density $\rho = 1250$ kg/m^3. Before application, the polymer was dehydrated by heating it for four hours at 80 °C. Multilayered CNTs (JSC "Spetsmash", Ukraine) were fabricated from ethylene by chemical vapor deposition. The content of mineral impurities did not exceed 0.1%. The specific surface area was 190 m^2/g, the outer diameter was 20 nm, the length was $5\div10$ μm, and the aspect ratio was $L/d = 250 \pm 170$. When preparing materials to study, the polymer was dissolved in a 1 : 1-mixture of solvents, dimethyl sulfoxide and dimethylacetamide, and a 10% polymer solution was obtained.

Nanocomposites were prepared by the ultrasonic mixing in the polymer solution using an ultrasonic disperser UZD A-650 (Ukraine). A continuous dispersion process was carried out for 5 min at a frequency of 22 kHz; the ultrasound power was 300 W. The CNT content was varied within an interval of $(0.1\div1.5)$ wt.%. After the ultrasonic treatment, the resulting mixture was poured onto a glass surface and dried at a temperature of $80\div100$ °C until the solvents were completely removed.

The study of dielectric properties and relaxation effects was carried out using the dielectric relaxation spectroscopy method and with the help of a Z-2000 impedance meter. A specimen was placed between the cell electrodes, and the real, Z', and imaginary, Z'', parts of impedance were measured. The dielectric parameters were calculated from the impedance values. The measurements were performed at room temperature in a frequency interval of 1 Hz$\div$2 MHz. The constant gap between the electrodes was 80 μm.

3. Influence of CNTs on the Dielectric Properties of Systems Based on Polylactic Acid

At the next stage of the experiment, the influence of the carbon nanotube content in the PLA matrix on the dielectric permittivity of the composite was studied. The corresponding results are shown in Fig. 1. The figure illustrates the frequency dependences of the real part ϵ' of the dielectric permittivities of PLA and nanocomposites containing CNTs, which are plotted on a log-log scale. The plots demonstrate a behavior typical of polymer insulators subjected to relaxation processes.

In the low-frequency interval ($f < 100$ Hz), a high value of ϵ' is observed, which testifies to a substantial contribution of the polarization mechanisms, in par-

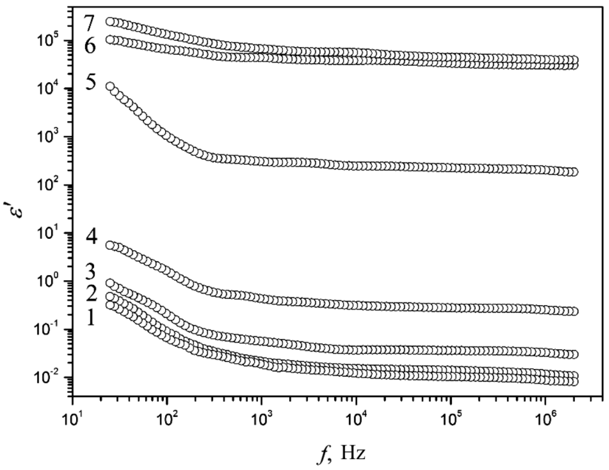

Fig. 1. Frequency dependences of dielectric permittivity for systems based on the PLA matrix and containing 0 (*1*), 0.1 (*2*), 0.25 (3), 0.5 (*4*), 0.75 (*5*), 1 (6), and 1.5 wt.% of CNTs (*7*)

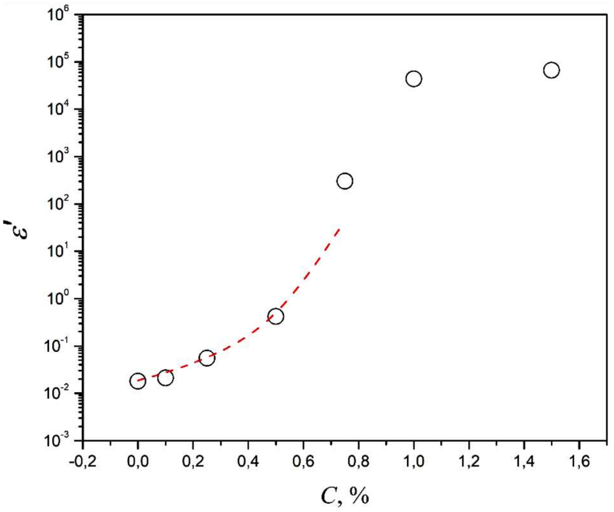

Fig. 2. Concentration dependence of dielectric permittivity for systems based on PLA and CNTs at a frequency of 1 kHz. Dashed curve corresponds to percolation model (1)

ticular, interfacial and dipole polarizations. With the increasing frequency, the value of ϵ' decreases rapidly, which is the characteristic of relaxation losses in the system. At frequences above 10^3 Hz, the decrease rate of ϵ' slows down, which points to the dominance of electronic and ionic polarizations, which are less sensitive to frequency changes in this interval. At high frequencies ($f > 10^5$ Hz), the dielectric permittivity values approach saturation, which may be associated with a restricted dipole mobility and a reduction of the influence of macroscopic polarization.

4. Percolation Behavior of Dielectric Permittivity

For a more convenient analysis, the dependence of the real part of dielectric permittivity ϵ' on the CNT content φ at a frequency of 1 kHz is shown in Fig. 2. For betten visualization and model simplification, the $\epsilon'(\varphi)$-plot is shown on a semi-log scale.

The figure demonstrates a drastic growth of the function $\epsilon'(\varphi)$ with the increasing filler content in the system. Such a behavior is explained by the formation of a branched cluster of CNT particles in the polymer matrix. According to the scaling approach of the critical percolation theory, the concentration dependence of the dielectric permittivity of the system below the percolation threshold is described by the equation [14]

$$\epsilon_{\text{eff}} = \epsilon_m \left(\frac{\varphi_c}{\varphi_c - \varphi}\right)^q, \qquad (1)$$

where ϵ_{eff} is the dielectric permittivity of the nanocomposite system, ϵ_m is the dielectric permittivity of the matrix, φ_c is the percolation threshold (a critical concentration at which the filler forms a continuous network in the polymer matrix), and q is a critical index for the dielectric permittivity (based on experimental results, it varies from 0.3 to 1.8 [15–17]).

After fitting the data in Fig. 2 using Eq. (1), it is found that $\varphi_c = 0.8\%$ and $q = 1.8$. The value $q = 1.8$ of the critical index considerably exceeds values of $0.7 \div 1.3$, typical of the classical percolation theory. However, a similar value, $q = 1.78$, was obtained in work [16] for the polyurethane-nanocarbon system. Such large q-values for the PLA–CNT system can be explained by several factors, which are related to the specificity of the composites and the interaction between the filler and the matrix.

First, the system has a fractal structure, i.e., the nanotubes form hierarchical clusters, so that the q-values can be significantly larger. Carbon nanotubes can form branched structures with tunnel contacts and change the effective dimension of the percolation process. Second, the matrix and the nanotubes themselves are highly polarizable, so an anomalous growth of ϵ' is observed. This induces a more drastic increase of permeability near φ_c, which results in high q-values. Third, the CNT distribution in the matrix is non-uniform, and the system contains aggregates of nanotubes. Therefore, the real percolation mechanism differs from idealized models. In such cases, the critical index can vary in a wide range.

It is worth noting that the percolation threshold $\varphi_c = 0.8\%$ for the dielectric permittivity in PLA–CNT polymer nanocomposites turned out larger than that for the electrical conductivity, which lies within an interval of $0.2 < \varphi_c < 0.5$ for most systems of the PLA–CNT type [4,18]. This effect is explained by various physical mechanisms that govern those properties. The electrical conductivity is determined by the formation of a continuous network of electrically conductive particles through which a current can flow. As soon as at least one connected chain of nanotubes appears, the system begins to conduct an electric current. This leads to a drastic increase in the electrical conductivity, if a low percolation threshold is reached.

The dielectric permittivity also depends on the material polarization and the concentration of conductive particles. An increase in the number of conductive nanoparticles leads to an increase in polarization due to the strengthening of interactions between the particles. However, the dielectric permittivity changes noticeably only if a substantial part of the system is already filled with conductive particles, which requires their higher concentration.

5. Effect of CNTs on Relaxation Processes in PLA-Based System

The use of electric moduli M^* to describe the relaxation behavior of polymer nanocomposites is particularly useful, because it makes it possible to analyze the polarization dynamics and the charge transfer mechanism [19]. The electric modulus M^* is related to the complex permittivity ϵ^* through the relationship

$$M^* = \frac{1}{\epsilon^*} = M' + iM'', \tag{2}$$

where M' is the real part, which reflects the elasticity of the medium before its electrical excitation, and M'' is the imaginary part, which is associated with relaxation processes. In contrast to the analysis vis ϵ^*, which emphasizes the contribution of conductivity and polarization, the quantity M^* better reveals processes with low charge mobility (for example, dipole relaxation and restricted conductivity).

In Fig. 3, the frequency dependences of the imaginary part of the electric modulus for PLA-based nanocomposites are plotted. The dependences $M''(f)$ have a maximum at low frequencies, which is associated with relaxation processes. As the filler content increases, the position of this maximum shifts toward higher frequencies. In addition, at high frequencies, the dependences $M''(f)$ demonstrate some signs of a maximum.

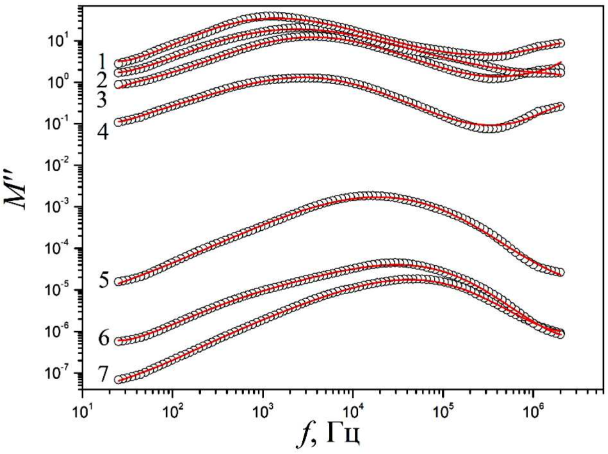

Fig. 3. Frequency dependences of the imaginary part of electric modulus for systems based on the PLA matrix and containing 0 (*1*), 0.1 (*2*), 0.25 (3), 0.5 (*4*), 0.75 (*5*), 1 (6), and 1.5 wt.% of CNTs (*7*). Empty circles correspond to experimental data, and solid curves to their approximations with the empirical Havriliak–Negami function (3)

In order to quantitatively analyze the spectrum of the imaginary part of the complex electric modulus, the following empirical function was used:

$$\begin{aligned} M''(\omega) &= \Delta M' \times \\ &\times \frac{\sin(\beta\theta)}{(1 + 2(\omega\tau)^{1-\alpha}\sin(\alpha\pi/2) + (\omega\tau)^{2(1-\alpha)})^{\beta/2}}. \end{aligned} \tag{3}$$

where

$$\theta = \mathrm{arctg}\left(\frac{(\omega\tau)^{1-\alpha}\cos(\alpha\pi/2)}{1 + (\omega\tau)^{1-\alpha}\sin(\alpha\pi/2)}\right), \tag{4}$$

$$\Delta M' = M'_u - M'_r, \tag{5}$$

τ is the characteristic time of relaxation process, which corresponds to the frequency of the maximum in the spectrum $M''(f)$, M_u and M_r are the low- and high-frequency, respectively, limits of the real part of the electric modulus, and α and β are parameters that describe the distribution of relaxation times [20]. The solid curves in Fig. 3 demonstrate the approximations of the frequency dependences of the imaginary part of the electric modulus by empirical function (3). The values of the approximation parameters are quoted in Table. From Fig. 3 and Table, one can see that as the CNT concentration increases, the maximum shifts toward high frequencies, which testifies to the mobility growth of both macromolecular segments and charge carriers.

From Table, we can also see that the relaxation processes in the PLA–CNT system differ substantially from the classical Debye relaxation, which is characterized by a single, clearly defined relaxation time. For all studied systems, β is close to 1, and $\alpha \neq 0$, which is very close to the Cole–Cole relaxation model characterized by a wide distribution of relaxation times, typical of heterogeneous systems [21].

It is also worth noting that the parameter α decreases with the increasing filler content; this testifies to an increase in the system heterogeneity so that the relaxation becomes more distributed. A similar behavior is observed for the parameter β; this points to an asymmetry increase, which occurs due to the appearance of barrier effects (for example, tunneling transitions between the nanoparticles).

In Fig. 3, one can also observe a shift of the maximum of the imaginary part of the electric modulus $M''(f)$ towards higher frequencies with the increase of the CNT content. This maximum is related to the characteristic relaxation time τ_0 through the frequency $f_{\max}$, at which the peak is observed: $f_{\max} = 1/(2\pi\tau_0)$ [22]. From Table, one can see that the growth of the CNT concentration in the PLA matrix brings about a reduction of the characteristic relaxation time because nanotubes facilitate charge transport. This means that the relaxation processes are faster, and, therefore, their characteristic frequencies move upward.

Parameter values for the empirical Havriliak–Negami approximation of the dependencies $M''(f)$ for nanocomposites based on PLA–CNTs

CNT content, %	$\Delta M'$	α	β	f_m, kHz	$\tau \times 10^{-6}$, c	lg(HHW)
0	0.08	0.12	0.99	1.3	120	2.19
0.1	0.10	0.12	0.99	2.8	93	2.44
0.25	0.10	0.10	0.99	3.2	63	2.25
0.5	0.07	0.09	0.98	2.7	45	2.55
0.75	0.08	0.09	0.97	15.9	10	2.7
1.0	0.06	0.08	0.99	31.2	5	3.03
1.5	0.06	0.08	0.98	65.1	2	3.26

The dipole relaxation also becomes faster in this system due to interfacial interactions. The nanotubes create interfacial layers, where the mobility of polymer chains changes. Polarized groups in the polymer interact with nanotubes and become more confined, but their dipole relaxation becomes faster. Therefore, this effect is an important indicator of changes in the transport properties of nanocomposites.

An important component of the dielectric studies of polymer nanocomposites is the analysis of the half-height width (HHW) of the maxima in the dependences $M''(f)$ [23, 24]. The HHW values of the relaxation maxima for all examined systems are quoted in Table, and they are considerably larger than the frequency decade. This fact testifies to a distribution of relaxation times, which is also typical of the structural relaxation processes in most flexible-chain polymers. From Fig. 3 and Table, one can see that the HHW of relaxation maxima tends to increase as the filler content in the system grows. The main reasons of the HHW increase are the system heterogeneity and interfacial interactions. There is a multiphase structure in polymer nanocomposites, which includes a polymer matrix, carbon nanotubes, and interfacial layers, where the molecular mobility is changed. As a result, particles and polymer segments have different relaxation times, which broadens the relaxation maxima.

Another reason for the broadening of the maxima is percolation, which is responsible for the charge transport features [25, 26]. If the nanoparticle concentrations are lower than the percolation threshold, charges are mainly transferred due to local polarization effects. With an increase in the concentration of nanoparticles capable of transferring charges in the system, new paths for charge transport (for example, tunneling) [27, 28] begin to appear, which creates additional relaxation mechanisms and broadens the spectrum of relaxation times.

6. Conclusions

During the study of the influence of carbon nanotubes on the dielectric properties of nanocomposites based on polylactic acid, it is found that the introduction of CNTs into a polymer matrix causes a substantial increase of the dielectric permittivity, which is associated with the interfacial and dipole polarizations. The frequency analysis shows a characteristic relaxation behavior of the permittivity, with the dominance of polarization mechanisms in the low-frequency region and the permittivity saturation at high frequencies.

The percolation threshold for the dielectric permittivity is determined at a level of 0.8%, which exceeds the typical threshold for electrical conductivity; its availability is explained by the action of various physical mechanisms giving rise to the formation of corresponding properties. The observed value of the critical index q testifies to a complicated fractal structure of the system and its high heterogeneity.

The study of relaxation processes that manifest themselves through the electric modulus showed that the increase of the CNT content makes the maximum of the imaginary part of the modulus shift toward higher frequencies, which is a result of a faster dynamics of macromolecule segments and charge carriers. The application of the empirical Havriliak–Negami function allowed us to confirm the presence of a wide distribution of relaxation times and an asymmetry growth, if the filler concentration increases. This result evidences the important role of interfacial interactions and percolation effects in the formation of the relaxation behavior of composite systems. The results obtained demonstrate the potential of PLA–CNT nanocomposites as functional materials with controlled dielectric properties, in particular, for their applications in electronics and sensor technologies.

This work was carried out in the framework of the research project "Creation of multifunctional polymer nanocomposite materials reinforced with para-aramid fibers and carbon nanotubes, in which the relationship between their structure and properties is foreseen and established", which is financed from the state budget by the Ministry of Education and Science of Ukraine (state registration number 0124U001739). The authors are also sincerely grateful to all courageous defenders of Ukraine who allow us to continue our scientific work.

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Е.А. Лисенков, С.А. Білий, С.Д. Несін, В.В. Клепко

ДІЕЛЕКТРИЧНІ ВЛАСТИВОСТІ ТА РЕЛАКСАЦІЙНІ ПРОЦЕСИ У НАНОКОМПОЗИТАХ НА ОСНОВІ ПОЛІМОЛОЧНОЇ КИСЛОТИ ТА ВУГЛЕЦЕВИХ НАНОТРУБОК

У роботі досліджено діелектричні властивості та релаксаційні процеси у нанокомпозитах на основі полімолочної кислоти з додаванням вуглецевих нанотрубок. Встановлено залежність діелектричної проникності від частоти та концентрації наповнювача, визначено поріг перколяції, а також проаналізовано релаксаційну поведінку системи за допомогою електричного модуля. Отримані результати свідчать про вплив структурних та міжфазних ефектів на діелектричні характеристики композита, що є важливим для розробки нових функціональних матеріалів.

Ключові слова: полімолочна кислота, вуглецеві нанотрубки, діелектрична проникність, перколяція, електричний модуль, релаксація.