



REGULAR ARTICLE

Percolation Behavior of Electrical Conductivity of Polylactic Acid-Based Nanocomposites

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This study investigates the electrical conductivity characteristics of polymer nanocomposites based on a polylactic acid matrix with the addition of carbon nanotubes (CNTs) were investigated using the impedance spectroscopy method. This approach allowed us to analyse the frequency dependences of electrical conductivity and identify the features of the percolation behavior of the studied systems. The results revealed that the electrical conductivity of the composites demonstrates a typical alternating current character: maintaining a constant value at low frequencies with a subsequent increase when reaching a critical frequency, which indicates a change in the charge transfer mechanism. A study of the influence of the CNT concentration on the electrical characteristics of the composites showed the presence of a clearly pronounced percolation threshold, which corresponds to the formation of a continuous network of conductive particles inside the polymer matrix. The data enabled a quantitative description of this transition using models of the critical percolation theory. It was found that even at low concentrations of CNTs (~ 0.5 %) there is a sharp increase in conductivity, which indicates the efficiency of nanotube dispersion in the polymer. Special attention was paid to modelling the frequency dependence of conductivity using Jonscher's law, which allowed us to establish a change in the nature of the interaction between charge carriers and the medium as the concentration of CNTs increases. Analysis of the parameter n demonstrated the presence of anomalous values that are not explained by classical conductivity models and indicate complex charge transfer processes associated with the formation of fractal-like topology or manifestations of anomalous diffusion. The results obtained are consistent with modern theoretical concepts of electrical conductivity in complex disordered systems. The study contributes to a deeper understanding of the mechanisms of electrical conductivity in polymer-CNT nanocomposites, which is of great importance for the further development of composite materials with controllable electrophysical properties optimized for specific functional applications.

Keywords: Polymer nanocomposites, Carbon nanotubes, Percolation, Fractal structures, Electrical conductivity.

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1. INTRODUCTION

Polymer nanocomposites with conductive nanofillers constitute a promising group of materials that combine the lightness, flexibility and processability of a polymer matrix with the functional properties of nanoscale inclusions [1]. In recent years, composites modified with carbon nanotubes (CNTs) have attracted particular attention, which, due to their high electrical conductivity, specific surface area and ability to form conductive networks, allow to significantly change the electrophysical behavior of polymer systems [2]. Such composites are widely studied not only from the standpoint of applied problems, such as the creation of sensors, electromagnetic shielding or biocompatible materials, but also as model systems for studying complex charge transfer processes in disordered environments.

From a physical point of view, polymer-CNT nanocomposites exhibit multicomponent conductivity, where classical electron or ion transport processes are supplemented by tunneling and hopping mechanisms [3]. With increasing concentration of CNTs in the pol-

mer matrix, the system undergoes a phase transition – from dielectric to conductive state – accompanied by the formation of a continuous network of conductive elements. Such a transition is described within the framework of percolation theory and is characterized by the presence of a critical concentration at which global connectivity occurs in the system [4].

Beltran et al. demonstrated that for PLA/CNT composites, the percolation threshold was theoretically modeled and determined, which is crucial for predicting conductivity within different frequency regimes (DC and RF) [5]. This theoretical foundation is supported by empirical findings from Dios et al., which reported a notably low percolation threshold (0.1 wt %) for PLA nanocomposites reinforced with CNTs, highlighting the efficiency of CNTs in forming conductive pathways within the polymer matrix [6]. Additional studies, such as those conducted by Gnanasekaran et al., emphasize the functional consequences of fine-tuning the filler concentrations, with results suggesting that an increase in CNT content can enhance the electrical conductivity [7]. The findings across these

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studies suggest that the interplay between the volume fraction of CNTs and the resultant changes in conductivity is a pivotal aspect of designing efficient composite materials. In turn, Klepko and Lysenkov showed that the percolation behavior of electrical conductivity of systems filled with CNTs is universal and does not depend on the type of polymer matrix and preparation conditions in the entire studied range of concentrations and temperatures [8].

In parallel, the frequency dependence of electrical conductivity demonstrates properties inherent in heterogeneous and fractal-like media: the behavior obeys the power laws described by the Jonscher model [9, 10], and sometimes deviations are observed that require the use of new models to predict charge transport within these systems. Moreover, interfacial interactions between CNTs and PLA have been posited as another crucial factor influencing electrical conductivity. The encapsulation of CNTs within the polymer matrix results in altered structural properties that improve electrical pathways [11]. In this context, the work by Xie et al. sheds light on the morphology and design strategies vital for reduced percolation thresholds, advocating for the strategic placement of CNTs to achieve enhanced conductivity at lower proportions [12]. These findings support the assertion that maximizing conductive network design can allow for lower filler loading without sacrificing conductivity, as previously noted in the literature.

In this work, systems based on a polylactide matrix with the addition of CNTs are considered as model nanocomposites for studying the effects of percolation and alternating current in heterogeneous solids. Particular attention is paid to the analysis of the parameters that determine the behavior of electrical conductivity under alternating current, and the interpretation of the obtained results within the framework of modern models of electron transport in dispersed media. The presented results have both applied significance for the development of functional materials and fundamental significance for deepening the understanding of physical regularities in systems with topological disorder and correlated transport processes

2. EXPERIMENTAL PART

2.1 Materials

Poly(lactic acid (PLA), manufactured by Devil Design (Poland), was selected as the polymer matrix. At $T = 20^\circ\text{C}$, PLA is a solid with a density of $\rho = 1250\text{ kg/m}^3$. Before use, the polymer was dehydrated by heating for four hours at 80°C .

Multi-walled CNTs manufactured by "Spetsmash", LTD. (Ukraine) were made of ethylene by chemical vapor deposition. The content of mineral impurities was no more than 0.1%. According to the manufacturer, the specific surface area is $190\text{ m}^2/\text{g}$, the outer diameter is 15 nm , the length is $(5\div 10)\text{ }\mu\text{m}$, the aspect ratio $L/d \approx 250 \pm 170$, and the density of CNTs is $\rho = 2045\text{ kg/m}^3$.

2.2 Preparation of Nanocomposites

To prepare the materials for the study, the polymer was dissolved in a mixture of solvents dimethyl sulfoxide and dimethylacetamide (1:1). In this case, a 10 % solution of the polymer was prepared. Nanocomposites were prepared by ultrasonic mixing in the polymer solution using an ultrasonic disperser UZD A-650 (Ukraine). Dispersion continued continuously for 5 min at a frequency of 22 kHz, the ultrasound power was 300 W. The CNT content was varied within $(0.1 \div 1.5)\text{ wt. \%}$. (hereinafter %). After ultrasonic treatment, the resulting mixture was poured onto a glass surface and dried until the solvents were completely removed at a temperature of $80\text{--}100^\circ\text{C}$.

2.3 Experimental Methods

The electrical conductivity was studied using the impedance spectroscopy method, implemented on the basis of the E7-20 impedance meter. The sample was placed between the electrodes of the cell, while its real (Z') and imaginary (Z'') parts of the impedance were subsequently measured. According to the obtained complex impedance dependences, the electrical conductivity at alternating current was subsequently determined. Measurements were conducted at room temperature in the frequency range of $25\text{ Hz} - 2\text{ MHz}$ at room temperature.

3. RESULTS AND DISCUSSION

Impedance spectroscopy is a powerful method for studying the electrical properties of polymer nanocomposites containing carbon nanotubes. It allows analyzing the conductivity, dielectric characteristics, and charge transfer mechanisms in a wide frequency range. Due to the high sensitivity of the method, it is possible to detect the influence of the concentration and dispersion of nanotubes on the electrical behavior of the material, which is critically important for the development of new functional composites.

Fig. 1 shows the frequency dependences of the real part of the electrical conductivity in logarithmic coordinates for systems based on PLA and CNT. It can be seen that the electrical conductivity remains almost constant at low frequencies, and then begins to increase when a certain threshold is reached. In the high-frequency region, a sharp increase in conductivity is observed, which may be associated with polarization processes, quantum effects, or a transition to another charge transfer mode. This behavior is typical for polymer nanocomposites, where at low frequencies the effects of electrode blocking dominate, and at high frequencies the tunneling and hopping mechanisms of charge transfer dominate.

Fig. 1 shows that the amount of introduced filler significantly affects the electrophysical properties of materials. As a result of the research, it was found that at a frequency of 1 kHz, the electrical conductivity of the PLA matrix is $5.4 \times 10^{-12}\text{ S/cm}$. It should be noted that when nanotubes are introduced into the polymer matrix, the electrical conductivity of polymer nanocomposites increases. The introduction of CNTs in an

amount of 1.5 % leads to a significant increase in conductivity to $\sigma = 3.2 \times 10^{-4}$ S/cm. Such a rapid increase in electrical conductivity is a consequence of percolation processes that occur in the system when CNTs are introduced.

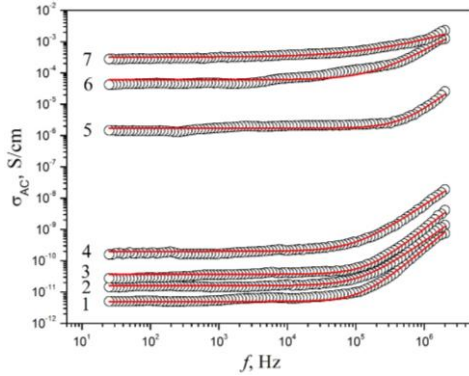


Fig. 1 – Frequency dependences of electrical conductivity at alternating current for systems based on polylactic acid matrix (1), which contains: 0.1 % (2); 0.25 % (3); 0.5 % (4); 0.75 % (5); 1 % (6); 1.5 % (7) CNT. Solid line – Jonscher model (Eq. (1))

At low frequencies, charge carriers travel a long distance inside the composite before the direction of the electric field changes. In this case, the average distance traveled by charge carriers at frequencies below the critical frequency is greater than the correlation length (the size of the largest finite-size cluster). At high frequencies, electrical conductivity is realized by hopping charge carriers between localized states, so an almost linear increase is observed on the $\sigma(\omega)$ graph [13]. The alternating current conductivity in disordered solids $\sigma(\omega)$ exhibits universal behavior and is described by the Jonscher power law [14, 15]:

$$\sigma(\omega) = \sigma_{DC} + \sigma_{AC} = \sigma_{DC} + A\omega^n, \quad (1)$$

where σ_{DC} is the direct current conductivity (frequency-independent behavior), ω is the cyclic frequency of the applied electric field ($\omega = 2\pi f$), A is a coefficient, and n is a power exponent. The exponent n characterizes the degree of interaction between the charge carriers and the matrix medium.

The obtained frequency dependences of the electrical conductivity of the PLA-CNT system (Fig. 1) were modeled within the framework of Eq. (1). The calculated parameters of Eq. 1 are given in Table 1.

Table 1 – Parameters of the Jonscher model (Eq. 1)

CNT content, %	σ_{DC} , S/cm	A , S/cm	n
0	$7.5 \cdot 10^{-12}$	$2.7 \cdot 10^{-21}$	1.82
0.10	$1.0 \cdot 10^{-11}$	$1.0 \cdot 10^{-20}$	1.79
0.25	$3.0 \cdot 10^{-11}$	$3.2 \cdot 10^{-20}$	1.72
0.50	$2.0 \cdot 10^{-10}$	$4.1 \cdot 10^{-19}$	1.55
0.75	$1.3 \cdot 10^{-6}$	$3.2 \cdot 10^{-16}$	1.39
1.00	$4.6 \cdot 10^{-5}$	$2.0 \cdot 10^{-11}$	1.20
1.50	$2.7 \cdot 10^{-4}$	$5.5 \cdot 10^{-8}$	0.70

Fig. 2 shows the dependence of the parameter n on the CNT content in the system.

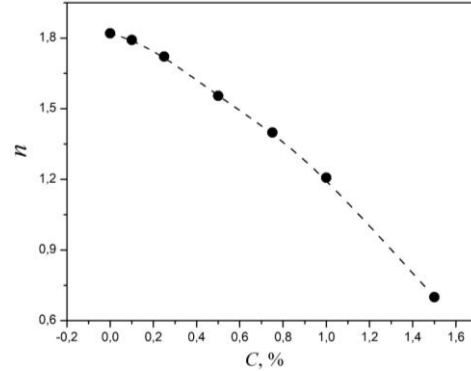


Fig. 2 – Concentration dependence of the interaction parameter for PLA-CNT systems

As illustrated in Table 1 and Fig. 2, the interfacial interaction parameter n decreases exponentially with increasing filler content in the system. This indicates a decrease in the degree of interaction between charge carriers and the matrix, which is a consequence of significant aggregation of CNT particles into denser aggregates, the area of interaction of which with the polymer is much smaller. The main mechanism of conductivity is associated with the dipole motion of polymer chains and space charges that are localized at the phase boundary between the matrix and conductive clusters of CNTs [16].

Notably, the value of the parameter n ranges from 0.7 to 1.82. However, Jonscher's power law assumes that the exponent of n lies in the range from 0 to 1. However, there were experimental cases when $n > 1$. These anomalies indicate complex mechanisms of electrical conductivity that differ from the usual jump or relaxation models. For example, Ravindran et al. investigated epoxy nanocomposites reinforced with different types of graphene nanoplatelets [17]. In this study, it was shown that the AC conductivity of these nanocomposites increases with the size and surface area of the nanoplatelets. They found that at higher frequencies, the AC conductivity is dominated by mechanisms such as interlayer hopping and tunneling, potentially leading to $n > 1$.

The most typical explanation for the situation when n exceeds 1 is the effects of interfacial polarization (Maxwell-Wagner-type phenomena) [18] and the presence of fractal-like conductive networks, which are most characteristic of systems containing CNTs [19, 20]. This phenomenon arises because in fractal or disordered systems, charge carriers do not follow a straight path, but rather move in a complex topology of voids, clusters and bottlenecks. This can be explained within the framework of an approach that combines generalized conductivity scaling and an anomalous diffusion model. In the framework of anomalous diffusion, the AC conductivity looks as follows: $\sigma(\omega) \propto \omega^a$, where a is related to the waiting time distribution and the random walk dynamics in the fractal medium. In standard percolation-based diffusion, we usually have

$0 < \alpha < 1$, which corresponds to the expected sublinear frequency dependence.

However, for the case where $n > 1$ to occur, we need $\alpha > 1$. This is not possible in normal percolation diffusion, but it can occur in cases of anomalous transport. These cases are well explained in the framework of continuous random walks in time with a superlinear frequency dependence [21], Levy flights and superdiffusion transport [22], and strongly correlated jumps in disordered systems [23].

Hence, in classical models of percolation and anomalous diffusion, the case where $n > 1$ is theoretically impossible. However, in the framework of generalized models of anomalous diffusion, it is possible to predict cases where $n > 1$ occurs, in which the mechanisms of charge movement will be associated with the formation of a fractal-like percolation network with CNTs [24].

In polymer nanocomposites, a transition from a dielectric to a conductive state is often observed when a certain concentration of an electrically conductive filler is reached. This transition is associated with percolation processes, in which a branched network of conductive particles is formed in the matrix, which provides charge transfer. The percolation threshold depends on factors such as the shape and size of the filler, its dispersion in the polymer and interaction with the matrix material [25]. Studying the electrical conductivity of such systems allows for a deeper understanding of the mechanisms of conductivity and optimizing the composition of nanocomposites for specific applications.

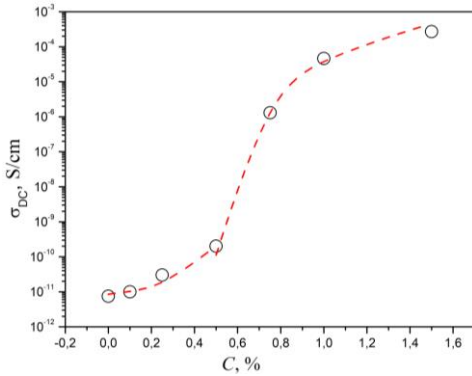


Fig. 3 – Concentration dependence of electrical conductivity at constant current for systems based on polylactide and CNTs. The dashed red line is the percolation model (Eq (2))

Fig. 3 shows the concentration dependence of the electrical conductivity at constant current ($\sigma_{DC}(\varphi)$) for the studied PLA-CNT systems. The graph demonstrates the characteristic percolation behavior typical of nanocomposite systems containing conductive fillers. Fig. 3 shows that at a low content of CNTs (~ 0.4 %), the value of σ_{DC} remains extremely low ($\leq 10^{-11}$ S/cm), which corresponds to the dielectric nature of the polymer matrix. However, with an increase in the CNT content, a sharp increase in electrical conductivity by several orders of magnitude is observed, starting from a concentration of ~ 0.5 %, which indicates the formation of a percolation network of conductive particles in the PLA matrix.

After reaching the percolation threshold (~ 0.6 %),

the electrical conductivity increases by several orders of magnitude, reaching values of the order of 10^{-3} S/cm at a CNT concentration close to 1.5 %. This behavior aligns well with the classical percolation model, according to which the electrical conductivity σ_{DC} varies according to power laws [26]:

$$\sigma_{DC} = \begin{cases} \sigma_m(\varphi - \varphi_c)^t & \text{for } \varphi > \varphi_c \\ \sigma_f(\varphi_c - \varphi)^{-s} & \text{for } \varphi < \varphi_c \end{cases} \quad (2)$$

where σ_{DC} , σ_m , σ_f are the electrical conductivities of the composite, matrix and filler, respectively, φ_c is the percolation threshold, i.e. the lowest filler content at which a continuous cluster of particles is formed; t and s are the critical conductivity indices (for a three-dimensional system, the theoretical values are $t \approx 2$ and $s \approx 0.73$ [27]. This system of equations is universal and allows us to describe the electrical conductivity of filled polymer systems in the vicinity of the percolation transition with a high degree of accuracy.

The percolation behavior of PLA-CNT nanocomposites was analyzed using scaling equations that describe the electrical conductivity as a function of the filler concentration. It was subsequently determined that the percolation threshold is 0.5 %, and the critical conductivity indices above and below this threshold are $t = 3.3$ and $s = 0.8$, respectively.

Percolation threshold. The percolation threshold of 0.5 % observed in PLA-CNT nanocomposites is relatively low, indicating the efficient formation of an electrically conductive network with minimal CNT loading. This value is comparable to or even lower than the threshold values obtained for other polymer-carbon nanotube composites. For example, for most polyether-CNT systems, the percolation threshold value is 0.45 % [28]. Notably, in this work the authors used similar CNTs, which may indicate the influence of nanotubes on the percolation processes, excluding the role of the polymer matrix in the formation of percolation clusters. For similar systems, the values of φ_c typically range from 0.1 to 1 % and depend on factors such as CNT dispersion, aspect ratio, and polymer-CNT interaction. For example, polystyrene-CNT systems are often characterized by percolation threshold values in the range of 0.2–0.6 %, while polyethylene-CNT composites can have values exceeding 1% due to different interfacial interaction energetics [29].

Critical indices. The obtained value $t = 3.3$ is within the typical range observed for nanocomposites, but above the universal value of 2.0 predicted by classical percolation theory for three-dimensional systems [30]. This deviation suggests that factors such as tunneling effects and CNT aggregation play a role in the formation of the conductive network. Similar high values of t have been reported in the literature for other CNT-based composites, such as polyurethane-CNT ($t \approx 3.1$) and PVC-CNT ($t \approx 3.3$), indicating non-ideal percolation mechanisms [31]. The value $s = 0.8$ deviates from the classical value $s \approx 0.73$ for three-dimensional percolation systems, although its value is quite close to the theoretical one [30]. The slight deviation can be explained by the strong CNT-CNT interaction, as well as

possible saturation effects during the formation of conductive paths. Similar values of s (from 0.36 to 3.26) were obtained for other nanofilled polymer composites and depended on the quality of dispersion and the structure of the filler particle network [32, 33].

Thus, the analysis confirms that the electrical conductivity behavior of PLA-CNT nanocomposites is well described by percolation and anomalous diffusion models, and the parameters obtained from the Jonscher model allow not only to quantitatively assess the changes in the system, but also to understand the internal structure of the conductive paths more deeply. The obtained results create a theoretical basis for further prediction of the properties of polymer nanocomposites with given electrophysical characteristics.

4. CONCLUSIONS

In the course of studying the electrophysical properties of nanocomposites based on polylactic acid and carbon nanotubes using impedance spectroscopy, it was found that the introduction of even a small amount of filler significantly affects the behavior of the system in a wide frequency range. The frequency dependence of the electrical conductivity revealed the classical features of polymer nanocomposites, including the presence of electrode blocking at low frequencies and hopping-tunneling charge transfer mechanisms at high frequencies. A sharp increase in electrical conductivity with the concentration of CNTs, starting from a critical concentration of $\sim 0.5\%$, indicates the onset of a percolation transition with the formation of a continuous network of conductive particles. Modeling the behavior of electrical conductivity based on the Jonscher power law confirmed that the charge transfer mechanism changes with increasing CNT concentration. In particular, the power-law decrease index n decreases exponentially, which is a consequence of a change in

the nature of the interaction between charge carriers and the matrix due to nanotube aggregation. The detected values of $n > 1$ in some cases indicate the existence of anomalous transport mechanisms, such as interfacial polarization, fractal structure of conductive paths, superdiffusion processes, and complex charge transfer topologies that go beyond classical percolation models.

Analysis of the concentration dependence of the electrical conductivity at constant current confirmed that the transition from the dielectric to the conductive state occurs when the percolation threshold is reached, which for the PLA-CNT system is 0.5% . The use of the scaling approach allowed us to determine the critical indices $t = 3.3$ and $s = -0.8$, which indicates a significant influence of CNT aggregation, tunneling effect and structural disorder on the formation of conductive clusters. The detected percolation threshold is low compared to similar polymer systems, which indicates a high efficiency of charge transfer even at low CNT concentrations.

Therefore, the findings are not only of fundamental significance for understanding the mechanisms of electrical conductivity in polymer-CNT nanocomposites, but also of practical value for creating materials with controlled conductive properties for use in electronics, sensors, energy and biomedicine.

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Перколяційна поведінка електропровідності нанокомпозитів на основі полімолочної кислоти

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У даній роботі досліджено електропровідні властивості полімерних нанокомпозитів на основі матриці полімолочної кислоти з додаванням вуглецевих нанотрубок (ВНТ) за допомогою методу імпедансної спектроскопії. Такий підхід дозволив проаналізувати частотні залежності електропровідності та виявити особливості перколяційної поведінки досліджуваних систем. Встановлено, що електропровідність композитів демонструє типовий характер змінного струму: збереження постійного значення на низьких частотах з подальшим зростанням при досягненні критичної частоти, що свідчить про зміну механізму переносу заряду. Дослідження впливу концентрації ВНТ на електричні характеристики композитів показало наявність чітко вираженого порогу перколяції, який відповідає формуванню безперервної сітки з провідних частинок усередині полімерної матриці. Отримані дані дозволили кількісно описати цей перехід за допомогою моделей теорії критичної перколяційної. Було встановлено, що навіть при малих концентраціях ВНТ (~ 0,5 %) відбувається різке зростання провідності, що свідчить про ефективність диспергування нанотрубок у полімері. Особливу увагу приділено моделюванню частотної залежності провідності з використанням закону Йоншера, що дозволило встановити зміну характеру взаємодії між носіями заряду та середовищем у процесі зростання концентрації ВНТ. Аналіз параметра n продемонстрував наявність аномальних значень, що не пояснюються класичними моделями провідності, і вказують на складні процеси переносу заряду, пов'язані з утворенням фракталоподібної топології або проявами аномальної дифузії. Отримані результати узгоджуються з сучасними теоретичними уявленнями про електропровідність у складних неупорядкованих системах. Проведене дослідження сприяє глибшому розумінню механізмів електропровідності в нанокомпозитах типу полімер-ВНТ, що має велике значення для подальшої розробки композитних матеріалів з регульованими електрофізичними властивостями, оптимізованих під конкретні функціональні застосування.

Ключові слова: Полімерні нанокомпозити, Вуглецеві нанотрубки, Перколяція, Фрактальні структури, Електропровідність.