



REGULAR ARTICLE

Features of Imparting Antistatic Properties to Epoxy Systems Using Carbon Nanotubes

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(Received 21 December 2025; revised manuscript received 20 February 2026; published online 25 February 2026)

The study investigates the development of antistatic properties in an epoxy system through the incorporation of carbon nanotubes (CNTs). The influence of CNTs of various types and manufacturers was examined. The minimum content of multi-walled and single-walled CNTs required to achieve an electrical resistivity of $10^6 \Omega \text{ cm}$ was determined. A comparative analysis of multi-walled CNTs from Cabot Corporation ("Cabot" product), Graphistrength ("Arkema" product), and single-walled CNTs from CHASM Advanced Materials (Signis product, model CG300) revealed that single-walled Signis CNTs provide the required conductivity at concentrations above 0.02 wt. %, ensuring antistatic properties on the specimen surface. The effect of CNT pre-treatment technologies on the efficiency of electrical resistivity reduction in the final polymer material was analyzed. It was shown that annealing and acid treatment have only a minor effect on achieving the desired characteristics of the polymerized product. It was established that the target electrical resistivity can also be reached through mechanical deagglomeration, although ultrasonic processing remains the most effective method. Ultrasonic dispersion is feasible only at a controllably low viscosity of the system, which becomes challenging due to viscosity increase with higher CNT content, especially for single-walled nanotubes. Moreover, prolonged ultrasonic exposure imposes limitations on its practical use. Utilizing viscosity as a factor that promotes CNT deagglomeration through shear-induced mechanical mixing of the epoxy resin enables efficient and relatively rapid dispersion at a minimal content of Signis and Cabot nanotubes.

Keywords: Epoxy system, Carbon nanotubes, Antistatic properties, Mechanical mixing, Percolation threshold.

DOI: [10.21272/jnep.18\(1\).01031](https://doi.org/10.21272/jnep.18(1).01031)

PACS numbers: 81.07.De, 72.80.Tm,
72.20. - i, 82.35. - x, 72.80.Le

1. INTRODUCTION

Epoxy resins (ER) are among the most technologically important classes of thermosetting polymers, characterized by high adhesion, mechanical strength, chemical and thermal stability. Epoxies are used in composite structural materials, compounds, and self-leveling floors. Such a wide range of applications sometimes requires specific properties from the product, in particular, certain electrical conductivity or antistatic properties [1].

Aromatic diene resins based on diphenylolpropane and epichlorohydrin, which account for more than 70% of global production, are primarily used to create epoxy polymers. The polymerization/curing process of such systems occurs under the action of amine, anhydride, or latent curing agents (in particular, dicyandiamide), which ensures the formation of a rigid, spatially cross-linked network with high strength and thermal stability [1]. Epoxy thermosetting compounds usually form an amorphous (non-crystalline) three-dimensional network during polymerization. [2]

Typically, unfilled epoxy systems are dielectrics after curing and have a surface electrical resistance of $10^{11} \dots 10^{13} \text{ Ohm cm}$. To obtain lower resistance and achieve antistatic properties (10^6 Ohm cm is the value most often considered antistatic) [3], epoxy systems are

modified (filled). The determining parameter here is the percolation threshold – the critical concentration of filler at which a continuous conductive class is formed in the insulating matrix.

Traditional filler modifiers are powders and nanoparticles of metals, graphite, and carbon fiber. Carbon nanotubes (CNTs) have the most effective influence on the electrical properties of polymers. For metal fillers, the percolation threshold is 10-30 vol. %, while for CNTs it can decrease to 0.002-0.1 vol. % depending on the morphology of the system and the conditions of composite formation [2-4].

Carbon nanotubes (CNTs) have a unique length-to-diameter ratio (100...1000) [4-5, 9]. There are single-walled (SWCNT) and multi-walled (MWCNT) carbon nanotubes. Single-walled tubes consist of a single graphene layer and have a minimum diameter. Multi-walled tubes are formed by several concentric layers and are cheaper and more common. In addition to their electrical properties, their introduction into materials also has a positive effect on the mechanical properties of the resulting composites.

Many classic experimental studies show that adding only CNTs to thermoplastic or thermosetting matrices can cause a sharp transition from an insulator to a conductor (percolation effect). The exact critical concentration

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(percolation threshold) and the properties obtained directly depend on the type of CNT (SWCNT vs MWCNT), their length, the method of dispersion (melt mixing, solution casting, in-situ polymerization, high-shear, ultrasound, etc.) and the degree of agglomeration [5].

Despite the high and proven efficiency of CNTs, their widespread use is significantly limited by problems associated with the tendency of tubes, primarily single-walled ones, to form agglomerates. Due to the strong aggregation of nanotubes, achieving uniform distribution is a difficult task, especially since the use of CNT functionalization or the addition of accompanying components for better dispersion can reduce electrical conductivity [6, 7].

Tube parameters such as purity, diameter, length, etc. vary significantly between manufacturers and can radically affect the effectiveness of their application.

High-quality, although time-consuming, deagglomeration of carbon nanotubes (CNTs) in a matrix using ultrasound allows low percolation thresholds to be achieved: for SWCNTs in epoxy, this level can reach $10^{-3} \dots 10^{-2}$ wt. % [1, 5, 6, 8].

The use of mechanical deagglomeration methods can be beneficial, although it does not allow for the production of high-quality dispersions of dispersed nanotubes. There is a certain compromise between deagglomeration, preservation of tube length, and polymer destruction: increasing the processing time, cycles, and pressure can cause a loss of properties of both the tubes and the plastic filled with them, although the relationship between quality and the application of the selected technology for different types of tubes remains insufficiently studied [9].

In addition, certain technological techniques are used to achieve ultra-low concentrations for the percolation effect: artificial segregation of nanotubes on the surface of polymer granules followed by their sintering into the product [10].

Thus, despite the proven effectiveness of nanotubes on the electrostatic properties of epoxy polymers, the task of obtaining the lowest NTP content to achieve the percolation threshold remains unresolved, which also requires high-quality and rapid deagglomeration in the matrix. This determines the relevance of research aimed at studying the effect of different types of carbon nanotubes on reducing electrical resistance and finding optimal methods for their dispersion.

2. MATERIALS AND METHODS

The composition is based on CHS Epoxy 510 epoxy resin and Telalit 0492 hardener (100.0 : 27.0) and various types and manufacturers of CNT.

Epoxy 510 Bis-A (bisphenol-A) resin, unmodified – without built-in fillers or plasticizers, has a low tendency to crystallize, has a viscosity of about 12.5...16 Pa·s at +25 °C, i.e., it is medium-flow and is used for self-leveling floors [11, 12].

The influence of carbon nanotubes of various types and manufacturers, differing in morphology, specific surface area, length, etc., was investigated: nanotubes from Cabot (USA), Arkema (France), Signis (model CG300) (USA), and experimental nanotubes manufactured in Ukraine (ECNT “Sumy”, Ukraine).

Cabot carbon nanotubes, manufactured by Cabot Corporation, belong to the type of multi-walled nanotubes (MWCNT). The manufacturer does not disclose data on dimensions, indicating only that the product contains “special compounds that include mixtures of carbon with polymer” and the possibility of obtaining antistatic properties in the system by mechanical dispersion.

Arkema tubes (Graphistrength®) are multi-walled with an average diameter of 10-15 nm, a length of 1-10 μm, and 5-15 walls.

CG300 – single-walled carbon nanotubes from CHASM Advanced Materials (Signis®) with a carbon content of ≥ 95 % by mass, an average diameter of ~ 0.84 nm, and a length of ~ 1 μm.

ECNT – experimental carbon nanotubes, which are a by-product obtained at the experimental production facilities of a private machine-building enterprise. The manufacturer classifies them as multi-walled, with an average diameter of 30 nm and a length of up to 15 μm. Other parameters are unknown.

The effectiveness of mechanical deagglomeration for different types and manufacturers of nanotubes was studied in comparison with ultrasonic deagglomeration. In each case, 50 g of resin was processed. Mechanical dispersion was performed using a 30 mm diameter “cutter” attachment at a frequency of 2000 rpm and a power of 600 W. Ultrasonic processing was performed at a frequency of 20 kHz and a power of 100 W.

The hardener was added to the resin after deagglomeration: the mixture was stirred manually for 5 min and poured into a mold. The electrical resistance was measured in the resulting disc samples with a diameter of 30 mm and a thickness of 3 mm.

Electrical resistance was evaluated according to a known method and scheme [13] using a teraohm meter.

To control the degree of deagglomeration at the stage of composition preparation, electrical resistance was measured at regular intervals, and when its decrease ceased, the treatment was stopped.

Optical microscopy was used for initial control of the absence of agglomerates in the liquid composition, and scanning electron microscopy of the hardened composite was performed on a field emission scanning electron microscope (FE-SEM) manufactured by ZEISS for final confirmation.

3. RESULTS AND DISCUSSION

Dispersion using different technologies showed significant differences in the ability of tubes to deagglomerate: the results, which are not the same even for tubes of the same type but from different manufacturers, are shown in the Table 1.

Among multi-walled tubes, Cabot multi-walled tubes have been found to be the most effective in imparting antistatic properties to epoxy systems. At a concentration of 0.15 % by weight, the composite has a resistance of the order of megohms. Such values can be achieved both by using ultrasound and by mechanical deagglomeration. It should be noted that when using a milling cutter, the deagglomeration of these nanotubes is relatively fast, and in terms of energy consumption, this method is even more effective than ultrasound at

Table 1 – Measurement table

CNT	Concentration in composition (% by weight)	Resistance of epoxy composite obtained after mechanical deagglomeration, Ohm·cm	Time of mechanical deagglomeration to resistance stability, min	Resistance of epoxy composite after ultrasonic deagglomeration, Ohm·cm	Time of ultrasonic dispersion to resistance stability, min
Cabot	0.015	2×10^{10}	10	2×10^{10}	120
	0.04	1×10^{10}	15	5×10^{10}	120
	0.15	1×10^6	30	8×10^7	180
CG300	0.015	5×10^7	10	1×10^7	140
	0.04	6×10^6	15	2×10^6	200
	0.15	4×10^5	20	1×10^6	300
Arkema	0.015	7×10^{11}	60	1×10^{11}	240
	0.04	5×10^{10}	120	9×10^9	240
	0.15	1×10^7	240	3×10^6	500
ECNT	0.015	9×10^{12}	120	1×10^{12}	120
	0.04	8×10^{12}	200	3×10^{11}	200
	0.15	5×10^{10}	300	1×10^{10}	300

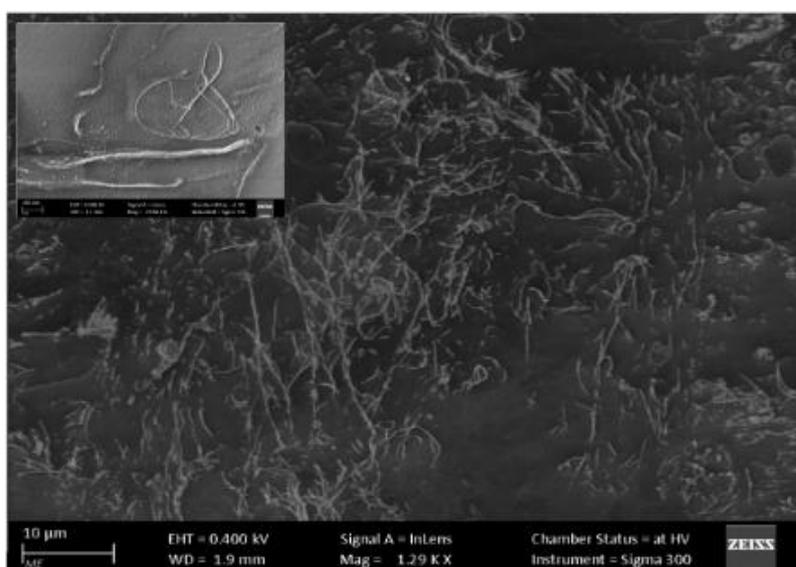


Fig. 1 – SEM image of the surface of a cured epoxy composite with 0.04 % by weight CG300 – a single single-walled tube and a cluster of several tubes after mechanical deagglomeration

a concentration of 0.015 %, if we disregard the incomplete unattainability of the resistance values obtained using ultrasonic vibrations. On the other hand, increasing the content of this type of multi-walled nanotubes by an order of magnitude makes ultrasound less effective than the mechanical method. A noticeable increase in the viscosity of the composition (not measured in this work), which complicates the work of ultrasound, contributes to the effective dispersion of agglomerates due to the high shear stress in a viscous medium.

Arkema multiwalls are significantly inferior to Cabot in terms of their “technological” use for providing antistatic properties: only exposure to ultrasound for 500 minutes at a content of 0.15 % allowed an acceptable resistance of $3 \cdot 10^6$ Ohm·cm to be obtained. The agglomerates of these tubes are quite durable – clusters up to 5 μm in size were present in the composition even after many hours of ultrasonic treatment.

Single-walled tubes have the most effective impact

on the electrical resistance of the epoxy composition. Even at a concentration of 0.04 % by weight in the composition, it was possible to achieve a resistance of about 10^6 Ohm·cm. For single-walled CG300, the efficiency of mechanical deagglomeration increases with an increase in the percentage of nanotubes: here, a positive effect of significantly increasing viscosity can be traced, although it was not measured in this work.

Electron microscopy of a composite with 0.04 % by weight of single-walled tubes after mechanical deagglomeration shows the presence of both single tubes and clusters of tubes, although the required resistance has already been obtained (Fig. 1).

A further increase in the content of single-walled tubes does not lead to a significant decrease in electrical resistance: neither a mechanical cutter nor, even more so, ultrasound is capable of “dispersing” agglomerates at such a concentration and viscosity of the composition, which resembles tar.

Unfortunately, experimental ECNTs have almost no effect on the electrical resistance of the composition: even long-term treatment does not produce results. Most likely, this is due to the low purity of this product and requires more thorough research.

4. CONCLUSIONS

The tests conducted proved the effectiveness of most of the nanotubes studied in reducing electrical resistance and achieving antistatic properties in filled epoxy composites.

The use of single-walled tubes allows for a significant reduction in their number and achievement of the percolation threshold through rapid and effective mechanical

deagglomeration with a milling cutter. This technology has limitations and cannot ensure quality when their content exceeds a few hundredths of a percent.

Although multi-walled Cabot tubes are inferior to single-walled ones, they do not significantly increase the viscosity of the system and can be used in quantities an order of magnitude greater than single-walled ones with the use of mechanical deagglomeration.

It has been determined that the use of resistance measurement can serve as an alternative measure of the degree of deagglomeration within a single system.

The optimal use of single-wall and multi-wall tubes can be achieved by applying a sequential combination of several technologies, which requires further research.

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Особливості надання епоксидним системам антистатичних властивостей з використанням нанотрубок

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У роботі досліджено особливості надання антистатичних властивостей епоксидній системі шляхом введення вуглецевих нанотрубок. Розглянуто вплив нанотрубок різних типів і виробників. Визначено мінімальний вміст багатостінних та одностінних нанотрубок, необхідний для досягнення електричного опору 10^6 Ом·см. Порівняння впливу багатостінних нанотрубок фірми Cabot Corporation (продукт "Cabot"), фірми Graphistrength (продукт "Arkema") та одностінних нанотрубок фірми CHASM Advanced Materials (продукт "Signis", модель CG300) показало, що одностінні нанотрубки Signis забезпечують необхідну провідність уже при концентрації понад 0,02 % мас., надаючи антистатичних властивостей поверхні зразка. Проаналізовано вплив технологій попередньої обробки нанотрубок на ефективність зниження електричного опору в кінцевому полімерному матеріалі. Показано, що відпалювання та кислотна обробка мають незначний вплив на досягнення бажаних характеристик кінцевого продукту полімеризації. Встановлено, що досягнення необхідного електричного опору можливе й за умов застосування механічної деагломерації, хоча ефективність ультразвукового обладнання залишається найвищою. Диспергування ультразвуком можливе лише за контрольовано низької в'язкості системи, що важко реалізувати через її зростання зі збільшенням вмісту нанотрубок, особливо одностінних. Крім того, тривалий час експозиції ультразвуком накладає обмеження на його застосування. Використання в'язкості як чинника, що сприяє деагломерації нанотрубок під дією зсуву при механічному змішуванні епоксидної смоли, дозволяє досягти ефективної та відносно швидкої деагломерації за мінімального вмісту нанотрубок Signis і Cabot.

Ключові слова: Епоксидна система, Вуглецеві нанотрубки, Антистатичні властивості, Механічне змішування, Перколяційний поріг.