



## Original articles

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## Electrophysical properties of composite materials based on graphene oxide and polyaniline

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### Abstract

The Hall method was used to study the electrical characteristics of composite materials based on polyaniline (PANI), graphene oxide (GO), and manganese. A comparison of these characteristics of GO-PANI and GO-PANI-Mn composite systems with GO and PANI monomaterials was carried out.

It was demonstrated that the electrical conductivity of composites was significantly higher than that of monomaterials and was determined by the charge carrier mobility.


Based on UV-visible and IR spectroscopy data, it was shown that the optical band gap ( $E_g$ ) of the GO-PANI composite increased with the addition of metal, but decreased compared to PANI; the shift of characteristic vibrations to lower frequencies indicated a covalent interaction of the GO-PANI composite with manganese cations.

**Keywords:** Composite materials, Polyaniline, Graphene oxide, Specific conductivity, Band gap

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## 1. Introduction

The combination of the advantages of various materials for the formation of electroactive composites is a technological breakthrough in the creation of advanced materials for the improvement of the performance of electronic devices and super capacitors. This is confirmed by a number of publications that have shown that nanocomposites based on polymers, nanostructured carbon and transition metal oxides can have high electronic conductivity [1–5]. At the same time, a sharp increase in charge transfer in hybrid structures was observed depending on the type of chemical bond between the components of the composites. In the process of diagnosing the properties of such nanocomposites, the significance of the influence of interfacial interactions and chemical bonds on their conductive properties becomes obvious.

As a conducting polymer, PANI is characterized electrical conductivity, which can be controlled by synthesis conditions [6]. The inclusion of components such as transition metals or their oxides into the PANI structure is often used to improve the selectivity and sensitivity of chemical interactions [7]. However the influence of the dopant on the characteristics of PANI is not clear. Doping PANI with divalent ions of copper, zinc, nickel, cobalt, and cadmium leads to an increase in the electrical conductivity of the composites compared to pure polyaniline, associated with the ordering of polymer chains as a result of interaction with metal ions [7, 8]. At the same time, modification of polyaniline with ZnO nanorods leads to a decrease in the electrical conductivity of the composite compared to pure PANI [9]. Modification of PANI with MnO<sub>2</sub> oxide showed that this material has high intrinsic electrical conductivity and good kinetic reversibility as a result of the interaction between MnO<sub>2</sub> and PANI [10]. It is assumed that the introduction of inorganic dopants into the conductive polymer matrix of polyaniline is the most promising way to create nanocomposites, where delocalized  $\pi$ -electrons can interact with inorganic nanoparticles, leading to an improvement in its conductive properties [11].

Graphene can form numerous nanocomposites with other elements or functional groups. The high conductivity of graphene and its oxide is

ensured by low resistance to ion diffusion, which leads to an increase in power and energy density [12, 13]. However, the processing of graphene materials is difficult due to agglomeration and repacking of graphene sheets as a result of interplanar  $\pi$ - $\pi$  interactions and van der Waals forces, which can significantly limit the diffusion of electrolyte ions between graphene layers [14]. A solution to the problem is to separate graphene by suitable spacers such as carbon-based materials, metals or metal oxides and other pseudo-capacitive materials such as conductive polymers [15]. Graphene-PANI nanocomposites are promising electrode materials due to the fact that graphene and nanostructures obtained from it have a significant effect on improving the properties of conducting polymers, such as electrical properties and structural stability [16]. The distribution of graphene in PANI matrix allows avoiding graphene aggregation, increasing the number of pathways for charge transfer and improving the charge carrier mobility. Functional groups of graphene oxide provide high interaction with polyaniline due to hydrogen and electrostatic interactions.

In this study, the electrical characteristics and structure of synthesized composite systems with the GO-PANI and GO-PANI-Mn compositions, in comparison with monomaterials were studied.

## 2. Materials and methods

Graphene oxide was prepared using a modified Hummers method [17]. So, 10 g of graphite was mixed with 10 g of sodium nitrate and 450 ml of concentrated H<sub>2</sub>SO<sub>4</sub> for 30 minutes at 0 °C. Then 60 g KMnO<sub>4</sub> was added, maintaining a temperature of 15–20 °C. The solution was stirred for 2 h at 40 °C. After addition of 800 ml of deionized water, the solution was stirred for 30 min, raising the temperature to 85–90 °C. Then 2000 ml of water and 60 ml of 30% H<sub>2</sub>O<sub>2</sub> were added. The resulting mixture was washed with a 30% HCl solution to remove sulphates from the graphite oxide solution. PANI powder was synthesized using an acid-free technique described previously [18]. To obtain the GO-PANI composite, at the first stage, GO was functionalized with an amino groups by a simple solvothermal method [19] by the following procedure. 100 mg of GO was dispersed in 35 ml of ethylene glycol, then the dispersed mixture was

stirred at ambient temperature for 30 min and placed in an autoclave. The autoclave was placed in a preheated convection oven at 180 °C for 6 h. The autoclave was then cooled to ambient temperature, the suspension was washed with deionized water and dried at 70 °C for 6 h. GO-PANI composites were prepared by mixing GO-NH<sub>2</sub> powder with aniline/ammonium persulfate solution. The aniline/GO ratio was 2:1. In the case of the GO-PANI-Mn composite, manganese sulphate was added to the specified solution in a way that the proportion of Mn in the sample was 3 wt. %.

IR absorption spectra were recorded using Fourier spectrophotometer (FSM-1202) in transmission mode in the wave number range 350–4250 cm<sup>-1</sup> with a step of 2 cm<sup>-1</sup>.

Absorption spectra in the UV and visible region were recorded using dual-beam spectrophotometer (Unico 2804, USA) in the wavelength range from 190 to 1100 nm. Optical band gap ( $E_g$ ) of the test samples were determined using the Tauc method:

$$(\alpha hv)^{1/n} = f(hv),$$

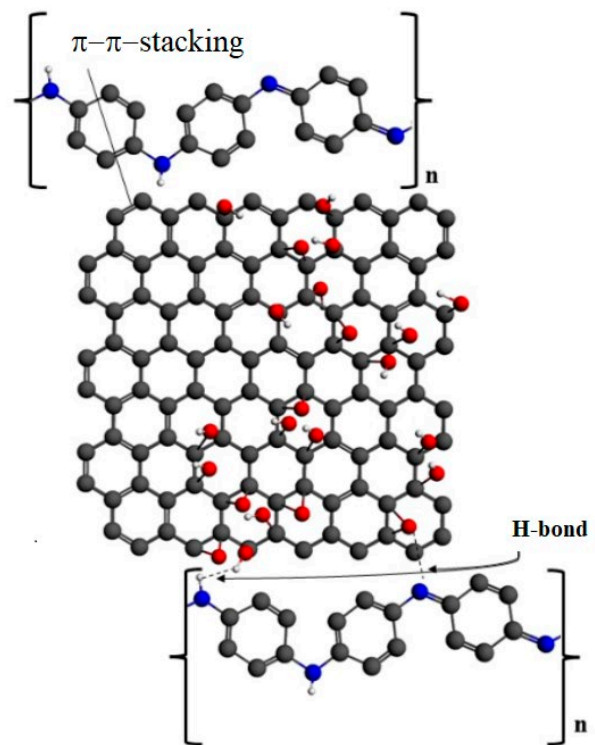
where  $\alpha$  – absorption coefficient,  $h$  – Planck's constant,  $\nu$  – photon frequency,  $n = 1/2$ .  $E_g$  was determined by extrapolating the tangent to the linear part of the spectrum to the intersection with the axis ( $h\nu$ ).

The specific conductivity, concentration and charge carrier mobility of the synthesized materials were measured using the four-probe Hall method on an HMS-3000 instrument. The powder samples were pressed under a pressure of 30 atm in the form of square tablets measuring 7×7 mm and 1 mm thick. The measurements were carried out at room temperature.

### 3. Results and discussion

As a result of Hall measurements, it was established (Table. 1), that the electrical conductivity of monomaterials is significantly

lower than that of composites. Thus, the specific conductivity of graphene oxide and polyaniline was  $1.12 \cdot 10^{-4}$  and  $1.1 \cdot 10^{-7}$  Ohm<sup>-1</sup>·cm<sup>-1</sup> respectively. For composites a significant increase in specific conductivity up to 1.16 (GO-PANI) Ohm<sup>-1</sup>·cm<sup>-1</sup> and 0.78 Ohm<sup>-1</sup>·cm<sup>-1</sup> (GO-PANI-Mn) compared to monomaterials (GO, PANI) was observed. This anomalous increase in specific conductivity may be due to  $\pi$ - $\pi$  stacking (interplanar interactions) between the polymer base and graphene oxide plates, Fig. 1 [20]. On the surface and in the pores of graphene oxide there are OH groups that promote the formation of hydrogen bonds between graphene oxide and nitrogen in the



**Fig. 1.** Schematic representation of the interaction of graphene oxide with polyaniline (blue – nitrogen, red – oxygen, black – carbon, white – hydrogen). The figure is based on the figure from [19]

**Table 1.** Comparative electrophysical characteristics of GO, PANI, GO-PANI, GO-PANI-Mn, obtained by measurements with the four-probe Hall method

Characteristics	GO	PANI	GO-PANI	GO-PANI-Mn
Charge carrier density $N$ , cm <sup>-3</sup>	$2.74 \cdot 10^{12}$	$6.04 \cdot 10^{10}$	$5.8 \cdot 10^{18}$	$1.3 \cdot 10^{19}$
Charge carrier mobility $\mu$ , cm <sup>2</sup> /V·s	$2.5 \cdot 10^2$	$1.15 \cdot 10$	1.2	$3.6 \cdot 10^{-1}$
Specific resistance $R$ , Om·cm	$8.87 \cdot 10^5$	$8.9 \cdot 10^6$	$8.5 \cdot 10^{-1}$	1.27
Specific conductivity $C$ , Om <sup>-1</sup> ·cm <sup>-1</sup>	$1.12 \cdot 10^{-4}$	$1.1 \cdot 10^{-7}$	1.16	0.78
Tupes of conductivity	<i>p</i> -type	<i>p</i> -type	<i>n</i> -type	<i>p</i> -type

benzenoid and quinoid fragments of the polymer chain. The presence of a stretched hydrogen bond between polyaniline and graphene oxide affects the stretch of the  $\pi$ -conjugated bond in the polymer. In addition, polymerization on the surface and in the pores of graphene oxide sheets limits the twisting of the polymer chain, which plays an important role in increasing electrical conductivity [21]. When comparing the electrical parameters of composite materials, it was found that the introduction of manganese reduces the specific conductivity from 1.16 to 0.78  $\text{Ohm}^{-1}\cdot\text{cm}^{-1}$ , despite the fact that the concentration of charge carriers in the GO-PANI-Mn sample is higher than that of GO-PANI. Based on these results, it can be concluded, that the conductivity of composite materials is determined by the charge carrier mobility, which is noticeably lower for manganese-containing materials ( $3.6\cdot 10^{-1} \text{ cm}^2/\text{V}\cdot\text{s}$ ) compared to the GO-PANI sample ( $1.2 \text{ cm}^2/\text{V}\cdot\text{s}$ ).

In UV-visible spectra PANI, GO-PANI and GO-PANI-Mn three absorption bands were observed, characteristic of the transition  $\pi \rightarrow \pi^*$  (peak A, A\*), and shoulder B, characteristic of the transition  $n \rightarrow \pi^*$  (Fig. 2, inset). The peak A was due to the  $\pi$ - $\pi^*$  electronic transition in benzene rings and is characteristic of all forms of PANI. The A\* peak was due to the polaron/bipolaron transition [22]. The long-wavelength feature of B is associated with the excitation of an electron from the highest occupied molecular orbital of benzene rings to the lowest unoccupied molecular orbital of quinoid rings [23], i.e., an excitonic transition from the benzenoid fragment to the quinoid fragment [24]. Peaks A, A\* reflect intrachain interactions, and shoulder B indicates both intrachain and interchain interactions. The estimation of the band gap of GO, PANI, GO-PANI and GO-PANI-Mn are shown in Figure 2. Band gap values  $E_g$  were 2.7 eV for GO, 3.8 eV for PANI, 3.5 eV for GO-PANI, and 3.6 eV for GO-PANI-Mn. It can be noted that the addition of GO to the polymer sample led to a decrease in the band gap (GO-PANI). This is consistent with earlier results [25], where it was shown that the band gap in GO-PANI systems decreased with increased proportion of graphene in the composite. Optical absorption in conjugated polymers can be caused by the transition of charge carriers through the forbidden energy gap. Decrease in

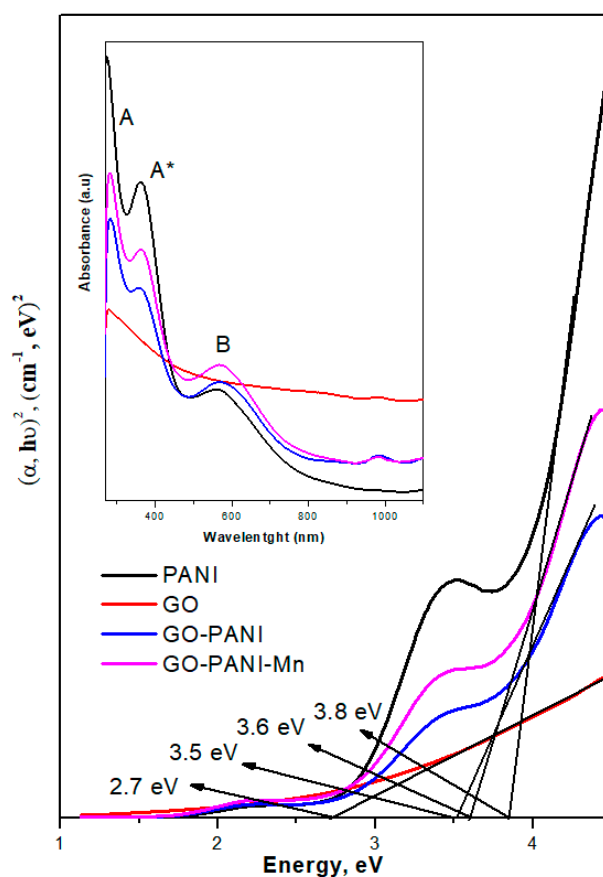


Fig. 2. UV-visible spectra (inset) and optical band gap ( $E_g$ ) of GO, PANI, GO-PANI and GO-PANI-Mn

$E_g$  was due to the appearance of new excitation energy levels due to charge transfer from GO to PANI. The addition of manganese increased the band gap of the GO-PANI-Mn sample, probably due to ordering of the structure. This led to a decrease in the number of localized states in the band gap, contributing to its increase. As a consequence, a decrease in specific conductivity with a simultaneous decrease in the charge carrier mobility was observed.

The IR-spectra of PANI were characterized by the presence of absorption bands corresponding to the C=N and C=C vibrations of the benzenoid and quinoid rings located at 1582 and 1505  $\text{cm}^{-1}$ . Peaks at 1297 and 1306  $\text{cm}^{-1}$  were associated with C=N vibrations of secondary aromatic amines [26] and also the presence of stretching vibrations in the O-H group, approximately from 3650 to 3050  $\text{cm}^{-1}$ . When graphene oxide was functionalized with an amine, bands that overlap with the O-H group at 3433, 1726, and 1619  $\text{cm}^{-1}$  appeared. Absorption bands between 3285 and 3527  $\text{cm}^{-1}$  were due to

the stretching of N-H bonds in amine groups, and the band at  $1580\text{ cm}^{-1}$  were determined by the stretching of N-H bonds in the same plane. During the formation of a composite GO-PANI three bands appeared at  $1481$ ,  $1293\text{ cm}^{-1}$ , and in the range  $\sim 1235\text{--}1022\text{ cm}^{-1}$ , which correspond to the C=C vibration of the benzene ring, the C-N vibration of the benzene ring, and the C-N stretching vibration of aromatic amines (Fig. 3) [18, 27]. When manganese was added to the GO-PANI composite, a shift of the bands (Fig. 3) corresponding to the characteristic vibrations of C=C, C-N, N=C, and C-H in GO-PANI-Mn to lower frequencies was observed in comparison with GO-PANI, which indicates the interaction of GO-PANI with metal cations.

#### 4. Conclusions

A comparison of the electrical characteristics of composite systems of GO-PANI and GO-PANI-Mn composition with GO and PANI monomaterials showed that the electrical conductivity of monomaterials is significantly lower than that of composites. During the formation of composites, an anomalous increase in electrical conductivity was observed, which may be due to interfacial interactions between the polymer base and graphene oxide plates, effective for improving the conductive properties of materials. It has

been shown that the conductivity of composite materials is determined by the charge carrier mobility. Optical band gap ( $E_g$ ) of GO-PANI composite increased with the addition of metal, but decreased compared to PANI. The decrease in  $E_g$  was due to the appearance of new excitation energy levels due to charge transfer from GO to PANI. The addition of manganese increased the band gap of the GO-PANI-Mn sample, probably due to ordering the structure. This led to a decrease in the number of localized states in the band gap, contributing to its increase. The shift of the characteristic vibration bands of C=C, C-N, N=C, and C-H in GO-PANI-Mn to lower frequencies indicates the covalent interaction of the GO-PANI composite with manganese cations.

#### Author contributions

Nedodkova O. V. – IR and UV measurements, analysis of results, design of text and drawings. Myasoedova T. N. – development of synthesis technology and synthesis of samples, measurements by the Hall method, writing of the article. Yalovega G. E. – concept of the article, scientific supervision of research, text writing.

#### Conflict of interests

The authors declare that they have no known competing financial interests or personal

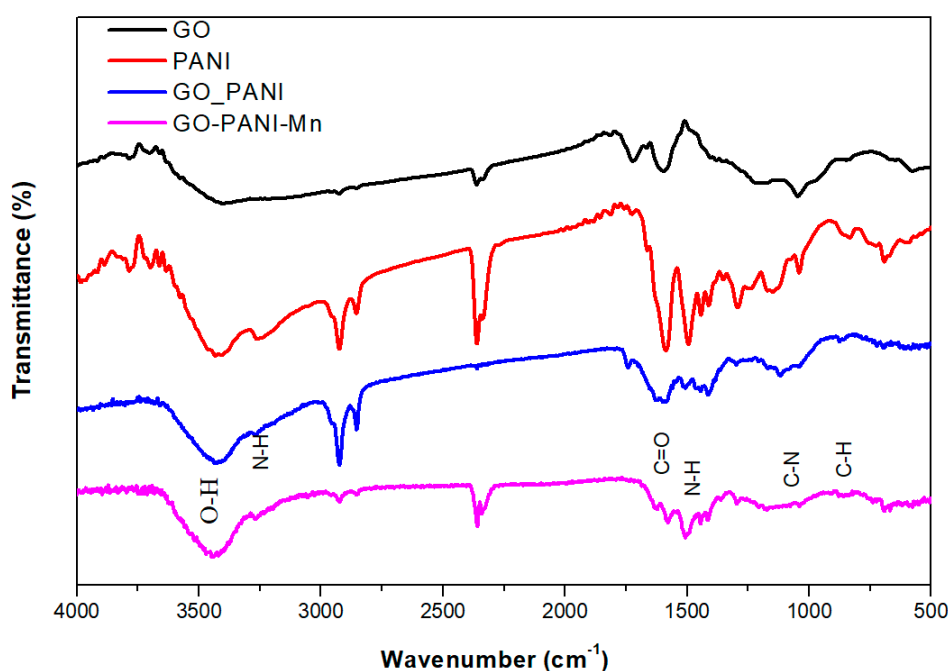


Fig. 3. IR-spectra of GO, PANI, GO-PANI and GO-PANI-Mn

relationships that could have influenced the work reported in this paper.

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