

## The influence of synthesis parameters and thermal treatment on the optical and structural properties of zinc oxide-based nanomaterials

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In this paper, the synthesis of high-quality nanostructured zinc oxide (ZnO) and zinc oxide-graphene oxide (ZnO-GO) nanocomposites was carried out by a simple and efficient cost-effective chemical bath deposition method. The effect of different GO concentrations as well as thermal treatment on the structural, optical and photoluminescent properties of ZnO-GO semiconductor composites was investigated. All the synthesised samples were studied by electron microscopy, energy dispersive X-ray and Raman spectroscopy. ZnO usually exhibits luminescence in the visible region of the spectrum due to various intrinsic defects. This emission can be controlled by surface modification, doping and various treatments of the synthesised materials after synthesis. The considered ZnO-GO composites were subjected to thermal treatment in air at 375°C for one hour. The study of photoluminescence spectra showed that the heat treatment affects different types of optical recombination in the considered samples of zinc oxide – graphene oxide composites. The improved photoluminescence properties of ZnO-GO nanocomposites make them promising for application in various optical devices of nano- and microelectronics.

**Key words:** zinc oxide, chemical bath deposition, optical properties, photoluminescence, raman spectra, heat treatment.

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### 1 Introduction

Zinc oxide based nanostructures are very interesting for scientist materials due to its wide range of applications [1, 2]. In addition, nanostructured ZnO is an inexpensive, safe and relatively simple semiconductor material to fabricate [3]. The chemical and physical properties of zinc oxide nanoparticles can be easily modified by controlling the morphology, using different synthesis methods or different precursors [4]. ZnO belongs to inorganic compounds of semiconductor group II-IV. Zinc oxide nanoparticles are white powder, insoluble in water. The bandgap width is 3.37 eV and binding energy is 60 MeV, which provides high chemical, electrical and thermal stability [5, 6]. Due to their optical, electrical and photocatalytic properties, zinc oxide nanoparticles are used in solar cells, as

photocatalysts, in chemical sensors, piezoelectric nanogenerators, light-emitting diodes and many other electronic devices [7 – 11].

Currently, many studies are devoted to graphene, graphene oxide and composites based on it. The interest in these materials is due to the fact that they have unique electronic and crystalline properties arising from the nature of charge carriers that behave like relativistic particles [12]. Graphene consists of only carbon atoms, connected by  $sp^2$  – hybrid orbitals forming a honeycomb lattice [13]. In turn, graphene oxide is a two-dimensional material with such peculiarities as large specific surface area, high optical transparency and electron transfer capabilities [14]. The above properties allow to consider one as a promising material for bio-application. Commercially produced graphene oxide can be used in solar cells, hydrogen storage, transparent conductive films,

polymer composites, in biomedicine, nanoelectronic devices and in biosensors [15 – 17].

An analysis of literature data showed that modification of nanostructured zinc oxide by adding graphene oxide to the solution effectively prevents aggregation of ZnO particles and provides high stability in the environment [18]. The large specific surface area of graphene oxide and wide forbidden zone of ZnO particles can significantly improve the electrochemical properties of ZnO-GO composites compared to pure zinc oxide or graphene oxide.

One of the most urgent issues is the synthesis of nanostructures with specified, i.e. necessary for this or that application, properties. For this purpose, it is necessary to develop controlled methods for obtaining nanoparticles and determine the optimal parameters [19]. In order to obtain active ZnO-GO composites with the required properties, various treatments are used. Air annealing, hydrogen atmosphere annealing, hydrogen plasma treatment, etc. are often applied. The thermal treatment is one of the most valuable techniques that can significantly affect the properties of nanostructured materials. Many researchers reported that treatment affects the physicochemical properties of zinc oxide and zinc oxide based composites [20-22]. Annealing as a surface treatment process can not only remove surface impurities or defects, but also it can change the surface desorption and absorption of oxygen molecules, leading to the surface states improvement of nanocomposites [23]. It is commonly known that photoconductivity in nanostructures is controlled by the desorption or adsorption of oxygen on the surface, hence the thermal treatment leads to the improves the photosensitivity as well as photoresponse of ZnO by modifying the surface and improving the structure [24]. In turn, annealing in hydrogen atmosphere is often used to improvement of the optical and electrical characteristics of nanoparticles, because hydrogen atoms can passivate broken bonds on the surface and energy band gap states [25].

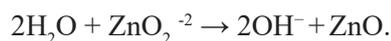
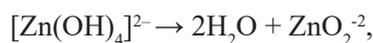
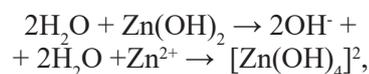
In this work, the optical, structural, as well as photocatalytic properties of ZnO, ZnO-GO semiconductor materials synthesised by a simple, economical and low temperature chemical solution deposition method subjected to heat treatment in air were studied.

## 2 Materials and methods

Zinc oxide (ZnO) and zinc oxide-graphene oxide (ZnO-GO) nanostructured semiconductor materials were synthesized at room temperature by chemical bath deposition [2]. An aqueous solution of 50mM

zinc acetate dihydrate ( $(\text{CH}_3\text{COO})_2\text{Zn}\times 2\text{H}_2\text{O}$ , 98% purity) was used for the synthesis of zinc oxide. The beaker containing this solution was placed on a magnetic stirrer. 0.7M aqueous solution of sodium hydroxide (NaOH, 98% purity, Component-Reaktiv, Russia) was added gradually to the zinc acetate solution while stirring thoroughly. Subsequently, the precipitated zinc oxide was thoroughly washed with distilled water during centrifugation and dried in atmosphere at 115°C for half a day. Sample #1 was synthesised in this way.

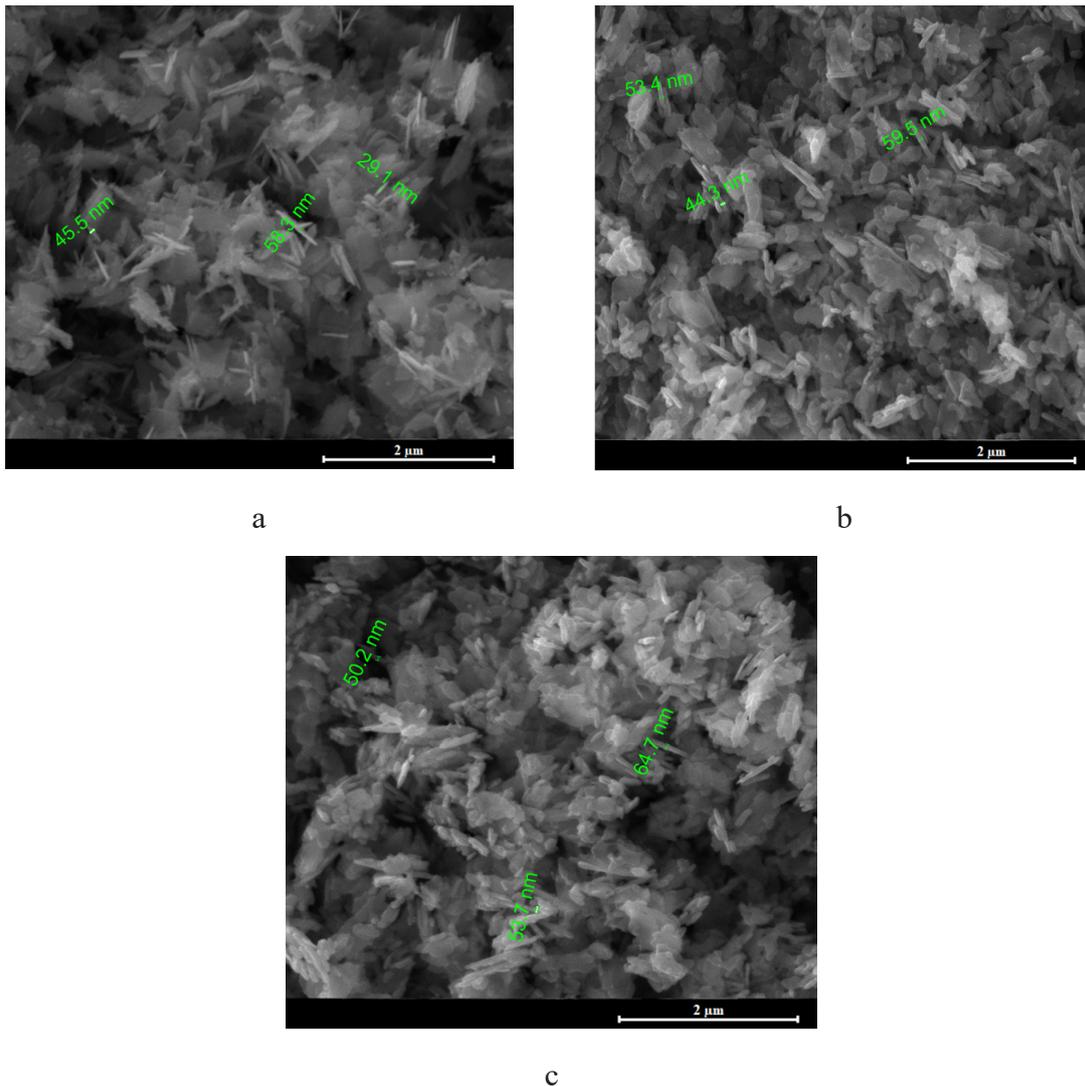
According to the literature [26], the following chemical reactions occur during low temperature chemical bath deposition of zinc oxide:



During zinc oxide-graphene oxide composites synthesis the difference was only that an aqueous solution of zinc acetate dihydrate was mixed with an aqueous solution of prepared graphene oxide synthesised by Hammer's method [27], and then a solution with alkali was added dropwise. The GO concentration was 0.1 wt.% (sample #2) and 0.2 wt.% (sample #3). Samples #4 and #5 were synthesised by thermal annealing a portion of samples #2 and #3, respectively, in an atmosphere at 375°C for one hour. The temperature of 375°C is the minimum at which significant changes in the photoluminescence spectra and Raman spectroscopy spectra of ZnO-GO samples are observed.

## 3 Results and discussion

An electron scanning microscope (JEOL, JSM-6490 LA) was used to study the morphology of ZnO and ZnO-GO synthesised samples. The analysis of the morphology of ZnO and ZnO-GO samples by SEM showed that zinc oxide and zinc oxide – graphene oxide composites at low-temperature synthesis by chemical bath deposition grow as two-dimensional thin plates with length and height of the order of several hundred nanometres. The thickness of the plates was of the order of a few tens of nanometres (Figures 1, 2). No significant difference in the structure of ZnO-GO and ZnO samples was observed.



**Figure 1** – SEM of samples: a – ZnO (sample #1), b – ZnO-GO (sample #2), c – ZnO-GO (sample #3)

Elemental analysis (EDAX) was carried out for samples #2, #3, #4 and #5 by energy dispersive X-ray spectroscopy, which showed that all the samples considered contain carbon (C), oxygen (O) and zinc (Zn) atoms (Figure 3).

It is noticed that in sample #3 there is more carbon than in sample #2, which is explained by the large content of GO in the growth solution. After thermal annealing of ZnO-GO samples #2 and #3 at 375°C in air, the content of carbon atoms in these samples decreased, which is apparently due to the release of carbon dioxide during thermal air treatment of the samples.

Optical density spectra of the synthesised nanostructured samples were studied on a PerkinElmer, UV/Vis Lambda35 spectrophotometer. The optical properties of ZnO and ZnO-GO samples

were studied by spectroscopy in the UV and visible region. Optical density spectra of ZnO and ZnO-GO samples are presented in Figure 4a. The spectra demonstrate significant UV-absorption and negligible absorption in the visible region. ZnO possesses by a broad UV-absorption peak (360-400 nm), characterized hexagonal wurtzite phase of ZnO. The band gap of a pure ZnO sample and ZnO-GO composites was estimated using the Tauc's ratio for allowed direct transitions:

$$(\alpha h\nu) = A (h\nu - E_g)^n,$$

where  $h\nu$  is the photon energy,  $\alpha$  is the coefficient of absorption,  $A$  is the proportionality factor,  $n = 0.5$ , since zinc oxide is a direct-gap semiconductor material [28].

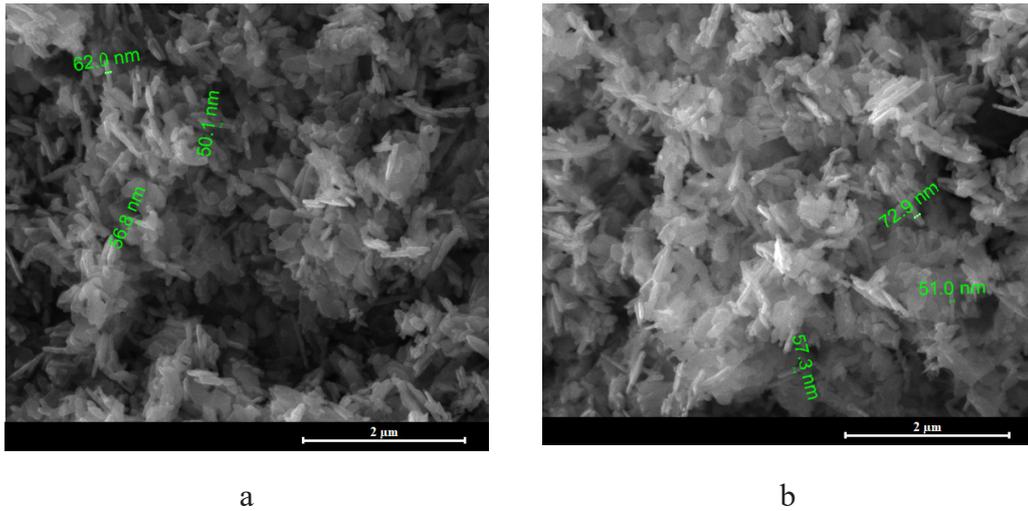
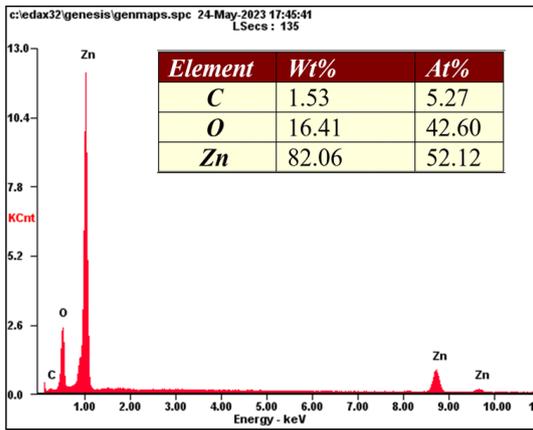
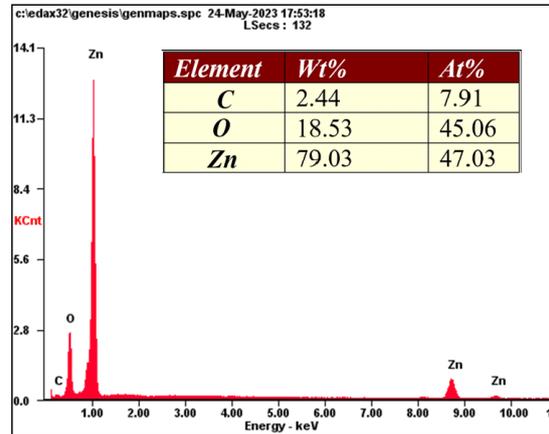


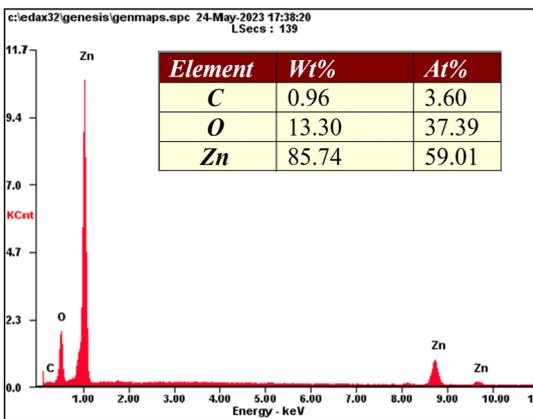
Figure 2 – SEM of samples: a – ZnO-GO (sample #4), b – ZnO-GO (sample #5)



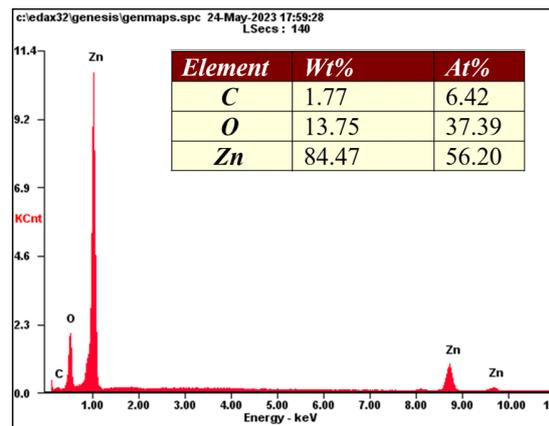
a



b

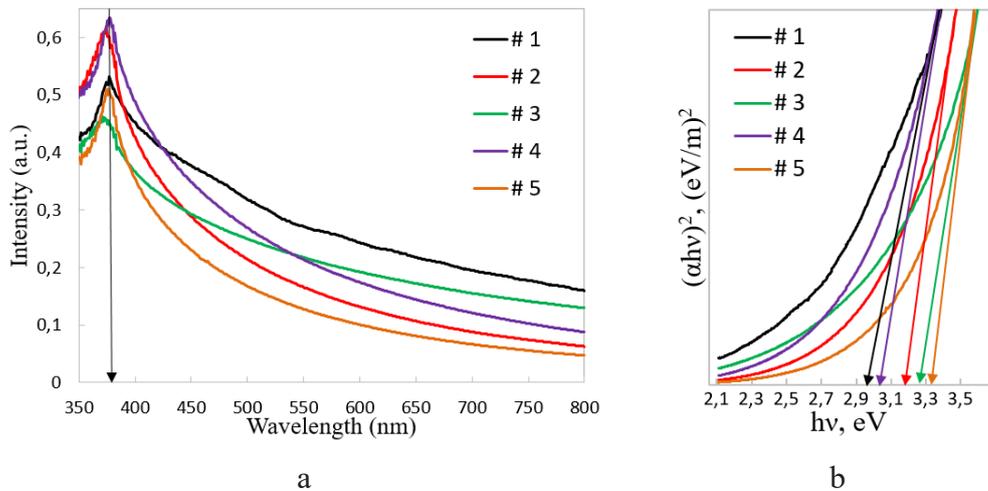


c



d

Figure 3 – EDAX ZnO-GO samples: a – sample #2, b – sample #3, c – sample #4, d – sample #5



**Figure 4** – Optical properties of ZnO and ZnO-GO samples:  
(a) absorption spectra, (b) Tauc Plot diagrams

The extrapolation the linear part of the dependence of  $(\alpha h\nu)^2$  on  $h\nu$  allow estimate band gap energy in the optical range  $E_g$  (Figure 4b). The  $E_g$  value of all samples is on the order of  $(3.2 \pm 0.15)$  eV.

Photoluminescence (PL) spectra were measured at room temperature using a Cary-Eclipse spectrophotometer. A xenon lamp emitting at a wavelength of 300 nm provided optical excitation.

Photoluminescent properties of semiconductor nanostructures are actively investigated with the prospect of subsequent application in biological markers, lasers, organic light-emitting diodes, quantum electronics, etc. Photoluminescence spectra were measured and studied for all ZnO and ZnO-GO samples. Figure 5 shows the photoluminescence spectra measured at room temperature. The excitation energy used (4.1 eV) is higher than  $E_g$  of ZnO (3.37 eV), so the electron from the valence band due to excitation can both move to the conduction band and to deep levels inside the forbidden band [29]. In the PL spectra of the considered ZnO and ZnO-GO samples, two emission bands are observed: one in the UV region (385 nm) and the other in the visible region (450-650 nm). The UV emission peak at 385 nm corresponds to the near-band emission (NBE) of ZnO and is due to the radiative recombination of free excitons [30]. When the size of nanocrystals decreases, sample impurities and various defects come to the surface as a result an increasing in the fraction of surface atoms providing impurity photoluminescence (DLE). The ratio of NBE and DLE intensities is often related to the crystal quality and the state of trapped defects.

The photoluminescence spectra show that samples #2 and #3 exhibit visible emission in the

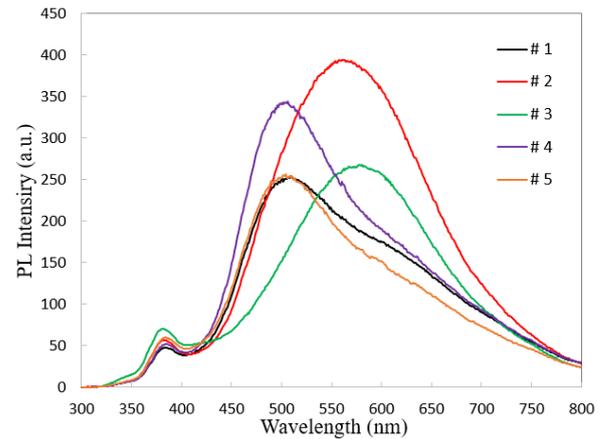
yellow and yellow-orange regions with a peak at wavelengths of 565 nm and 581 nm, respectively. The radiative recombination of localised electrons with deeply trapped holes in oxygen interstitials located at 2.2 and 2.14 eV below the conduction band results in yellow and yellow-orange luminescence (DLE) bands, respectively [31]. It was observed that after atmospheric heat treatment at 375°C in ZnO-GO samples #4 and #5, the DLE band shifts from yellow to green region. The nature of green luminescence in ZnO is the most controversial; there are many hypotheses to explain this luminescence [30-33]. According to some of them, the green luminescence band observed at 505 nm can be associated with recombination of electrons in singly ionised oxygen vacancies with photoexcited holes in the valence band [30, 32]. In this case, samples # 4 and # 5 showed typical edge emission of the band at 383 nm. It was noted that the PL intensity of the UV-band decreased after heat treatment, which may be explained by the partial dissociation of the exciton bound to the donor [32]. Thus, it is obvious from the photoluminescence spectra that annealing of the synthesised nanoparticles clears the samples of moisture and affects various types of optical recombination [33].

Raman scattering for all synthesised samples was examined by a Solver Spectrum spectrometer. The excitation was carried out by means of a blue laser at 473 nm. The Raman spectra of the synthesised samples are shown in Figure 6. The spectrum of ZnO sample #1 (Figure 6a) shows the presence of peaks at  $100 \text{ cm}^{-1}$ ,  $330 \text{ cm}^{-1}$ ,  $437 \text{ cm}^{-1}$ ,  $575 \text{ cm}^{-1}$  and  $1150 \text{ cm}^{-1}$ . The peak at  $330 \text{ cm}^{-1}$  corresponds to zone-boundary phonons of hexagonal ZnO, while the peak at  $437 \text{ cm}^{-1}$ , belongs to the characteristic E2 (High) mode of the wurtzite

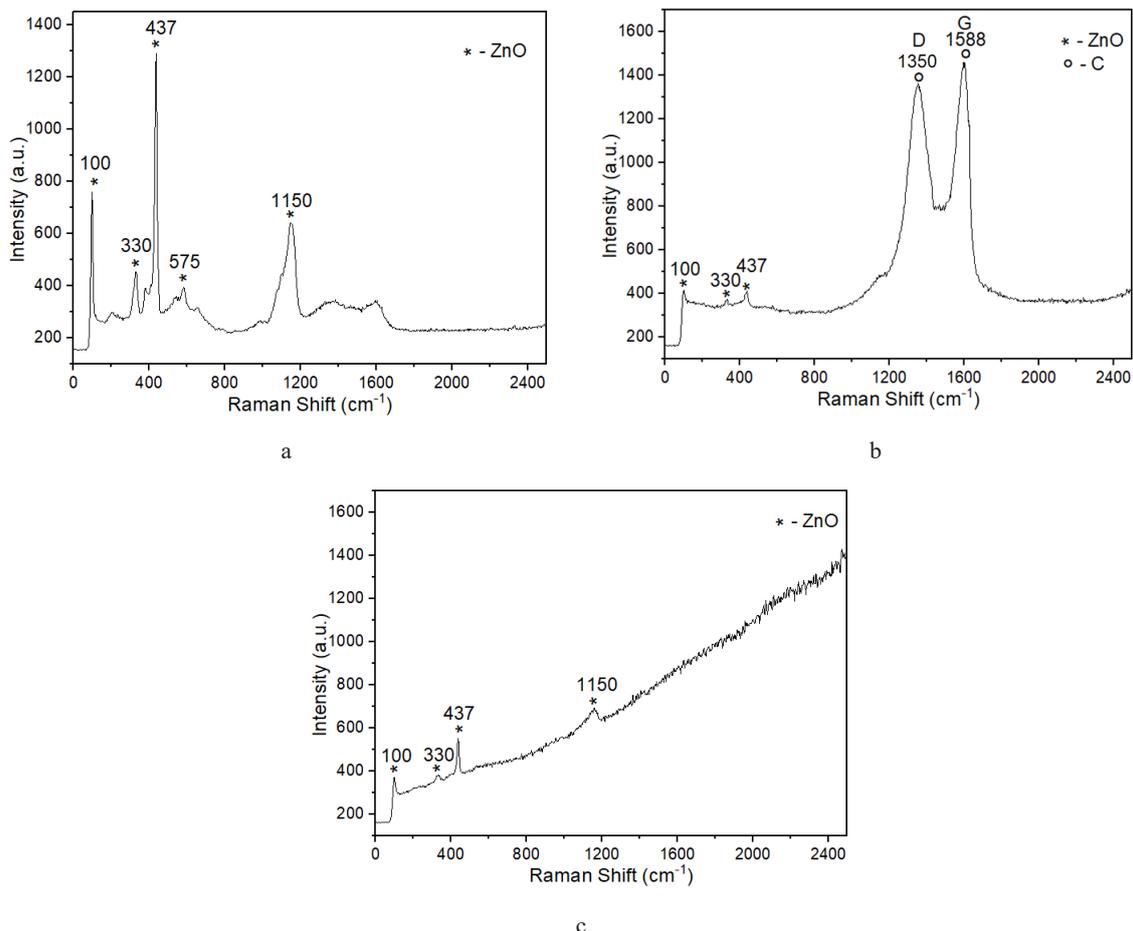
phase of ZnO [35]. The peak present at  $575\text{ cm}^{-1}$ , belong to transverse optical modes with A1 symmetry and longitudinal optical (Low) modes characteristic of defects (oxygen deficiency) present in ZnO [36]. The observed peak at  $1150\text{ cm}^{-1}$  corresponds to the phonon scattering phenomenon [37].

The spectra of ZnO-GO samples (Figure 6b) show two main peaks at  $1350\text{ cm}^{-1}$  and  $1588\text{ cm}^{-1}$ , characterised by the D and G bands, respectively. The D band arises due to defects  $sp^3$ , and the G band due to the planar vibrations of carbon atoms  $sp^2$  and the twice degenerate phonon mode [38].

After thermal treatment of the ZnO-GO samples, the peaks characteristic of the presence of GO in the samples do not appear (Figure 6c), which seems to be due to the reduction of carbon atoms (Figure 3) and also as a result of the green DLE luminescence increasing after thermal treatment (Figure 5).



**Figure 5** – Photoluminescence (PL) spectra of ZnO-GO and ZnO samples



**Figure 6** – Raman spectra: a – ZnO (sample #1), b – ZnO-GO (sample #3), c – ZnO-GO (sample #5)

## 4 Conclusions

Nanostructured ZnO and ZnO-GO samples were synthesised by a simple low-cost method of chemical bath deposition. The morphology, as well as electrochemical and structural characteristics of the synthesised ZnO and ZnO-GO samples were studied. The results of the study of the synthesised samples by SEM showed that the synthesised samples grow as two-dimensional thin plates with length and height of the order of a few hundred nanometres and thickness of the order of a few tens of nanometres. It was demonstrated that the thermal treatment of the synthesised ZnO-GO nanoparticles in air at 375°C is not only able to

affect various types of optical recombination. Thus, the proposed method of synthesis of ZnO-GO and ZnO nanoparticles with subsequent heat treatment allows controlling their luminescent and structural properties, which makes these materials promising for application in the production of white light-emitting diodes, display devices, biological labelling and other optical devices of nanoelectronics.

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