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Transformation of absorption and luminescence spectra of carbon nanodots under UV irradiation

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The effect of ultraviolet radiation on the optical absorption and fluorescence spectra of the colloidal system of carbon nanodots in ethanol has been investigated. The laser dye Nile red was used as a precursor to synthesize the nanodots. The emission spectrum of the nanodots has the shape of a broad weakly structured asymmetric band with a maximum at $\lambda \approx 652$ nm. Ultraviolet irradiation (250−400 nm) leads to a significant transformation of the optical spectra of the dots, in particular, to the disappearance of the intense absorption band of the nanodots in the visible region, bleaching of the solution and an increase in absorption in the ultraviolet region of the spectrum. The changes in absorption are accompanied by quenching of fluorescence of nanodots and a change in its spectral composition: with increasing irradiation time, the fluorescence color changes from red to green, and short-wavelength radiation with a maximum at ~ 500 nm begins to dominate in the spectrum. The observed effects are associated with photochemical reactions in carbon nanodots, leading to changes in their composition and structure.

Keywords: carbon dots, optical spectra, fluorescence, ultraviolet radiation, photochemical reactions.

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1. Introduction

Carbon dots (CD) attract attention due to a good combination of such qualities as bright fluorescence, low toxicity, low price and relative simplicity of synthesis. CDs are carbon nanoparticles, usually of spherical shape, with size of up to 10 nm, the main elements of which are layers of graphite (graphene) and graphene oxide fixed by sp^3 hybridized carbon inserts [1,2]. In process of CD synthesis their surface turns out to be enriched with various functional groups (carbonyl, carboxyl etc.), which substantially impact the optical properties, chemical activity, degree of hydrophilic behavior and other characteristics of CDs [3–7]. The ability to include various functional groups into the CD composition opens the way towards the intended modification of their properties [8,9]. Apart from using in optoelectronics, information storage and transformation devices, catalysis, chemical probing, CDs are of special interest from the point of view of various biomedical applications [10–14].

Successful use of CDs in various applications assumes understanding of the role of various external factors in formation of their luminescent properties. In particular, luminophore interaction with light may result in both photoluminescence excitation, and various photochemical transformations, which, in their turn, may impact the luminophore properties. First of all, we mean ultraviolet (UV) radiation, which is widely used to excite luminescence. The results of UV radiation influence on the CD luminescent properties are contradictory. Some researchers note high photostability of CDs under UV radiation [15,16], while other experiments noted substantial decrease of fluorescence (FL) intensity of CDs as a result of UV radiation [15,17], while the spectrum shape hardly changes [18]. The effect is related to photochemical transformations in CDs (in particular, to their partial de-oxygenation [19,20]), causing the change in the structure of functional groups in CDs, which are responsible for luminescent properties. On the other hand, you may expect that the change of the CD structure under the exposure to UV radiation may result in formation of new radiation centers within a CD. Such changes of the structure may substantially impact the radiation spectrum of CD, in particular, cause appearance of new (or reinforcement of the available) bands in the CD radiation spectra. The possibility to create structural defects in graphite under exposure to UV radiation was demonstrated experimentally [21]. This paper reports substantial transformation of optical spectra in colloidal CD solutions under the exposure to UV radiation, which is manifested by, in particular, their bleaching and change of the FL spectral composition.

2. Methods

CDs were synthesized in nanopores of spherical particles of the mesoporous silica (SiO_2). The precursor was laser dye Nile red (NR) ($C_{20}H_{18}N_2O_2$, Sigma-Aldrich). Particles of mesoporous silica were prepared using the method described in [22]. The particle diameter was 500 ± 20 nm, the diameter of nanopores — 3.10 ± 0.15 nm. Silica particles were impregnated with 33 % (vol) NR solution

in methanol (99.8 %, Acros), dried at 50 °C and annealed on air at 330 °C for 2 h. Thermal decomposition of NR led to the formation of highly monodisperse CDs in silica nanopores [23]. To separate the CDs from the silica matrix, the latter was dissolved in fluoric acid (ACS reagent grade). After dissolution of the silica particles, the CDs sediment was cleaned from SiO_2 dissolution products. The concentrated CD suspension was used to prepare colloidal CD solutions in ethanol. CD concentration in the solution was $\sim 10^{15}\,\mathrm{cm}^{-3}$ at pH ≈ 6. Analysis of IR absorption spectrum of synthesized CDs [24] indicate the presence of the graphite structure in their composition, as well as hydroxyl, carbonyl and carboxyl functional groups.

Electronic transmission spectra of CD solutions in the area of 200–1000 nm were recorded using spectrophotometer SF2000. CD's FL was excited by radiation of laser with wavelength of $\lambda_{exc} = 405$ nm. Excitation density did not exceed $0.5\,\mathrm{W/cm}^2$. The CD solution was placed into a standard quartz cuvette 10×10 mm. The exciting radiation was normally incident on the surface of the cuvette, CD's FL was recorded at the right angle to the direction of the exciting light propagation. The radiation spectra were recorded using a diffraction spectrometer. The analysis of the temperature dependence of CD's FL anisotropy on the basis of the Perrin equation [25] makes it possible to estimate the dot size as 3.5 ± 0.3 nm, which agrees well with the results of the CD size measurement by electron microscopy. For UV exposure of CD solutions, UV radiation of ultrahigh pressure mercury lamp DRSh-250-3 was used with the wavelength 250–400 nm.

3. Experimental results and discussion

Electronic absorption spectra of CD in ethanol are shown in Figure 1. You can see that the absorption spectra of CD and NR are similar to each other. Bands with maxima at 206, 248 and 490 nm are identified in the CD spectrum. Short-wave bands in the area $\lambda < 300$ nm are usually associated with $\pi\pi^*$ transitions in the graphite core with participation of carbon atoms in state of sp^2 hybridization [26]. (Band 206 nm is observed, in particular, in absorption of submicron particles of pure graphite [27]). Band at 248 nm is caused by $\pi\pi^*$ transitions in C=C/C=N groups [28], even though some authors relate peak of 248 nm with optical transition in O–H group coupled with carbon nucleus [29]. Longer-wave absorption is related to the defects of the CD surface caused by the presence of C=O- and C=N-groups in their composition [30]. For comparison, Figure 1 also shows the spectra of NR solution in ethanol. In the NR spectrum you can clearly see the absorption bands with maxima at 202, 223, 265, 305 and 552 nm. The longest-wave band 552 nm responds to the optical transition with transfer of electron density from the nitrogen atom in diethyl amino group and the backbone of NR molecule to heteroatoms, mainly, to cyclic nitrogen and carbonyl oxygen (electron transition HOMO → LUMO) [31,32]. Based on the similarity of CD and NR absorption spectra, you can assume that if the band 490 nm in the CD spectrum has the same nature as the band 552 nm in the NR spectrum. Such situation is quite expected, since molecules or fragments of molecules of the precursor may be incorporated into CD in process of synthesis [33,34].

FL spectra of CD and NR are presented in Figure 2. Spectra are dominated by the intense band of red FL with wavelength 605 nm (in case of CD) or 642 nm (in case of NR). The 642 nm band depends on radiative transition LUMO → HOMO in an NR molecule. The difference in positions of the longest wave bands in CD (490 nm) and NR (552 nm) absorption spectra and their corresponding radiation bands (605 and 642 nm) defines the Stokes displacement of FL in these systems: 0.45 and

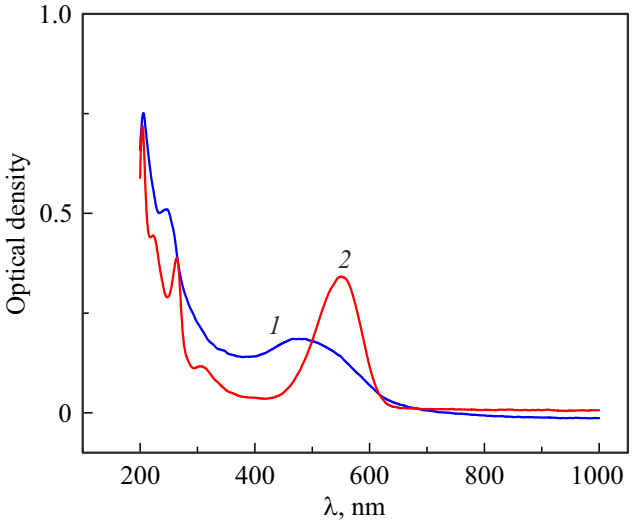

Figure 1. Absorption spectra of CD (*1*) and NR (*2*) solutions in ethanol.

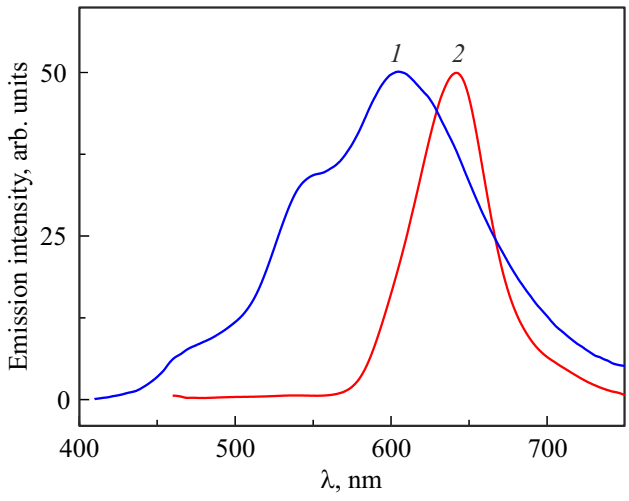

Figure 2. Emission spectra of CD (*1*) and NR (*2*) solutions in ethanol. $\lambda_{exc} = 405$ nm. $T = 300$ K.

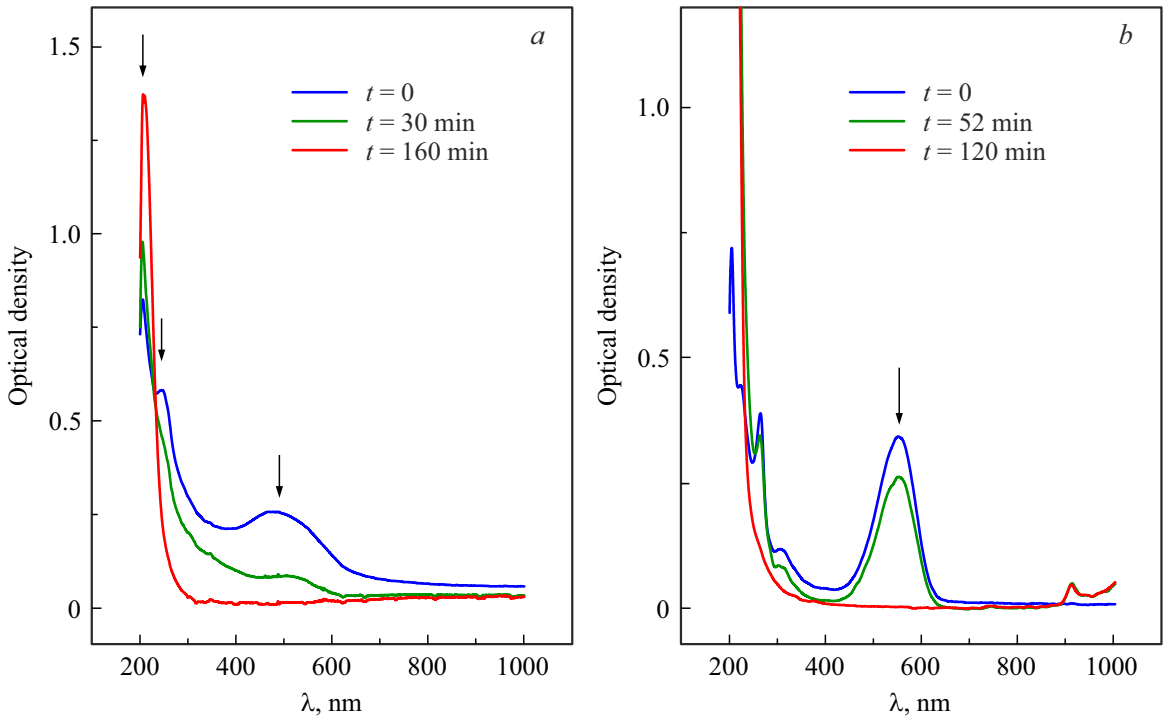

Figure 3. Effect of UV radiation on absorption spectra of CD (*a*) and NR (*b*) in ethanol. Radiation time *t* is specified on the insert.

0.32 eV, respectively. More complex construction of CD compared to NR molecule is manifested in a more complex structure for their radiation spectra. As opposed to NR, in case of CD the FL band is wider, has asymmetric shape with a short-wave wing, on the background of which you may note weak maxima at 470 and 545 nm, being probably caused by radiative transitions with participation of functional groups absent in NR molecule.

Effect of UV radiation. Effect of UV radiation on spectra of CD optical absorption is illustrated by Figure 3. The effect of radiation is diverse. As you can see in Figure 3 UV radiation of CD solution in ethanol causes weakening (up to complete disappearance) of absorption bands in the region of 248 and 490 nm with simultaneous reinforcement of a short-wave peak of absorption in the area of 206 nm. Weakening of absorption bands at 248 and 490 nm may be related to partial loss of oxygen and nitrogen by carbon dots as a result of UV exposure, which causes reduction of mass shares of O and N and respectively to increase of the mass share of carbon in CD [35]. Additional graphitization of CD as a result of UV treatment is confirmed by the amplification in CD spectrum of the 206 nm absorption band specific for graphite. As you can see from Figure 3, similar changes also occur in NR spectrum under exposure to UV radiation.

Dependence of integral intensity of 490 nm absorption band of CD in ethanol on the time of UV radiation *t* is presented in Figure 4.

Similar changes are caused by UV radiation in the CD FL spectrum. As the CD solution is exposed to UV radiation, the width of the dominant band of red FL decreases, and its intensity drops quickly. At the same time a clear maximum of radiation is formed in the field of the short-wave wing of the band at $\sim$ 500 nm, which at long times of UV radiation becomes prevailing in the spectrum, and the integral intensity of FL as the time of UV radiation increases drops significantly (Figure 5). This drop, as shown below, is related mainly to the reduction in the absorptance of the solution in the area of $\lambda > 350$ nm caused by UV radiation

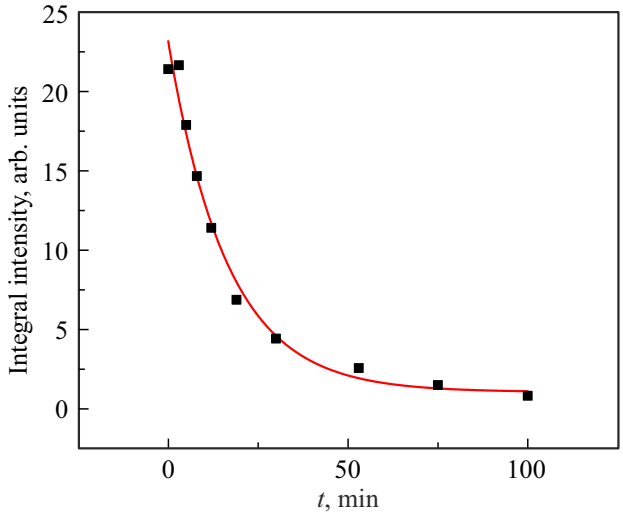

Figure 4. Dependence of integral intensity of CD 490 nm absorption band on time of UV radiation *t*. Dots — experimental data, solid line — approximation of the experimental dependence with equation (4).

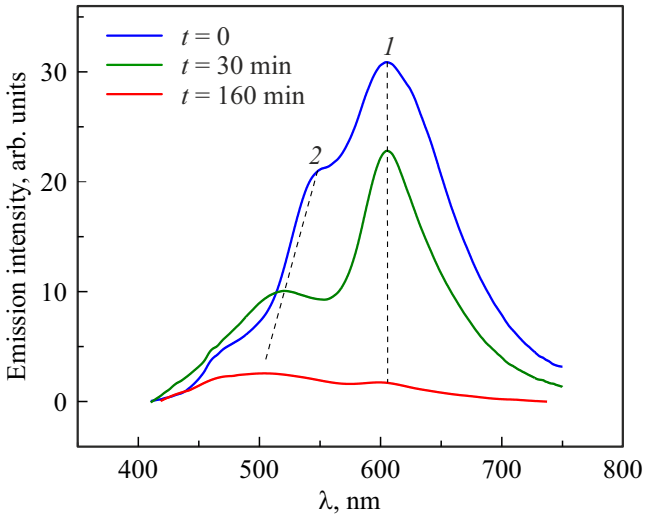

Figure 5. Impact of UV radiation on CD radiation spectrum in ethanol. Radiation time t is specified on the insert. Dotted lines illustrate the displacement of maxima of red (*1*) and green (*2*) FL bands with the course of time t. $\lambda_{exc} = 405$ nm. $T = 300$ K.

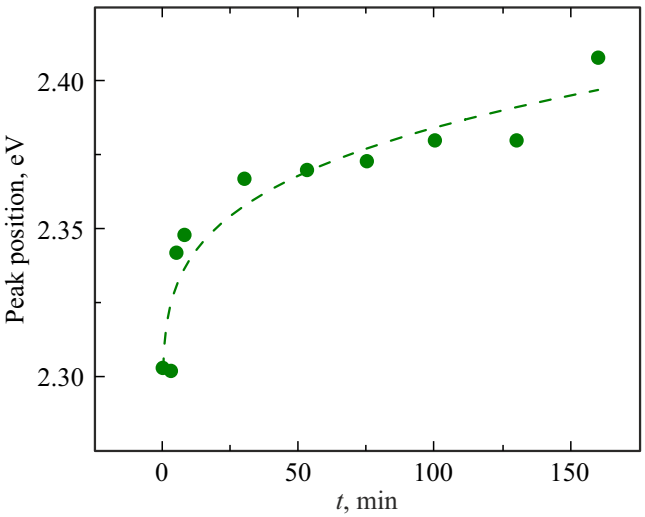

Figure 6. Experimental dependence of CD green FL band maximum position on time of UV radiation t. $\lambda_{exc} = 405$ nm. $T = 300$ K.

(Figure 3), i. e. with the reduction of the efficiency of optical excitation, the intensity of which remained invariable.

Note that if the spectral position of the red FL band almost never changes in process of UV radiation, the maximum of the green FL noticeably shifts with increase of t towards the blue side (Figure 6).

The intensity of the optical absorption and FL bands of nanoparticles is proportional to their concentration (number of nanoparticles per unit of volume) at this moment of time. It is natural to relate the above attenuation of absorption and CD FL in a certain spectral interval with photochemical conversions in CD under the impact of UV radiation, as a result of which CDs of another composition and structure are formed with other photophysical properties, and the number of initial CDs decreases. Let the density of initial CDs in the solution at the moment of time t be n. The light absorption coefficient of an ensemble of such CDs is $\alpha(\lambda) = \sigma(\lambda)n$, where σ — cross section of light absorption with one CD. The speed of changing the density of initial CDs as a result of a photochemical reaction depends on the power I of the absorbed UV radiation:

$$-\frac{dn(t)}{dt} = AI, \tag{1}$$

where A — proportion coefficient (speed of variation of n at unit absorbed power). At thickness of absorbing layer l and intensity of incident UV radiation I_0 the power of the absorbed UV radiation (let us consider the UV radiation monochromatic for simplicity):

$$I = I_0 - I_0 \exp(-\sigma nl) = I_0[1 - \exp(-\sigma nl)] \tag{2}$$

and the kinetic equation (1) will become the following:

$$-\frac{dn}{dt} = AI_0[1 - \exp(-\sigma nl)]. \tag{3}$$

Equation (3) is easily integrated. The solution is quite bulky, which in case of poor absorption (diluted solution: $\sigma n_0 l \ll 1$) becomes:

$$n(t) = n_0 \exp(-AI_0\sigma lt) = n_0 \exp(-t/\tau), \tag{4}$$

where $n_0 \equiv n(0)$, $\tau \equiv (AI_0\sigma l)^{-1}$. Ratio (4) describes the kinetics of the first order chemical reactions. As you can see in Figure 4, ratio (4) describes well the behavior of the CD absorption $\alpha(t)$ in the area of the band 490 nm: $\alpha(t) = \sigma n(t)$ with $\tau = 16$ min.

It is evident that in the general case the photoinduced decay of certain functional groups in CD must result in reduction of the corresponding absorption (for example, disappearance of 490 nm band in Figure 3, *a*), on the other hand, the products of photochemical reaction may also contribute to absorption (for example, amplification of absorption in the area of ~ 206 nm in Figure 3, *a*). At the frequency of exciting light ($\lambda_{exc} = 405$ nm) the light absorption drops significantly with the increase in the time of UV exposure. Therefore, FL quenching (Figure 5) may be caused by at least two factors: (i) photoinduced reduction of exciting light absorption by the solution (analysis shows that $\alpha(\lambda_{exc})$ drops exponentially with increase in the time of UV radiation (Figure 3) with $\tau = 30$ min) and (ii) destruction of the functional groups in CD, responsible for the corresponding FL. The role of the first factor in analysis of the radiation spectra may be excluded by normalization of the spectra intensity to the power of the light absorbed by the solution on the wavelength $\lambda_{exc} = 405$ nm. The spectra normalized in such a way are given in Figure 7, *a*.

FL spectra in Figure 7, *a* actually reflect the quantum yield of FL in CD solution at different t. Note, therefore, that the integral intensity of FL in Figure 7, *a* approximately

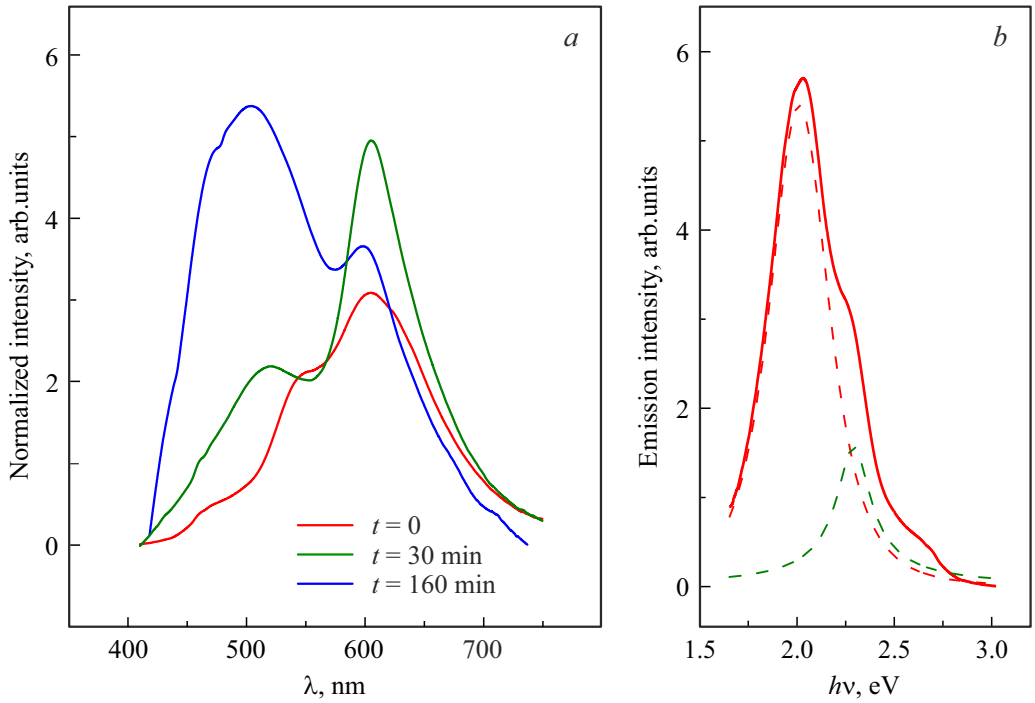

Figure 7. *a*: Impact of UV radiation on CD radiation spectrum in ethanol. Radiation time t is specified on the insert. Spectra are normalized to the light power absorbed by CD solution at wavelength $\lambda_{exc} = 405$ nm. *b*: Decomposition of CD FL contour at $t = 0$ into elementary bands (shown with a dotted line). $\lambda_{exc} = 405$ nm. $T = 300$ K.

doubles as the time of UV exposure changes from $t = 0$ to $t = 160$ min. To assess the effect of the UV radiation on the intensities of the bands of red and green FL, presented in Figure 7, *a*, the spectral contour of FL was decomposed into two bands with the shape close to Gaussian one (Figure 7, *b*). Analysis of the spectra shows that the intensity of the red FL band ($\lambda_{max} = 605$ nm, $h\nu_{max} = 2.03$ eV at $t = 0$), is first rising, but then at $t \gtrsim 75$ min starts falling. At the same time the intensity of green fluorescence ($\lambda_{max} = 545$ nm, $h\nu_{max} = 2.29$ eV at $t = 0$) increases up to $t \approx 130$ min and practically changes no more (Figure 8). It can be believed that the end trend to red FL quenching is caused by the disintegration of the corresponding radiating centers under exposure to UV radiation, and ignition of green FL complies with the reverse process — formation of new radiating centers, which are active in the green area of the spectrum. Within this model, the initial amplification of red FL (in the area of $t \lesssim 75$ min) may be explained by UV radiation-induced rupture of hydrogen bonds between atoms [36], included in the center of red FL (for example, N) and molecules of dissolvent (ethanol). Rupture of hydrogen bonds helps to reduce the speed of nonradiative deactivation of the excited electron state [37], growth of its occupation and, accordingly, increased intensity of radiative transitions. In the area of small UV exposures ($t \lesssim 75$ min) this effect becomes dominant versus the drop of radiation intensity as a result of disintegration of the red FL centers.

Amplification of green FL of CD (Figure 7, *a*) may be referred to the change in their chemical composition related,

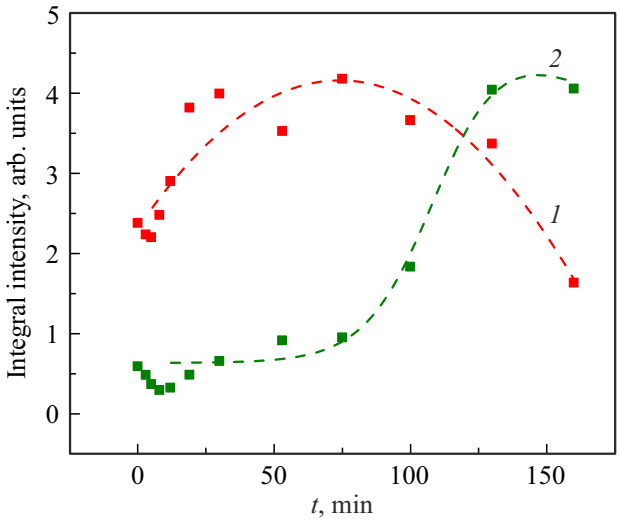

Figure 8. Impact of time of UV radiation t on integral intensity of red (*1*) and green (*2*) FL bands of CD in ethanol. The intensity is normalized to the light power of exciting radiation absorbed by the solution. Dots — experimental values, dotted lines — reference points for the eyes. $\lambda_{exc} = 405$ nm, $T = 300$ K.

in particular, to the effect of deoxygenation, since UV radiation has no effect on morphology and size of CD [38]. As specified above, the component of CD is graphene oxide (GO). GO is a layer of carbon atoms made of small islands of aromatic graphene, randomly distributed within

the layer and surrounded with the carbon atoms functionalized with the oxygen-containing groups. These groups may be arranged on a basal plane of the layer (hydroxyl and epoxide), and on its edge (carboxyl, carbonyl) [39]. Photoreduction of GO accompanied by removal of oxygen-containing functional groups causes appearance of new aromatic graphene domains in the GO carbon lattice with carbon atoms in the state of sp^2 hybridization, while the initially present graphene domains in GO do not increase in size upon reduction [40,41]. Photoreduction of GO under exposure to UV radiation is accompanied by displacement of the radiation band maximum from the red to the green one and further to the blue area of the spectrum [40]. It may be assumed that the similar process of aromatic graphene domain formation under exposure to UV radiation occurs in CD as well, and amplification of the green FL band in the spectrum of nanodots is the optical manifestation of this effect. This assumption is confirmed by the data that it is the aromatic domains with macromolecular carbon material within CD composition who are responsible for green FL of CD [42]. Note that the increase in the share of aromatic domains within the CD as a result of UV radiation is also confirmed by sharp amplification of CD optical absorption in the area of ~ 210 nm, provided for optical transitions with the participation of atoms of aromatic carbon in the state of sp^2 hybridization.

4. Conclusion

UV radiation provides significant impact on optical spectra of absorption and radiation of CDs synthesized on the basis of NR. Photoinduced changes in the spectra are of diverse nature. In case of optical absorption the UV radiation of CD solution in ethanol causes solution bleaching in the visible area and increase in the optical absorption in the UV region with $\lambda \approx 210$ nm. Under the conditions of CD excitation with the light with $\lambda_{exc} = 405$ nm the integral intensity of FL spectrum noticeably drops with the increase of time of UV exposure t, and the relative intensity of FL bands in the spectrum changes significantly. Analysis of the FL spectra normalized to absorbed light power with $\lambda_{exc} = 405$ nm demonstrates that the main effect of UV radiation consists in red FL quenching and green FL amplification, besides, the integral intensity of normalized spectra (quantum yield of FL) increases with increase of t. Photoinduced changes of the FL spectrum are connected with photochemical reactions in CD under exposure to UV radiation, in particular, loss of oxygen and formation of aromatic graphene domains. Therefore, UV radiation may provide noticeable impact on the optical spectra of absorption and FL of CD, which allows for targeted change of their properties. On the other hand, potential impact of UV radiation on CD FL must be taken into account when CDs are used as luminophores in various applications.

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Conflict of interest

The authors declare that they have no conflict of interest.

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