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## EFFECT OF GAMMA RADIATION ON THE THIN NANOCOMPOSITE MEH-PPV/C<sub>60</sub> FILMS

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In this work, the effect of gamma radiation on the photoluminescent properties of thin films of binary nanocomposites MEH-PPV/C<sub>60</sub> (polymer – fullerene C<sub>60</sub>) has been studied. Samples of pure MEH-PPV and MEH-PPV/C<sub>60</sub> nanocomposites were synthesized in ratios of 9:1 and 3:1 on the mica and silicon substrates. Effect of type substrate on photoluminescence of pure thin films and nanocomposites were studied. Experimental data on dose dependences of the shape and intensity of the photoluminescence spectra after exposure of samples with doses up to 67 kGy were obtained and analyzed. The nonmonotonic nature of the change in the intensity of various spectral bands of the photoluminescence of nanocomposites with increasing dose and the absence of a shift of these bands was established. The obtained experimental results were interpreted from the standpoint of competition between the processes of crosslinking and breaking of polymer chains under the action of gamma irradiation. A practically important result of the study is the conclusion that MEH-PPV/C<sub>60</sub> nanocomposites are highly resistant to gamma rays.

**Keywords:** nanocomposite, MEH-PPV, C<sub>60</sub> fullerene, gamma radiation, photoluminescence

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## ВЛИЯНИЕ ГАММА-ИЗЛУЧЕНИЯ НА ТОНКИЕ НАНОКОМПОЗИТНЫЕ ПЛЕНКИ МЕН-PPV/C<sub>60</sub>

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В работе исследовано влияние гамма-излучения на фотолюминесцентные свойства тонких пленок бинарных наноконпозитов МЕН-PPV/C<sub>60</sub> (полимер – фуллерен C<sub>60</sub>). Синтезированы образцы чистых МЕН-PPV, а также наноконпозитов МЕН-PPV/C<sub>60</sub> в соотношениях 9:1 и 3:1 на слюдяной и кремниевой подложках. Изучено влияние типа подложки на спектры фотолюминесценции чистых тонких пленок и наноконпозитов. Получены и проанализированы экспериментальные данные по дозовым зависимостям формы и интенсивности спектров фотолюминесценции после воздействия на образцы экспозиционных доз до 67 кГр. Установлен немонотонный характер изменения интенсивности различных спектральных полос фотолюминесценции наноконпозитов при увеличении дозы и отсутствие смещения этих полос. Полученные экспериментальные результаты трактуются с позиций конкуренции процессов сшивки и разрыва цепей полимера под действием гамма-облучения. Практически важным результатом исследования является вывод о высокой устойчивости наноконпозитов МЕН-PPV/C<sub>60</sub> к действию гамма-квантов.

**Ключевые слова:** наноккомпозит, МЕH-PPV, фуллерен  $C_{60}$ , гамма-облучение, фотолюминесценция

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## Introduction

Nanocomposites based on conductive polymer matrices with different inclusions have yielded encouraging results for the development of optoelectronic devices with flexible parameter settings [1, 2]. Thin-film nanocomposite donor-acceptor structures with bulk heterojunctions are used to create organic solar cells [3]. The  $C_{60}$  fullerene that exhibits strong acceptor properties is widely used in these structures as a component of bulk heterojunctions [4], in particular, combined with conjugated polymers as donor components [5]. Polyphenylene vinylene (PPV) and its derivatives, in particular, МЕH-PPV, are one of the most common conjugated polymers in organic electronics [6].

This polymer is commercially available, and its main characteristics, such as the band gap (about 2.2 eV), the energy of the highest occupied molecular orbitals (HOMO) (5.3 eV), the energy of the lowest unoccupied molecular orbitals (LUMO) (3.1 eV) and the molar mass (40,000–25,000 g/mol), have been obtained. The polymer is widely available at low cost and soluble in standard organic solvents. Good quality films of  $C_{60}$  can be obtained by spin-coating. The conducting polymer МЕH-PPV can act as an electron donor [7]; it has good electrical characteristics, such as conductivity and charge carrier mobility. A technology for creating flexible electroluminescent light-emitting structures and devices based on МЕH-PPV was described in [8, 9] along with the mechanisms of degradation and the methods for preventing it.

A binary compound of the conducting polymer МЕH-PPV with the  $C_{60}$  fullerene is one of the most promising and inexpensive materials for developing optoelectronic devices with the required parameters. The conductivity of the nanocomposite depending on the composition of the МЕH-PPV/ $C_{60}$  mixture was studied in [10]. It was discovered that at room temperature a slight change in the concentration of  $C_{60}$  leads to changes in the conductivity of composite films by several orders of magnitude. The relationship between photoluminescence (PL) quenching and

enhanced photovoltaic effect was established for increasing  $C_{60}$  concentration in [11]. This is associated with photoinduced charge transfer between the components of the mixture forming a bulk heterojunction.

There is currently active research into improving the photoelectric characteristics of the films obtained [12], including methods for film annealing that induce effects associated with rearrangement of polymer chains, polymer interchain interactions and formation of aggregates. For example, [13] considered the dependence of optical properties of nanocomposites on the evolution of their structure under thermal annealing, finding that the operating parameters of the nanocomposites improved due to crystalline transition of the film.

However, thermal modification has not this far yielded the desired results, and different polymer composites can be modified by other methods, for example, by irradiation. It was confirmed that a thermally stable polymer could be obtained by radiation crosslinking of its chains. Ref. [14] found threshold doses after which irreversible changes started, doses of complete subsequent oxidation of fragmented units and the fundamental physical and chemical properties of polymers under irradiation were [14]. Important results were obtained in [15], examining the changes in the molecular weight of a gamma-irradiated polymer induced by cross-linking (elongation) and degradation (shortening) of the main polymer chain depending on the irradiation dose. It was shown that both processes were competing, occurring simultaneously but with different probabilities, which complicates quantitative assessment and prediction of the final properties of the material.

The effect of different doses of gamma irradiation on the spectral properties of conducting polymers МЕH-PPV and BMP at 5°C was investigated in [16], confirming that radiation treatment of solutions of these polymers induced a blue shift in the fluorescence spectrum. This effect was attributed to crosslinking of conductive polymer chains but it was shown in [17] that crosslinking of conjugated polymers did not

cause a shift in the PL spectra. Studies considering the effect of ionizing radiation on fullerenes reported on the stability of fullerene properties under gamma irradiation [18]. The effect of different types of ionizing radiation on nanocomposite materials with added  $C_{60}$  fullerene were discussed in [19, 20]. The presence of fullerene was revealed to improve the absolute stability of the given nanocomposites.

Our study has considered for the first time the effect of gamma radiation on binary systems of the MEH-PPV/ $C_{60}$  (polymer/ $C_{60}$  fullerene) nanocomposite exhibiting photoluminescence upon excitation by laser radiation.

### Experimental procedure

The samples were prepared from industrially available materials by Sigma Aldrich: powder of the conductive polymer MEH-PPV with a number-average molecular weight of about 40,000–70,000 and powder of the  $C_{60}$  fullerene with a mass fraction of 99.95%. The components were dissolved in toluene to obtain thin nanocomposite films of MEH-PPV/ $C_{60}$ . Initial solutions of the MEH-PPV polymer and  $C_{60}$  fullerene with the same mass concentrations were used to obtain the composite. The solutions were first placed in an ultrasonic bath for 20 minutes and then in a thermostat at 50°C for 60 minutes to completely dissolve the polymer and make a homogeneous mixture. After that, the solutions were mixed

with different ratios of MEH-PPV and  $C_{60}$  (3:1 and 9:1). The finished solutions were deposited on silicon and mica substrates by spin-coating, which made it possible to obtain film thickness of the order of 1  $\mu\text{m}$ .

The PL spectra of the samples were recorded using the Horiba Jobin Yvon automated system including a FHR 640 monochromator with a 1200 gr/mm diffraction grating and a Symphony II 1024\*256 Cryogenic Open-Electrode CCD detector with a light yellow filter. PL was excited in the samples by a semiconductor laser with a wavelength  $\lambda = 405 \text{ nm}$ , operating in continuous mode.

The samples were irradiated with gamma-emitting radionuclides  $^{137}\text{Cs}$  with a photon energy of 661 keV in a quasi-closed oxygen-containing chamber. A monochromatic gamma-ray flux, achieved by simultaneously using 84 of these radionuclides, was applied to the samples. The thin films we have examined were exposed to the following irradiation doses (with respect to  $\text{H}_2\text{O}$ ), kGy: 0; 18.3; 42.8; 67.3.

All irradiations and measurements were performed under standard conditions.

### Experimental results and discussion

A bound electron-hole pair (exciton) is formed in the conjugated semiconductor polymer upon absorption of light (laser radiation); the binding energy for this pair in MEH-PPV is about 0.3 eV

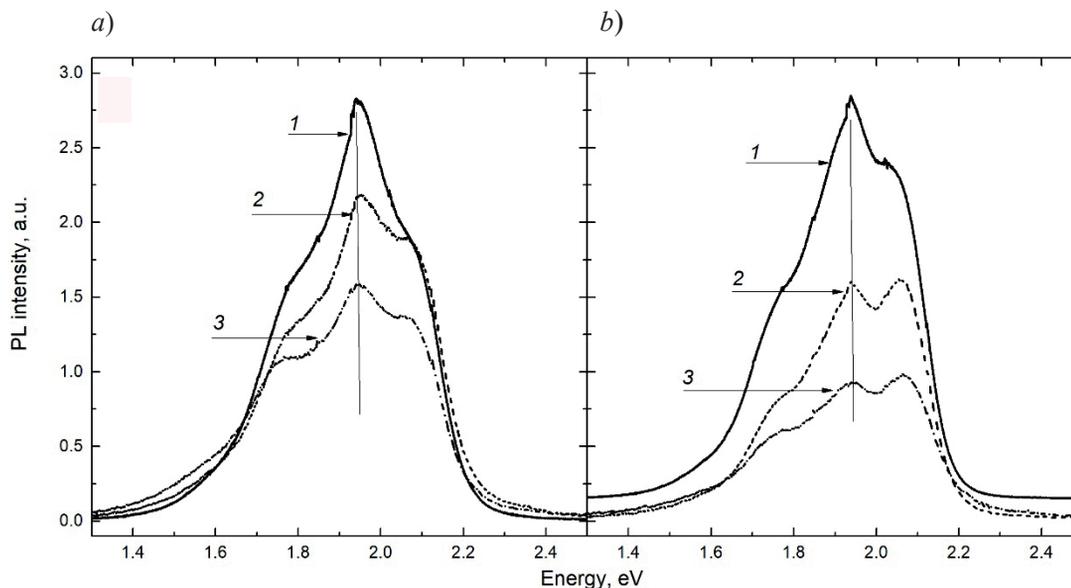


Fig. 1. PL spectra of non-irradiated samples on silicon (a) and mica (b) substrates; data are given for pure MEH-PPV (1) and for MEH-PPV/ $C_{60}$  nanocomposites in 9:1 (2) and 3:1 (3) ratios

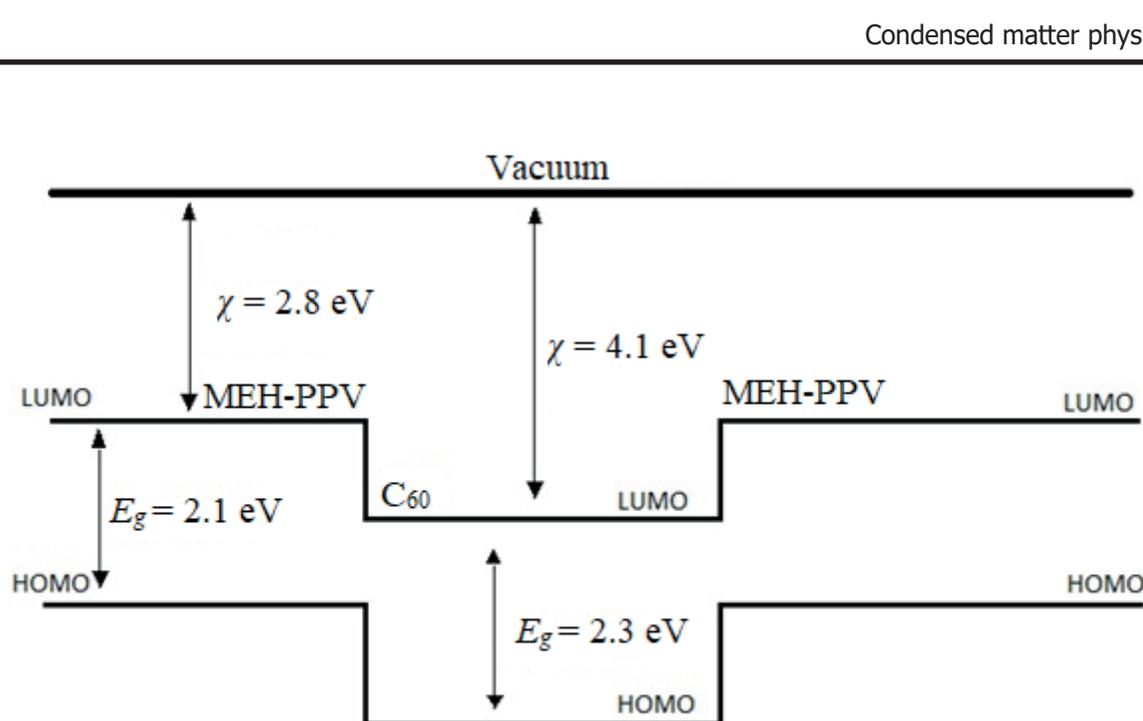


Fig. 2. Energy band structure of MEH-PPV/C60 composite:

HOMO and LUMO are the energy levels of the highest occupied and lowest unoccupied molecular orbitals, respectively;  
 $E_g$  is the band gap;  $\chi$  is the electron affinity energy

[21], which is significantly higher than the energy of temperature fluctuations at room temperature. Fig. 1 shows the changes in the PL spectrum of the nanocomposite depending on the composition of the mixture and the type of substrate. The components of the photoemission spectra of the polymer with energies of 1.94, 1.84, and 1.77 eV are associated, respectively, with the electronic transition and two electron-vibrational replicas.

Ref. [22] established that surface morphology of polymer films depends on the type of substrate. This morphology is characterized by the presence of separated phases, which affects the optical properties of the films. The effect that the substrate has on PL can be observed from the spectra (see Fig. 1). The mica substrate is oriented, while the silicon one (coated with native  $\text{SiO}_2$ ) is non-oriented [23]. The difference in the shape of the spectra of the samples on different substrates is most likely due to the presence of dipoles on the cleaved mica. Dipoles form an ordered crystal lattice and are absent on the silicon surface. As the solvent evaporates and polymer molecules are deposited on the mica substrate, these molecules become oriented (with the orientation depending on the substrate due to the effect of the dipole field), while polymer molecules on the silicon surface are randomly arranged. The type of substrate also affects the intensity ratio of electronic and the electron-vibrational transitions (compare the photoluminescence spectra shown

in Fig. 1, *a* and 1, *b*). Evidently, the intensity of the electron-vibrational transition with the participation of the first vibrational level is higher than that of the electronic transition for the nanocomposite film on mica, and these intensities are approximately equal before irradiation for the film on silicon.

Adding fullerene to the polymer leads to a decrease in the intensity of the emission peaks of the polymer (a similar effect was observed in [13]) due to strong electronegativity of fullerene (Fig. 2). PL quenching indicates photoinduced charge transfer from the polymer chain to fullerene upon photoexcitation of the nanocomposite.

PL quenching efficiency is significantly higher for semiconductor polymers than for low-molecular weight compounds because excitons can migrate along the chain during their radiative lifetimes [24]. Radiative singlet transitions are symmetry-forbidden in the  $\text{C}_{60}$  molecule; its weak phosphorescence region ( $T_1 \rightarrow S_0$  transitions) is in the range of 1.55–1.60 eV and partially overlaps with the “tails” of radiative transitions in MEH-PPV.

Calculations for the concentrations that we used for the composite film components show that there is one fullerene molecule per approximately 26 chain units of the conjugated polymer with a 9:1 composition and one per 9 units with a 3:1 composition (provided that the  $\text{C}_{60}$  molecules are evenly distributed in the composite).

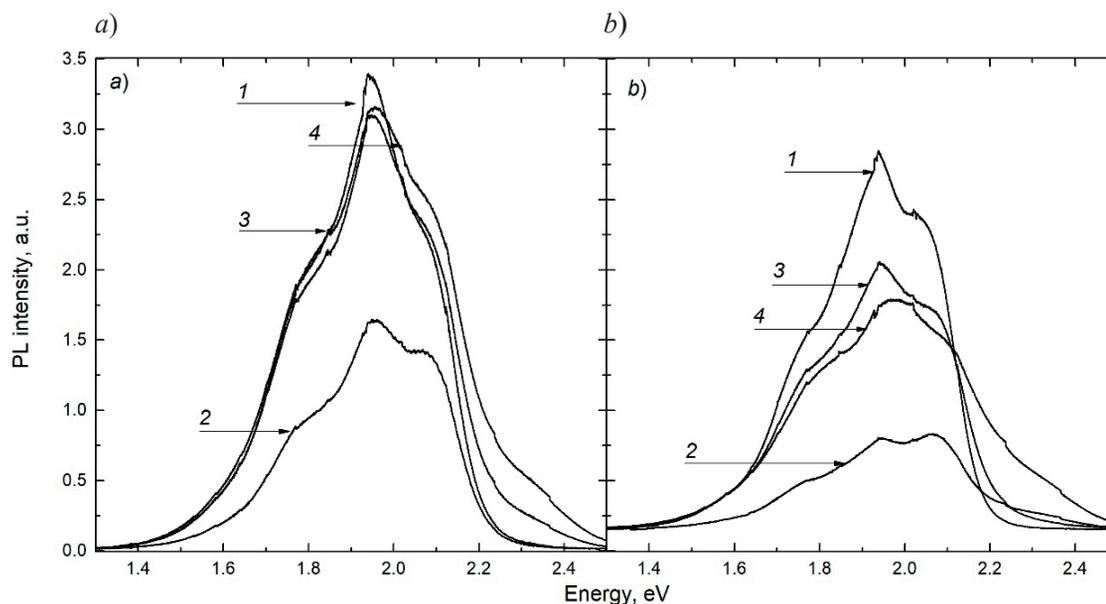


Fig. 3. PL spectra of non-irradiated samples (1) and samples gamma-irradiated with different doses (2–4) on mica (a) and silicon (b) substrates. Radiation doses, kGy: 18.3 (2), 42.8 (3), 67.3 (4)

The size of the delocalization region of the photoexcited exciton is about 14 MEH-PPV units after excitation [25], but it decreases to 5–7 units over a period of the order of 0.1 ps as a result of electron-phonon interaction [26]. If we assume that each  $C_{60}$  molecule forms a quenching center, then the PL intensity of the composite should decrease proportional to the fullerene content at the given concentrations. Analyzing the data in Fig. 1, we can see that the quenching effect is much stronger for films on a silicon substrate than for films on a mica substrate. A possible explanation for this result is that the nature of the substrate has a different effect on the structure of both the pure polymer and the nanocomposite, as well as on the distribution of phases in these materials. PL of the conjugated polymer depends not only on the concentration of quenchers but also on their location. If the quenchers are evenly distributed along the chain, PL quenching can be expected to be more efficient than if they are clustered or aggregated, since  $C_{60}$  molecules in the middle of the cluster do not contribute to quenching. The assumption that fullerene molecules are aggregated in nanocomposite films on mica (unlike its films on silicon) is confirmed by increased PL intensity observed in these samples with increasing fullerene concentration in the range of 1.50–1.55 eV, corresponding to phosphorescence of the  $C_{60}$  phase. Increased fullerene concentration in films on silicon leads to an

increase in the relative probability of electronic transitions, compared with electron-vibrational replicas, which may be due to the kinetics of the quenching process.

Fig. 3 shows the effect of gamma irradiation on the PL spectra of MEH-PPV films on different substrates. While gamma irradiation reduces PL intensity, in contrast with the data reported in [16, 27], no blue shift is observed in the spectral bands. The position of the lines of electronic and vibronic radiative transitions on the dose dependence of the PL spectra remains unchanged even at high doses of radiation. At the same time, a nonmonotonic dependence of PL intensity is observed for both types of substrate. Decreased PL intensity upon exposure to the lowest dose and the subsequent increase in PL intensity with increasing dose might be governed by the competing processes occurring in the conductive polymer exposed to gamma irradiation.

A decrease in PL intensity suggests that not all photoexcited excitons contribute to luminescence. For the bound electron and hole to recombine emitting a photon, they should be located in the same segment of the polymer chain. Recombination centers, which always evolve if the structure is disturbed, might be the reason for the decrease in PL intensity. The electron is trapped on a level with recombination centers, with potential subsequent nonradiative dissociation of the exciton. PL amplitude of the polymer drops sharply after

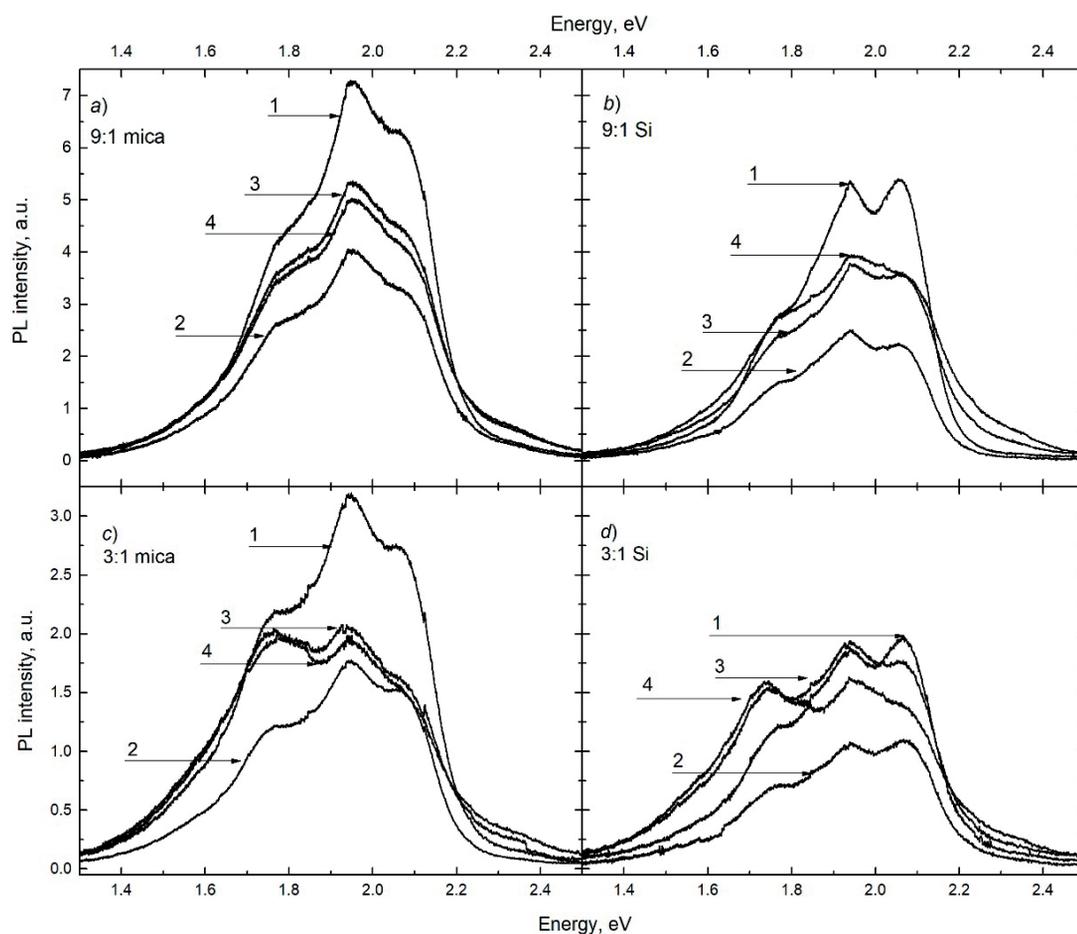


Fig. 4. PL spectra of non-irradiated (1) samples and samples gamma-irradiated with different doses (2–4) on mica (a, c) and silicon (b, d) substrates. The data obtained are for thin films of MEH-PPV/C<sub>60</sub> in ratios of 9:1 (a, b) and 3:1 (c, d).  
Irradiation doses, kGy: 18.3 (2), 42.8 (3), 67.3 (4)

the first dose of irradiation (18.3 kGy), see Fig. 3; this can be explained by the fact that polymer chains mostly undergo crosslinking in this case, while conformation and breaking do not occur yet and the conjugated segments of the chain remain long [28]. Exciton migration along polymer chains is rather long, and effective PL quenching occurs at a relatively low concentrations of recombination centers. Degradation of polymer chains into short coordinated conjugated regions whose length is still larger than the size of the delocalization region of the photoexcited exciton is predominant at higher doses of gamma radiation. Migration of photoexcitons to recombination centers is complicated, and this leads to an increase in PL amplitude (see Fig. 3).

Fig. 4 shows PL spectra of nanocomposite thin films with fullerene after exposure to gamma radiation. Evidently, irradiation has a different

effect on the intensity of electronic transition and its electron-vibrational replicas. This is particularly pronounced in compounds with a high fullerene content, where there is one C<sub>60</sub> molecule per about 9 polymer chain units. We assume a homogeneous molecular dispersion of quenchers along the chain of the conjugated polymer for such nanocomposites synthesized on a silicon substrate. It can be seen that gamma irradiation has little effect on such a composite but the probability of electronic transitions slightly decreases slightly, while probability of electron-vibrational transitions (at high doses of irradiation) actually increases. As our model presumes that chains are broken under exposure to large doses of gamma irradiation, this weak effect can be explained by the fact that the length of the shortened conjugated sites is still significantly larger than the average distance between the quenching centers.

In this case, polymer chains breaking into short sections have little effect on PL intensity of the nanocomposite (given the high intensity of exciting laser radiation) where it was initially quenched. The effect is less pronounced for nanocomposite films synthesized on a mica substrate, where we assume that  $C_{60}$  molecules aggregate into clusters, and, accordingly, the concentration of quenching centers is lower, which corroborates the explanation we proposed. However, gamma irradiation was found to reduce the intensity of the electronic transition more significantly than that of the electron-vibrational one for both types of nanocomposites. This behavior of luminescence in the composites is due to high stability of fullerene as a PL quencher under gamma irradiation.

### Conclusion

As a result of the study, we have obtained data on the effect of gamma irradiation with doses up to 67.7 kGy on the photoluminescence (PL) spectra of thin films of conjugated

polymer MEH-PPV and its nanocomposites with fullerene (in 9:1 and 3:1 ratios).

We have discovered that the oriented substrate affected the distribution of the PL quencher in the nanocomposite.

We have established that gamma irradiation of the samples does not lead to a shift in the position of radiative transitions in the PL spectra of the polymer; however, we have observed nonmonotonic decrease in the PL intensity with increasing dose.

For nanocomposites with a high fullerene content (ratio

No changes in the PL intensity have been found for nanocomposites with a high fullerene content (9:1 ratio) under large doses of gamma irradiation (starting with 42.8 kGy). This can be attributed to initial effective quenching of photoexcited excitons.

Thus, the main practically important finding of our study is that the MEH-PPV/ $C_{60}$  nanocomposites have high resistance to gamma radiation.

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