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INFLUENCE OF CdTe NANOPARTICLES ON THE FORMATION OF J-AGGREGATES OF THIAMONOMETHINECYANINE DYES

Conditions of J-aggregate formation have been studied for three types of thiamonomethinecyanine dyes, the structures of which differ from one another by their end groups, and the dependences of those conditions on the dye concentration and the type of dye interaction with CdTe nanoparticles with a diameter of 3 nm in aqueous dispersions have been analyzed. The dye structure is found to influence the efficiency of the J-aggregate formation in solutions and films. It is also found that CdTe quantum dots (QDs) stabilized by thioglycolic acid can adsorb J-aggregates of dye molecules on their surface. It is shown for the first time that the hybrid structure dye-CdTe can emerge owing to the interaction between the negatively charged dye molecules and the negatively charged surface of QDs forming neutral aggregates. No process of energy transfer from the dye to a CdTe particle was detected in the dimer-CdTe and J-aggregate-CdTe systems.

Keywords: thiamonomethine cyanine, thioglycolic acid, polymethine, pseudoisocyanine, floc cules, peptization, redispersion, Förster mechanism.

1. Introduction

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Polymethine dyes turn out to be unique converters of light energy in the visible and near infra-red spectral ranges [1, 2]. They are capable of enhancing or weakening radiation, as well as changing the radiation wavelength, depending on the environmental conditions. This fact provides an opportunity to use those dyes as active media in lasers, photoresistors, and so forth [3]. Under the influence of external factors, polymethine dyes form J-aggregates characterized by narrow absorption and luminescence bands, which enables them to be applied in photography and in the manufacture of LED displays [4–6].

J-aggregates of cyanine dyes attract a certain attention for a long time due to their unusual properties such as a high thermal stability [7] and a substantial increase of the luminescence intensity in binary compounds [8] and polymer films [9]. In particular, Filimonova et al. [7] have observed, the formation of J-aggregates of carbocyanine derivatives dye in solutions of reverse micelles and the structure of those aggregates were studied, and the corresponding photoluminescence spectra were shown to be dependent on the temperature. As a result, the cited authors came to the conclusion that the J-band can become narrower and its intensity higher if the solution temperature decreases. On the other hand, the results concerning the interaction of cyanine dyes of three types in solutions with the surfactant ncetylpyridinium bromide were reported in work [8]. The interaction was demonstrated to enhance the ag-

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Fig. 1. Chemical structures of dyes

gregation of those dyes, which, in turn, increases the luminescence quantum yield. The results obtained by the authors of work [9] are not less interesting. In particular, the efficiency of the excitonic transport in J-aggregates of PIC in thin films of polyvinyl alcohol (PVA) was researched, by using the excitonic trap method. It was shown that 50% of the luminescence by J-aggregates was quenched in those films.

Depending on specific conditions, polymethine dyes can form various molecular aggregates, in particular, dimers and J-aggregates [10–12]. Therefore, not less interesting is the study of the ability of those organic dyes to self-organize under condition of introduction of nano-dimensional impurities, which promote their J-aggregation, in the solutions. For instance, in work [13], the influence of a clay impurity on the spectral characteristics of PIC in the solution was considered. The authors of work [13] demonstrated that J-aggregates can be easily formed in such solutions and proved their high stability.

Recently, the attention has grown to the study of the influence of finely dispersed particles, such as CdSe, on the J-aggregation of organic dyes. In work [14], the formation of compounds between Jaggregates and quantum dots obtained by combining positively or negatively charged dye aggregates and oppositely charged quantum dots was considered. This effect stems from the electrostatic interaction between dye molecules and nanocrystals.

On the other hand, the interaction between identically charged particles has not been studied, because they are supposed to be unable to combine into a hybrid aggregate owing to the electrostatic repulsion between them. In this work, an attempt was made to obtain the dye J-aggregation on CdTe quantum

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dots, the surface charge of which had the same sign as the dye molecule had.

2. Experimental Part

2.1. Sample preparation

Nanocrystals of CdTe in an aqueous solution were obtained according to the following technique. Synthesis was carried out due to the interaction of CdI_2 with H_2 Te. The latter was obtained by electrochemically reducing tellurium on a tellurium electrode in the presence of thioglycolic acid (TGA). The resulting solution contained CdTe particles with size from 2 to 10 nm. Then the first-fraction floc was separated on a centrifuge.

Precipitated flocs of the separated fraction were peptized in deionized water. Addition of water to the separated precipitate was found [15] to result in the redispergation and the formation of strong colloid solutions containing quantum dots with a narrow distribution over their dimensions. The floc peptized in water was characterized by pH = 7.0. The size of CdTe nanoparticles was determined from the relation between the particle diameter D and the wavelength of the first excitonic absorption maximum [16]. According to the data obtained, D = 3 nm and the CdTe concentration $c = 8.5 \times 10^{-6}$ M.

We chose thiamonomethinecyanines with nonsymmetric structures (Fig. 1) to study. All the dyes were synthesized following the standard technique [17], by condensing 3-{2-[(3-sulfopropyl)thio]-1,3benzothiazol-3-yl-3}propane-1-sulfonate with ternary salts of 3-[2,5-dimethyl-6-methoxy-(1,3-benzothiazol-3-yl-3]propane-1-sulfonate, 3-[5-methoxy-2-methyl(thien-[2,3-e][1,3]benzothiazol-3-yl-3)]propane-1-



Fig. 2. Extinction of dye **1** normalized to 1 mol of the substances for various dye concentrations: (1) 4×10^{-7} , (2) 9.1×10^{-7} , (3) 1×10^{-5} , (4) 3.15×10^{-5} , (5) $6.21 \times \times 10^{-5}$, and (6) 6×10^{-3} M, (7) 1.09×10^{-4} M with water as a solvent, (8) 1×10^{-4} M in the electrolyte solution, and (9) 1×10^{-5} M in DMF

sulfonate or 3-[2-methyl-(benzothien[2,3-d]thiazol-3-yl-3)]propane-1-sulfonate (below, they will be referred to as dye 1, 2, and 3, respectively). The dye structures were confirmed by NMR spectra and the data of element analysis.

Initial aqueous solutions of dyes were prepared in a volume of 1.5 ml and with a concentration of 10^{-3} mol/l. Aqueous mixtures of dyes and nanoparticles were prepared as follows: first, the aqueous solution of dye was added in a cuvette with water; then a colloid solution of CdTe nanocrystals was added to the solution obtained. In order to compare the behavior of spectra, a solution of CdTe was separately added in a cuvette with pure water. To study the behavior of nanocrystals and dyes in the 0.1-M aqueous solution of KCl, the prepared solution was poured into a cuvette, and the dyes and CdTe nanoparticles were added in the same proportion as when into the aqueous solution without KCl. In the course of this process, the concentration of KCl in the solution decreased from 0.1 M to 0.06 M (in what follows, we will use the average value of concentration, i.e. 0.075 M). When carrying out the cycle of experiments with the same dye, the same cuvette was used for the preparation of solutions.

Composite films were prepared from final solutions by drop-cast of glass plates with the solutions with the use of a dozer.

2.2. Experimental technique

The absorption and photoluminescence spectra were measured on an Avantes-2048 spectrophotometer. We used a diode-pumped solid-state (DPSS) laser with a wavelength of emitted light of 408 nm and a power of 150 mW as an excitation source for luminescence spectrum measurements and a halogen lump Ocean Optics LS-1 for absorption spectrum measurements.

The film morphology was studied in polarized and non-polarized light on a biological microscope of the research class XY-B2. When carrying out polarizedlight researches, the polarizer was set between the light source and the sample, and the analyzer was mounted on the microscope eyepiece.

3. Results and Discussion

3.1. Spectral manifestations of the dye aggregation in aqueous solutions

Dye monomers in the diluted dimethylformamide (DMF) solution show an absorption band at 440 nm (Fig. 2, curve 9). The analogous band shifted by 5 nm to the short-wave side due to the solvatochromic effect is observed in the diluted aqueous solution (Fig. 2, curve 1). We found that dye 1 can form associates of two types in aqueous solutions depending on the concentration. First, the absorption band observed at about 435 nm and belonging to the monomer in the diluted solution gradually transforms into a shortwave band at about 415 nm, which testifies to the formation of dye dimers (Fig. 2). Similar regularities were detected for dyes 2 and 3, but their absorption spectra brought us to the conclusion that molecules of dye **2** do not form dimers in water so effectively as those of dyes 1 and 3.

Second, a further increase in the dye concentration gives rise to a reduction of the molar extinction coefficient and a broadening of the absorption band, which testifies to the formation of associates that are larger than dimers (Fig. 2, curves 5 and 6). On the other hand, we observed no pronounced manifestations of the dye J-aggregation in the absorption spectra of aqueous solutions.

When the same dye concentration of 10^{-4} M was introduced into the electrolyte solution (Fig. 2, curve 8), a decrease in the intensity of the dimer band and the appearance of a new peak at 475 nm were observed. It is worth noting that, for the aqueous solution with the same concentration, the spectrum did

not reveal an extinction growth in the given spectral interval (curve 7).

The photoluminescence spectra of the aqueous solutions of all dyes have one strongly pronounced peak. When the dye concentration increases, the band neither shifts toward short waves nor gets narrower. At first sight, this fact testifies to the absence of the formation of J-aggregates of those dyes in water. However, it has to be noticed that the spectrum of dye **1** (Fig. 3, a, curves 1 and 2) differs from those of other dyes by an appreciable asymmetry of the photoluminescence band. By decomposing this curve into Lorentzians, we obtain three peaks with the maxima at 488, 511, and 536 nm (Fig. 3, b). The wavelength of the short-wave band, $\lambda = 488$ nm, and its halfwidth, $\Delta = 20$ nm, correspond to the photoluminescence band wavelength, $\lambda = 483$ nm, and halfwidth, $\Delta = 19$ nm, of the first Lorentzian peak for the same dye in the electrolyte (see below), which, in turn, corresponds to the luminescence of Jaggregates. The contribution of J-aggregates in the aqueous dye solution can be approximately evaluated as the ratio between the areas under the photoluminescence Lorentzian curves corresponding to the photoluminescence by J-aggregates and the total photoluminescence by monomers and dimers (Fig. 3, b); the corresponding value turned out to be about 1/3. In addition, we should take into account the ratio between the quantum yields of J-aggregates in the electrolyte solution and monomers and dimers in the aqueous solution at the same concentration of dye; the corresponding ratio is about 2/1. According to those data, no more than 15% of the molecules in this solution (Fig. 3, b) compose J-aggregates.

3.2. Interaction between dyes and CdTe nanocrystals in aqueous solutions

The addition of a CdTe dispersion into the aqueous solution of dye **1** did not give rise to any qualitative changes in the dye absorption spectra. From Fig. 4, a, one can see that the absorption spectra of CdTe with the dye are simple superpositions of the spectra of separate components in the solution, and no formation of J-aggregates was observed. There was no shift of the emission band in the photoluminescence spectra as well (Fig. 4, b), whereas a relatively lower intensity of the CdTe photoluminescence in the presence of dye in comparison with the photoluminescence of a pure CdTe dispersion was associated with the ab-

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Fig. 3. Photoluminescence spectra of dyes 1, 2, and 3 with concentrations of 3.15×10^{-5} M (curves 1, 3, and 5, respectively) and 10^{-5} M (curves 2, 4, and 6, respectively) dissolved in water (a). Decomposition of the photoluminescence spectrum of dye 1 with a concentration of 3.15×10^{-5} M into Lorentzian components 1', 1", and 1''' (b)

sorption by dye molecules of radiation emitted by the excitation source, because this absorption occurred at the laser wavelength (Fig. 4, a). Consequently, the effective intensity of the CdTe dispersion excitation was lower than that in the absence of dye. The behavior of absorption and luminescence spectra of two other dyes in water has no essential difference from those demonstrated above; therefore, we exhibited the results obtained only for dye **1**.

Dye monomers and CdTe nanocrystals have the same charge and repulse from one another owing to



Fig. 4. Absorption (a) and photoluminescence (b) spectra of dye 1 in the aqueous CdTe dispersion with a concentration of about 1.1×10^{-6} M (1); spectra of dye 1 in the aqueous CdTe dispersions (solid curves) and of pure CdTe dispersions (dashed curves) for various CdTe concentrations: 9.3×10^{-8} (2), 2.7×10^{-7} (3), and 4.4×10^{-7} M (4)

the electrostatic interaction. Therefore, the formation of dye J-aggregates on the quantum dot surface was expected not to take place, which was confirmed by the photoluminescence spectra (Fig. 4, b). The resulting dye luminescence curve reduced its intensity when the CdTe dispersion was added, but its profile did not change, i.e. monomers did not transform into associates. The quenching of the dye luminescence was not associated with the energy transfer to nanocrystals, because no increase in the CdTe photoluminescence was observed. This conclusion agrees with the fact that the dye molecules and the CdTe particles have the same charge sign and repel one another, which does not allow them to approach closer to one another at a distance of a few nanometers, which is necessary for the energy transfer following the Förster mechanism [18] to take place. From the additional spectrum (Fig. 5, a), where the solid and short-dashed curves demonstrate the absorption spectrum of CdTe with concentrations of 5×10^{-6} and 8.5×10^{-6} M, respectively, one can see that CdTe nanocrystals are responsible for two absorption peaks, at 415 and 530 nm. The latter peak is not pronounced well at a concentration of 2.6×10^{-6} M, but its presence at the previous spectra gives us ground to assume that here we observe the overlapping between the donor luminescence and acceptor absorption bands.

3.3. Interaction between dyes and CdTe nanocrystals in the electrolyte solution

Dissolution of dye 1 in the electrolyte results in the appearance of a narrow absorption band at λ = = 471 nm shifted with respect to the monomer absorption band toward the red spectral side (Fig. 5), which testifies to the formation of J-aggregates. It was found that, by adding the CdTe dispersion into the dye solution in the electrolyte, we can affect the reconstruction of dye associates, which depends, in turn, on the structure of the dye itself. For instance, the addition of a CdTe dispersion into the solution of dye 1 did not lead to considerable changes in the absorption spectra. From Fig. 5, a, one can see that the absorption spectra of CdTe with the dye are almost superpositions of the spectra of separate components in the solution. Therefore, it is impossible to draw conclusion concerning the influence of CdTe on the dye on the basis of those data. However, the photoluminescence spectra of dye in the presence of CdTe reveal an enhancement of the photoluminescence intensity from J-aggregates (Fig. 5, b, curve 4).

The difference between the changes in the absorption and luminescence spectra can be explained by the fact that the quantum luminescence yield of J-aggregates is considerably larger than that of dye monomers, whereas the extinction coefficients of monomers and J-aggregates are close to each other (Fig. 6). Therefore, the effects of the J-aggregate reconstruction will be better appreciable in the luminescence spectra, being almost invisible in the absorption ones. Hence, a tendency for the number of



Fig. 5. Absorption (a) and photoluminescence (b) spectra obtained when the CdTe dispersion is added to the solution of dye 1 in the electrolyte: mixture of CdTe $(2.6 \times 10^{-6} \text{ M})$ and the dye (10^{-4} M) (1), CdTe $(2.6 \times 10^{-6} \text{ M})$ in the absence of dye (2), the dye (10^{-4} M) in the absence of CdTe (3), and the difference between spectra 1 and 2 (4). The decomposition of the dye photoluminescence curve into Lorentzian components is shown. The cuvette thickness is 1 mm. The insert in (a) shows the absorption spectra of the CdTe dispersion of concentrations of $5 \times 10^{-6} \text{ M}$ (dashed curve) and $8.5 \times 10^{-6} \text{ M}$ (solid curve)

J-aggregates of dye-1 molecules to grow under the influence of CdTe can be identified. However, as was in the case of monomers, the interaction between the J-aggregates of dye and CdTe does not result in the effects associated with the energy transfer.

A more distinct picture of the influence of CdTe particles on the dye is observed in the case of dye **2**.

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Fig. 6. Absorption (a) and photoluminescence (b) spectra of dye **1** with a concentration of about 10^{-4} M in the aqueous solution (1) and the aqueous solution of KCl electrolyte with a concentration of about 0.1 M (2). The cuvette thickness is 1 mm

The corresponding absorption spectrum of the dye in the electrolyte solution has three peaks at wavelengths of 426, 453, and 475 nm (Fig. 7, a). The first and second peaks correspond to the absorption by dye dimers and monomers, respectively. The maximum positions for those bands coincide with the corresponding bands for monomers and dimers obtained in the aqueous solutions (Fig. 2). In contrast to the aqueous solution case, the spectra in the electrolyte have an additional band shifted toward the red spectral side at 475 nm. This band corresponds to the formation of J-aggregates. It should be noted that the role of an electrolyte in the formation of J-aggregates consists in that electrolyte ions of the corresponding sign are capable of stabilizing the structure of a chain of charged chromophores.

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Fig. 7. Absorption (a) and photoluminescence (b) spectra of dye **2** with a concentration of about 10^{-4} M in the aqueous solution of KCl electrolyte (1) and the mixture of this solution with the CdTe dispersion with a concentration of 2.6×10^{-6} (2) and 1.5×10^{-6} M (2'). Curves 3 and 3' exhibit the corresponding spectra of CdTe in the absence of dye. Curve 4 is the difference between spectra 2 and 3

When CdTe is added to the solution of dye 2, an appreciable increase in the intensity of the absorption band of J-aggregates is observed, which is certainly testifies to their intensive formation owing to CdTe quantum dots. Simultaneously, we observed a gradual precipitation in the cuvette owing to the formation of an insoluble fraction composed of the mixture components. From the difference between spectra 2 and 3 in Fig. 7, a, one can see that the curve values become negative, because the CdTe absorption in the mixture with the dye diminishes. This effect is caused by the precipitation in the cuvette, because some part of the material is removed from the measurement zone, when CdTe particles and the dye coagulate.

When CdTe is added, the dye photoluminescence peak shifts toward the short-wave region from 530 to 502 nm and changes its shape by becoming narrower (Fig. 7, b), which confirms the conclusion made above concerning an enhancement of the J-aggregate formation in the presence of CdTe nanocrystals. It should be noted that the luminescence spectrum of CdTe in the presence of dye also changes and becomes less intense (Fig. 7, b). This fact cannot be explained as the dye absorption of light used for the excitation of the nanocrystal luminescence at 408 nm, because J-aggregates absorb insignificantly in this range (Fig. 7, a). A possible mechanism of nanocrystal luminescence quenching may consist in a nonradiative relaxation of the excited electron owing to the charge transfer from the CdTe surface to a J-aggregate. It should be noted that, in this case, a direct contact between a nanocrystal and dye molecules is necessary.

The change of the band shape in the dye luminescence and absorption spectra when nanocrystals are added, as well as the precipitation of the mixture, provides extra facts in favor of the interaction between dye molecules and nanoparticles. The same sign of the electric charges of components (dve molecules and CdTe nanocrystals) testifies that the interaction is not electrostatic. In our opinion, the formation of J-aggregates with the help of electrolyte ions is a crucial condition for the interaction. The aggregate is capable of being adsorbed on the nanocrystal surface, so that the equilibrium between free aggregates and aggregates bound with nanoparticles in the solution changes. The formation of bound aggregates stimulates the appearance of new J-aggregates from monomers and dimers in the solution, which are adsorbed again on the CdTe surface. A hybrid particle CdTe/dye J-aggregate is insoluble and precipitates. Another proof of a specific character of the composite material formation is given by the film structure.

3.4. Formation of composite films

Deposition of dye molecules from the solution in the film naturally leads to the association and the ag-

gregate formation. Even in the films created from aqueous solutions of dyes, the J-aggregation of their molecules is observed. Most clearly, this process manifests itself in the film of dye **3** (Fig. 8, a), where the aggregates form a chain-like structure. This chain can be observed with the use of polarization microscopy (Fig. 8, b), which means that such a structure rotates the light polarization angle and, hence, is a crystalline one.

The absorption spectra of film **3** (Fig. 9) demonstrate a narrow band with a maximum at 487 nm, which is inherent to J-aggregates. This fact evidences the formation of those aggregates in the film. One can see that the peaks of monomers and dimers are almost absent in the absorption spectrum, which gives us ground to assume that the photos exhibit a chain of J-aggregates that have crystallized on the glass surface. On the other hand, the morphology of films **1** and **2** obtained from the aqueous solutions (not shown) and their absorption spectra (Fig. 9) testify that the tendency to the formation of J-aggregates is less pronounced for those specimens.

The addition of the CdTe dispersion and the formation of a hybrid structure change the film morphology. While comparing Figs. 8, a and c, one can see a substantial difference between the morphologies of those films; namely, CdTe nanocrystals in the mixture with dye **3** create a well-discernible branching structure. A similar morphology was observed for hybrid films on the basis of two other dyes.

To elucidate the formation mechanism for those structures, we calculated the fractal dimensionalities for the structures exhibited in Fig. 8, c with the use of the method reported in work [19]. For this purpose, the photo was divided into a grid of cells, each of them was considered to be either filled or empty, and the number of filled cells was counted. The fractal dimensionality was calculated according to the formula

$$D = \frac{\ln N_1}{\ln(L/l_0)},$$

where N_1 is the number of filled cells, L the object size measured on the photo, and l the cell size. As a result, we obtained $D \approx 1.76$ for dye **2** and $D \approx 1.74$ for dye **3**. According to the results of work [19], these values correspond to the clustercluster aggregation (CCA) model; i.e., the initial particles form firstly clusters; then, the latter stick to-







Fig. 8. Images of (a and b) a dye-3 film obtained from the aqueous solution and (c) a hybrid film dye 3–CdTe nanocrystals from the electrolyte solution. The magnification is $\Gamma = 200$. Images (a) and (c) were obtained in non-polarized light and (b) in crossed polarizers



Fig. 9. Absorption spectra of the dye films obtained from aqueous solutions 1(1), 2(2), and 3(3)

gether. This model corresponds to the conclusion made above that the J-aggregates are adsorbed on the surface of CdTe nanoparticles. The latter form hybrid clusters, which precipitate in the form of a fractal structure.

4. Conclusions

On the basis of the results obtained, while studying the behavior of thiamonomethinecyanine dyes of three types in the aqueous solution, electrolyte solution, and films, their different abilities to form J-aggregates, which depend on the dye structure, are demonstrated. The absence of end groups in the phenolic ring of molecule **3** results in a more pronounced formation of J-aggregates in films. At the same time, the other two dyes reveal a better ability to form J-aggregates in the electrolyte solution. For all types of molecules, the formation of J-aggregates occurs through the dimerization as an intermediate stage.

The dyes revealed also different abilities to interact with CdTe nanocrystals. In the aqueous solutions, where J-aggregates are not formed yet, the forces of electrostatic repulsion between identically charged dye molecules and CdTe particles do not result in the formation of any hybrid structures. In the electrolyte solution, neutral J-aggregates can be adsorbed on the CdTe surface and, in such a way, they can form a hybrid structure, which precipitates. The equilibrium shift in the solution owing to the removal of hybrid particles leads to the further formation of J-aggregates from molecules in the solution. Dye **3** demonstrated the most effective formation of J-aggregates in the presence of CdTe particles. Therefore, the presence of end groups in the phenolic ring of a thiamononethynecyanine molecule turns out to be rather a crucial condition for the formation of J-aggregates in both the presence and absence of extraneous particles.

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В.Ю. Петренко, Ю.Л. Сломінський, Г.Л. Смирнова, І.А. Мазарчук, О.П. Дімітріев ВПЛИВ НАНОЧАСТИНОК Сdте НА УТВОРЕННЯ Ј-АГРЕГАТІВ ТІАМОНОМЕТИНЦІАНІНОВИХ БАРВНИКІВ

Резюме

Досліджено умови виникнення J-агрегатів для трьох типів тіамонометинціанінових барвників, структура яких відрізняється кінцевими групами, в залежності від їх концентрації та типу взаємодії з наночастинками CdTe розміром ~3 нм у водних дисперсіях. Виявлено вплив структури барвника на ефективність утворення J-агрегатів у розчинах та у плівках. Виявлено, що квантові точки (КТ) CdTe, стабілізовані тіогліколієвою кислотою, здатні адсорбувати Jагрегати барвника на своїй поверхні. Вперше показано, що гібридна структура барвник–CdTe може утворюватись за рахунок взаємодії від'ємно заряджених молекул барвника з від'ємно зарядженою поверхнею КТ через утворення нейтральних агрегатів. Не виявлено процесу переносу енергії із барвника на частинки CdTe ні для системи димер–CdTe, ні для системи J-агрегат–CdTe.