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UDC:

Investigation of the “self-desensitization” processes in anionic dye by means of the luminescence method

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The luminescence-based technique is proposed that permits to control the interaction processes of molecular and polymolecular forms of the anionic dye AgHal emulsions, responsible for the self-desensitization. It is found that the characteristic feature of the I-kind self-desensitization is the molecular dye phosphorescence occurring upon the immediate transfer of the non-equilibrium electrons and holes from the excited J-aggregate to the molecular dye. The characteristic feature of the II-kind self-desensitization is the anomalously retarded anti-Stokes fluorescence of the molecular dye associated with the transfer of the non-equilibrium electrons from the excited J-aggregate to silver in the atomic-molecular degree of dispersion, part of which can afterwards be transferred to the molecular dye and recombine there with the localized non-equilibrium holes.

Additional infra-red illumination enabled us to show experimentally that the anomalously retarded anti-Stokes luminescence of the molecular dye and the phosphorescence of the molecular dye upon excitation of the dye J-aggregates, take place in different dye molecules and are determined by different electron-hole processes and different interactions of the molecules with the dye J-aggregates.

Keywords: luminescence; dye; aggregate; self-desensitization.

У роботі запропонована люмінесцентна методика, що дозволяє контролювати процеси взаємодії молекулярних та полімолекулярних форм аніонного барвника у AgHal емульсіях, які відповідальні за самодесенсибілізацію.

З'ясовано, що особливість самодесенсибілізації I-роду полягає у фосфоресценції молекулярного барвника, яка відбувається при безпосередньому передаванні нерівноважних електронів та дірок від збудженого J-агрегату молекулярному барвнику. Особливість самодесенсибілізації II-роду є антистоксова аномально сповільнена флуоресценція молекулярного барвника, яка пов'язана з передаванням нерівноважних електронів від збудженого J-агрегату барвника срібла атомно-молекулярної дисперсності, частина з яких потім може передаватися молекулярному барвнику та рекомбінувати там з локалізованими нерівноважними дірками.

Додаткове інфрачервоне світло дозволило експериментально показати, що антистоксова аномально сповільнена флуоресценція молекулярного барвника та фосфоресценція молекулярного барвника при збудженні J-агрегатів барвника, мають відношення до різних молекул барвника та визначаються різними електронно-дірковими процесами та різною взаємодією молекул з J-агрегатами барвника.

Ключові слова: люмінесценція; барвник; агрегат; самодесенсибілізація.

В работе предложена люминесцентная методика, позволяющая контролировать процессы взаимодействия молекулярных и полимолекулярных форм анионного красителя в AgHal эмульсиях, ответственные за самодесенсибилизацию.

Выяснено, что отличительной особенностью самодесенсибилизации I-рода является фосфоресценция молекулярного красителя, которая происходит при непосредственной передаче неравновесных электронов и дырок от возбужденного J-агрегата молекулярному красителю. Отличительной особенностью самодесенсибилизации II-рода является антистоксовая аномально замедленная флуоресценция молекулярного красителя, которая связана с передачей неравновесных электронов от возбужденного J-агрегата красителя серебра атомно-молекулярной степени дисперсности, часть из которых затем может передаваться молекулярному красителю и рекомбинировать там с локализованными неравновесными дырками.

Дополнительная инфракрасная подсветка позволила экспериментально показать, что антистоксовая аномально замедленная флуоресценция молекулярного красителя и фосфоресценция молекулярного красителя при возбуждении J-агрегатов красителя, относятся к разным молекулам красителя и определяются разными электронно-дырочными процессами и различным взаимодействием молекул с J-агрегатами красителя.

Ключевые слова: люминесценция; краситель; агрегат; самозапускаемость.

Introduction

Molecules and aggregates of molecules of a dye efficiently absorb the light radiation of various spectral ranges and can transfer this excitation to various optically active compounds, for example, to silver halides, which is widely used for their spectral sensitization, or to the titanium-dioxide quantum dots, being important components of the photochemical cell optoelectronic devices [1].

In all such processes, the excitation transfer from the dye is determined by the transmission efficiency of the photoexcited electrons and holes to the active centers [2]. However, not all the electrons and holes photoexcited in the dye reach these centers; a part of them can be lost due to recombination in the dye itself – a so called self-desensitization effect [3].

For the photographic emulsions, in [4-6] the two types of self-desensitization were defined:

- if an electron and a hole of the photoexcited dye recombine inside the dye, without getting outside its boundaries, such a process leads to the self-desensitization of the I kind;

- otherwise, an electron of the photoexcited dye is delivered to the active center while a hole remains within the dye thus forming the cation-radical of the dye (Dy^+). After this, Dy^+ traps an electron of the active center which, in turn, either due to relaxation or in the course of recombination, transits to the ground non-excited state. This process of the electron-hole recombination is most probable for the J-aggregated dyes and is called self-desensitization of the II kind.

Accordingly, in order to evaluate and to control the efficiency of the dye utilization in all branches of its application, one needs to recognize the processes of interaction of J-aggregated and molecular dye forms which induce the self-desensitization processes [7] in the studied system and thus decrease the dye efficiency. For the silver halides this is a hard task when based exclusively on the spectral sensitometric data [4]; that is why in this work we study possibilities of the luminescence technique that would primarily allow to control the processes of interaction between the molecular and polymolecular forms of the dye, responsible for the self-desensitization.

Experimental results

The luminescence measurements of the samples were conducted at $T = 77$ K with the experimental setup enabled registration of the luminescence spectra in the following regime. The excitement lasts $\sim 10^{-4}$ s, then the “dark” interval of duration $\sim 10^{-3}$ s takes place, and only afterwards the luminescence registration starts and continues for $\sim 10^{-4}$ s. This cycle repeats periodically with frequency 800 Hz.

This mode of registration permits to exclude the contribution of fluorescence (decay time $\tau < 10^{-3}$ s), and to

register only the phosphorescence and anomalously retarded fluorescence (ARF) ($\tau > 10^{-3}$) [8], which are generally responsible for the self-desensitization processes. To clarify the role of the non-equilibrium charge-carriers' localization in the luminescence process, we employ the additional illumination by the IR light, obtained from non-modulated radiation of the KGM lamp (50 W) transmitted through a 2-mm thick germanium light filter ($\lambda_{IR} > 1000$ nm).

As an object of the luminescence study, we choose the homogeneous emulsion containing cubic microcrystals (MC) AgBrI (3% AgI) with the mean size $d \approx 0.24$ μm , formed in the gelatin medium by means of the two-stream emulsification during which concentration of the bromine or silver ions in the course of synthesis was kept constant within the limits pBr 2 to pBr 7.5 and was controlled by the ionometer [9]. In the fabricated emulsion, the content of the silver and bromine ions in the solution was correctable by adding solutions of KBr or AgNO_3 .

The spectral sensitization of the emulsion was performed by the anionic panchromatic J-aggregating dye – 3,3'-di- γ -sulfopropyl-9-ethyl-4,5,4',5'-dibenzothiacarbocyaninebetaine pyridinium salt (Dy in further references).

In Fig. 1 the spectra are presented of the low-temperature ($T = 77$ K) luminescence and of the luminescence excitation for the AgBrI emulsion (pBr 2) upon the introduced dye concentration $C_{Kp} = 0$, as well as $C_{Dy} = 10^{-5}$ mole Dy/mole AgBr. For the sample without dye ($C_{Dy} = 0$), the single luminescence band is observed in the green spectral segment with $\lambda_{max} = 540\text{--}570$ nm, stipulated by presence of the twin

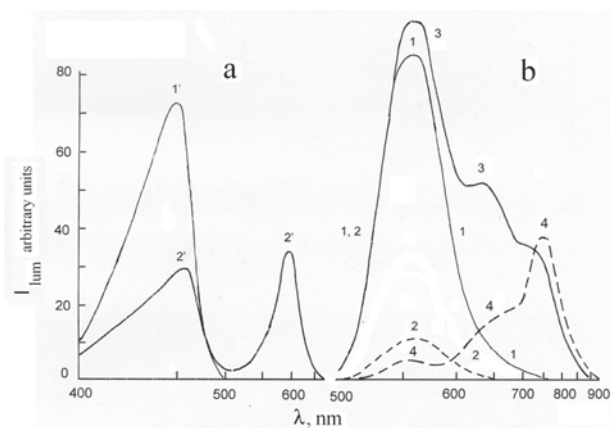


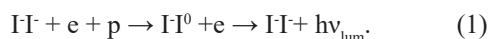
Fig. 1. Spectra of (a) excitation and (b) luminescence at $T = 77$ K for the emulsion AgBrI (3% AgI) with pBr 2 for the introduced dye concentration: $C_{Dy} = 0$ – curves 1', 1, 2-dashed; $C_{Dy} = 10^{-5}$ (mole Dy/mole AgBr) – curves 2', 3, 4-dashed.

(a) The excitation spectra are recorded for the luminescence bands with the wavelength λ : 1' – 560 nm; 2' – 750 nm.

(b) The luminescence spectra are recorded for the excitation with wavelength λ : 1, 2, 3, 4 – 450 nm; dashed 2 and 4 – in conditions of additional IR illumination with $\lambda > 1000$ nm.

iodine centers I-I⁻ localized at the surfaces of the AgBrI MC [10,11] (Fig. 1b, curve 1); excitation of this band is characterized by the single maximum at $\lambda_{\max} = 450-470$ nm and corresponds to the intrinsic absorption edge of AgBrI MC (Fig. 1a, curve 1'). Upon the IR action, emission in the green range with $\lambda_{\max} = 540-570$ nm shows the decrease of intensity – the luminescence quenching (Fig. 1b, dashed curve 2).

Emergence of the green luminescence is described by the following scheme. Initially, under the action of monochromatic light with $\lambda = 450$ nm, due to absorption of photons with energy $h\nu_{\text{exc}}$, free electrons and holes are generated, correspondingly, in the conduction band (CB) and in the valence band of the AgBrI MC: $\text{AgBrI} + h\nu_{\text{exc}} \rightarrow e + p$. Further, the holes are trapped by the I-I⁻ centers consisting of the dopant iodine ions situated on the AgBrI MC surface in the adjacent lattice sites [10,11]. After this, the electrons from the CB recombine with the holes localized by the iodine twin centers with emission of a quantum of the green luminescence $h\nu_{\text{lum}} = 540-570$ nm:

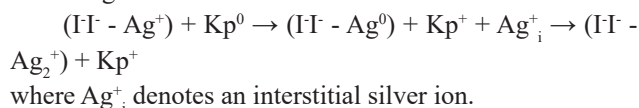


The lifetime of electrons in the CB and, respectively, duration of the green-band phosphorescence is determined by the concentration of the interstitial silver ions Ag_i^+ , which act as shallow traps for the photoelectrons [12].

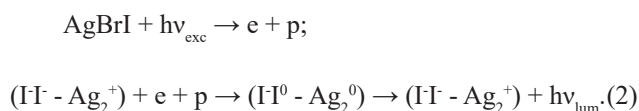
In the luminescence spectrum of the emulsion with introduced dye ($C_{\text{Dy}} = 10^{-5}$ mole Dy/mole AgBr, pBr 2) obtained when the edge of intrinsic absorption of the AgBrI MC is excited by monochromatic light with $\lambda = 450$ nm, along with the green emission band of the twin iodine centers with $\lambda_{\max} = 540-570$ nm, the two additional bands are observed with $\lambda_{\max} = 640$ nm and $\lambda_{\max} = 750$ nm (Fig. 1b, curve 3).

According to references [13,14], the luminescence band with $\lambda_{\max} = 640$ nm is associated with the ARF of the dye adsorbed on the surface of the AgBrI MC in the molecular state (M_{ARF}).

Following to our data [15-17], the luminescence band with $\lambda_{\max} = 750$ nm is related with the presence of (I-I⁻ - Ag_2^+) centers in the AgBrI MC, whose formation, upon adsorption of the dye on the MC surface, is realized according to the scheme:



In this case, for excitation by light from the intrinsic absorption range of AgBrI MC, origination of the luminescence with $\lambda_{\max} = 750$ nm can be described by the scheme:



For the green-band luminescence, at the given dye concentration, the excitation is characterized, as before, by a single maximum in the excitation spectrum with $\lambda_{\max} = 450-470$ nm. In the excitation spectrum of the luminescence with $\lambda_{\max} = 750$ nm, an additional band appears with $\lambda_{\max} = 600$ nm (Fig. 1a, curve 2'), that corresponds to the molecular dye absorption range. Upon the sample excitation by the monochromatic light ($\lambda = 450$ nm) and under the action of additional IR illumination ($\lambda > 1000$ nm), the luminescence bands with $\lambda_{\max} = 550$ and 640 nm experience the decay of the emission intensity – the luminescence quenching, whereas the band of $\lambda_{\max} = 750$ nm shows noticeable intensity increase – the luminescence enhancement (Fig. 1b, dashed curve 4).

In our opinion, the fact that IR illumination contributes to the emission increase in this luminescence band, testifies that the emission of the (I-I⁻ - Ag_2^+)-centers with $\lambda_{\max} = 750$ nm is determined by recombination along the scheme (2). According to the mechanism proposed, the luminescence of the (I-I⁻ - Ag_2^+) centers can increase due to the IR illumination, which provides the observed luminescence enhancement; at the same time, in the luminescence band with $\lambda_{\max} = 550-560$ nm, emerging from the recombination of the holes localized in the twin iodine centers at the MC surface with the free electrons in the conduction band (scheme (1)), the luminescence quenching will be observed.

With further growth of concentration of the dye introduced into the emulsion ($C_{\text{Dy}} = 10^{-4}$ mole Dy/mole AgBr) and pBr 2, excitation of the green luminescence associated with the twin iodine centers $\lambda_{\max} = 540-570$ nm (Fig. 2a, curve 1') is no longer characterized by a single maximum but shows the two ones at $\lambda = 450-470$ nm and at $\lambda = 670-690$ nm. The maximum of $\lambda = 670-690$ nm was never observed previously, and it corresponds to the absorption band of the polymolecular dye associations, so called J-aggregates of the dye [19].

Upon excitation of the absorption band of the J-aggregated dye by monochromatic light with $\lambda = 670$ nm, in the luminescence spectrum (Fig. 2b, curve 3), four emission bands are observed: (i) The anti-Stokes emission band of the twin iodine centers at $\lambda_{\max} = 540-570$ nm; (ii) ARF of the molecular dye at $\lambda_{\max} = 610-640$ nm that was first observed by us [20]; (iii) The luminescence band caused by the presence of (I-I⁻ - Ag_2^+) centers with $\lambda_{\max} = 750$ nm, and (iv) the band with $\lambda_{\max} = 800$ nm.

For the first time, the emission band of $\lambda_{\max} = 800$ nm excited by light from the absorption range of the dye J-aggregate was observed in [20]. The spectral position of this band coincides with the maximum of the molecular dye phosphorescence in gelatin, upon its excitation from the molecular dye absorption band with $\lambda_{\max} = 600$ nm. On the other hand, it is known [21] that the dye in the J-state in gelatin, as well as the molecular dye adsorbed at the

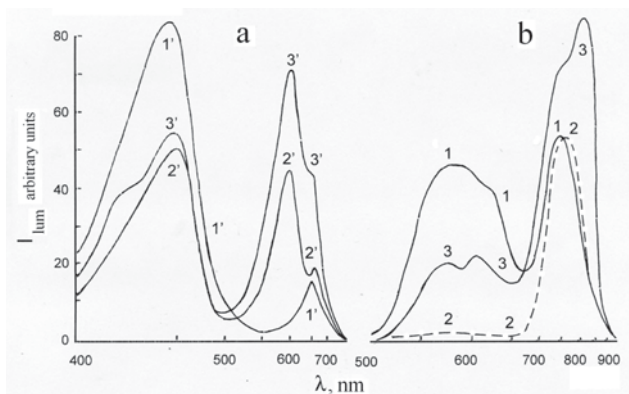


Fig. 2. Spectra of excitation (a) and of luminescence (b) at $T=77$ K of the emulsion AgBrI (3% AgI), pBr 2 with the introduced dye concentration $C_{Dy}=10^{-4}$ (mole Dy/mole AgBr)

(a) The excitation spectra are recorded for the luminescence bands with λ : 1' – 560 nm; 2' – 750 nm; 3' – 800 nm.

(b) The luminescence spectra are recorded for the excitation by the light with wavelength λ : 1, dashed 2 – 450 nm; 3 – 670 nm, dashed 2 – additional IR illumination with $\lambda > 1000$ nm.

surface of MC AgHal, show no phosphorescence. This fact permitted us to conclude [20] that this band is associated with the molecular dye phosphorescence (M_{ph}).

In Fig. 3, spectra of excitation and of the low-temperature luminescence are presented for the emulsion containing the dye $C_{Dy} = 5 \cdot 10^{-4}$ (mole Dy/mole AgBr), in which the content of the silver ions was changed to pBr 7.5. Upon excitation from the absorption range of the silver halide ($\lambda_{max} = 450-460$ nm), the emission spectrum contains the emission band of the twin iodine centers $\lambda_{max} = 560-570$ nm and the band corresponding to luminescence of the $(I\bar{I} - Ag_2^+)$ centers $\lambda_{max} = 750$ nm (Fig. 3b, curve 1), whose emission is efficiently excited from the absorption band of the silver halide MC ($\lambda_{max} = 450-460$ nm) and from the absorption range of the J-aggregated dye $\lambda_{max} = 690$ nm (Fig. 3a, curve 1'). Under the action of the additional long-wave light $\lambda_{IR} > 1000$ nm, in the excitation spectrum there occurs quenching of the green Stokes luminescence of the twin iodine centers $\lambda_{max} = 450-460$ nm, equally as the quenching of the anti-Stokes luminescence $\lambda_{max} = 690$ nm (Fig. 3a, dashed curve 2').

Upon exciting from the absorption range of the J-aggregated dye ($\lambda_{max} = 670-690$ nm), the luminescence spectrum of the emulsion sample consists of the emission band of the $(I\bar{I} - Ag_2^+)$ - centers ($\lambda_{max} = 750$ nm) and the overlapping band of the molecular dye phosphorescence ($\lambda_{max} = 800$ nm); because of the strong overlapping, the common maximum of those bands appears at $\lambda = 780$ nm. It should be emphasized that the anti-Stokes ARF of the molecular dye at $\lambda_{max} = 620-640$ nm is not observed in this case.

The excitation spectrum of the molecular dye phosphorescence with $\lambda_{max} = 800$ nm (Fig. 3a, curve 3')

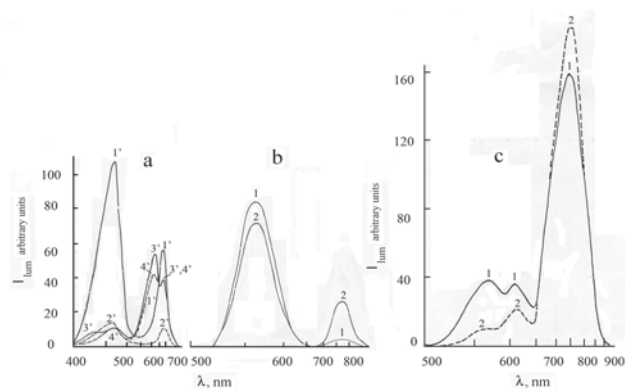


Fig. 3. Spectra of excitation (a) and of luminescence (b, c) at $T=77$ K of the emulsion sample AgBrI (3% AgI), pBr 7.5 with the introduced dye concentration $C_{Dy} = 5 \cdot 10^{-4}$ (mole Dy/mole AgBr). (c) Luminescence spectra of the emulsion AgBrI (3% AgI) and pBr 4 with the introduced dye concentration $C_{Dy} = 5 \cdot 10^{-4}$ (mole Dy/mole AgBr) and with the additionally introduced supersensitizer SS $C_{SS} = 6 \cdot 10^{-3}$ (mole SS/mole AgBr).

(a) The excitation spectra are recorded for the luminescence bands with wavelengths λ : 1', 2' – 560 nm; 3', 4' – 800 nm. The excitation spectra 2', 4' were obtained under additional IR illumination with $\lambda > 1000$ nm

(b) The luminescence spectrum is recorded upon excitation by the monochromatic light with the wavelength λ : 1' – 450 nm; 2' – 690 nm.

(c) The luminescence spectrum is recorded upon excitation by light with the wavelength $\lambda = 670$ nm. The additional IR illumination ($\lambda > 1000$ nm) was applied for recording the dashed curve 2.

contains three bands: the first one belongs to the absorption band of the silver halide MC ($\lambda_{max} = 450-460$ nm), the second one coincides with the absorption range of the molecular dye and the third one – with the absorption range of the J-aggregated dye at $\lambda_{max} = 630$ nm and $\lambda_{max} = 690$ nm, correspondingly. The additional IR illumination $\lambda_{IR} > 1000$ nm induces strong quenching of the luminescence intensity within the silver halide absorption range $\lambda_{max} = 450-460$ nm and weak quenching within the absorption range of the molecular dye $\lambda_{max} = 630$ nm; in the absorption range of the J-aggregated dye $\lambda_{max} = 690$ nm, no luminescence quenching is observed (Fig. 3a, curve 4').

In Fig 3c, the luminescence spectra are presented of the emulsion AgBrI (3% AgI) with pBr 4 for the introduced dye concentration $C_{Dy} = 5 \cdot 10^{-4}$ (mole Dy/mole AgBr), and with additionally introduced supersensitizer $C_{SS} = 6 \cdot 10^{-3}$ (mole SS/mole AgBr). As a supersensitizer, the undyed organic compound di(n-anizil)-fenilfosfin was used. As is shown in [4], the supersensitizers can change the charge state of the silver centers and promote their transformation into the complexes of the atomic-molecular degree of dispersion.

When this emulsion is excited by the light from the absorption band of the dye J-aggregate ($\lambda_{\max} = 690$ nm), as before, the luminescence spectrum contains the emission bands: the anti-Stokes luminescence of the twin iodine centers ($\lambda_{\max} = 560$ -570 nm), the anti-Stokes ARF of the molecular dye $\lambda_{\max} = 630$ nm and the luminescence band of the (II - Ag₂⁺)-centers ($\lambda_{\max} = 750$ nm). Noticeably, the phosphorescence band of the molecular dye ($\lambda_{\max} = 800$ nm), upon excitation of the J-aggregated dye, is absent in this case (Fig. 3c, curve 1). Under the action of the additional IR illumination $\lambda > 1000$ nm, both the anti-Stokes luminescence of the twin iodine centers and the anti-Stokes ARF of the molecular dye experience remarkable luminescence quenching whereas the emission of the (II - Ag₂⁺)-centers is enhanced (Fig. 3c, dashed curve 2).

Discussion of the results

As can be seen from the presented experimental data, the luminescence-based technique permits to establish the correspondence between the self-desensitization processes that occur in the anionic dye J-aggregates adsorbed on the surface of AgBrI MC upon the J-aggregate excitation, and the specific luminescence bands that can be used for the control and evaluation of the self-desensitization processes in the AgBrI MC. These are the Anti-Stokes ARF of the molecular dye M_{ARF} ($\lambda_{\max} = 610$ -640 nm) responsible for the I-kind self-desensitization, and the phosphorescence of the molecular dye ($\lambda_{\max} = 800$ nm) responsible for the II-kind self-desensitization.

The following facts testify in favor of this supposition. The ARF of the molecular dye, upon excitation by the light from the absorption range of the J-aggregated dye, experiences the strong influence of the IR illumination and of the variable concentration of silver ions on the surface of the AgBrI MC (Fig. 2a and Fig. 3). Consequently, the anti-Stokes ARF of the molecular dye realizes a channel for the photoexcitation transfer from the J-aggregate to the molecular dye that is controlled by the silver centers of the atomic-molecular degree of dispersion situated at the surface of the AgBrI MC. This provides a ground for the conjecture that this channel is responsible for the self-desensitization of the II kind.

Different reactions of the fluorescence excitation bands to the additional IR illumination supply supplementary ways for the self-desensitization control. For example, upon excitation from the absorption range of the molecular dye, the phosphorescence quenches under the IR action whereas the molecular dye phosphorescence excited by the light from the absorption range of the J-aggregated dye experiences no effect of the IR radiation (Fig. 3a, dashed curve 4'). As a result, for the phosphorescence of the molecular dye on the AgBrI MC surface, excitation is transferred from the J-aggregate to the molecular dye via the channel in which the silver centers of the atomic-molecular degree of dispersion

take no part. This leads to a suggestion that this channel of recombination is responsible for the self-desensitization of the I kind.

Mechanisms of the non-equilibrium electrons and holes' transition from the photoexcited J-aggregated dye to the molecular dye where the recombination takes place that gives rise to these luminescence bands, are considered in detail elsewhere [18,20].

Also, the presented results form the basis for an assumption that the anti-Stokes ARF and the phosphorescence, upon excitation from the absorption range of the J-aggregated dye ($\lambda_{\max} = 690$ nm), appear as a response of the dye molecules that undergo different interactions with the dye J-aggregates. This assumption is supported by the observation that, after introduction of the supersensitizer, the non-equilibrium charge-carriers' transfer from the J-aggregate to the molecular dye decreases, and only the ARF of the molecular dye is observed. An additional support to this view follows from the fact that the change of the silver ions' contents on the MC surface contributes to the decay of the molecular dye ARF.

It should be noted that account for the self-desensitization processes is especially essential for the case of spectral sensitization of fine-dispersed silver-halide emulsions used in holography.

Conclusion

1. A luminescence-based technique is proposed that enables to control the processes of interaction between the molecular and polymolecular forms of the dye adsorbed on the surface of AgBrI microcrystals, which are responsible for the self-desensitization of the I and II kinds. The technique can be applied for the control of the self-desensitization processes in the system.

2. It is revealed that upon excitation of the dye J-aggregates, the I-kind self-desensitization occurs due to immediate transmission of the non-equilibrium electrons and holes from the J-aggregate to the molecular dye, which entails the molecular dye phosphorescence.

3. The II-kind self-desensitization processes, upon excitation of the dye J-aggregates, are associated with the transmission of electrons from the J-aggregated dye to silver centers of the atomic-molecular degree of dispersion; a part of the electrons can pass to the energy levels of the molecular dye and recombine with the localized holes. Evolution of the II-kind self-desensitization processes with excitation of the dye J-aggregates is accompanied by the anti-Stokes anomalously retarded fluorescence of the molecular dye.

4. It is shown that the anti-Stokes anomalously retarded luminescence of the molecular dye and the phosphorescence of the molecular dye upon excitation of the dye J-aggregates are related to different dye molecules and are determined by different electron-hole processes and by different interactions of the molecules with the dye J-aggregates.

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