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V.M. BILOUS,[1] G.V. KLISHEVICH,[2] V.I. MELNYK[2]

[1] Odesa I.I. Mechnikov National University, Research and Development Institute of Physics
(27, Pasteur Str., Odesa 65082, Ukraine; e-mail: yosha@juno.com)
[2] Institute of Physics, Nat. Acad. of Sci. of Ukraine
(46, Nauky Ave., Kyiv 03028, Ukraine)

ANTI-STOKES LUMINESCENCE OF AgBr MICROCRYSTALS WITH ADSORBED POLYMETHINE DYE: ROLE OF cis- AND trans- STEREOISOMERS OF J-AGGREGATES OF DYE

The peculiarities of adsorption of cis- and trans-stereoisomers of the thiatrimethinecyanine dye molecules and aggregates on the surface of AgBr microcrystals, as well as the participation of those isomers in the generation of anti-Stokes luminescence in the system "AgBr microcrystal–adsorbed dye" have been studied.

K e y w o r d s: AgBr microcrystal, molecular stereoisomers, dye aggregates, anti-Stokes luminescence.

1. Introduction

The creative activity of M.T. Shpak was distinguished by a wide scope of scientific research and the solution of important applied problems. In the 1960s–1980s, a series of studies of the optical properties of polymethine dyes was carried out under the supervision of M.T. Shpak, which found applications in nonlinear optics and laser technology [1–12]. As is known, the indicated dyes were also used as spectral sensitizers in photographic emulsions, which are suspensions of light-sensitive silver halide microcrystals (pure AgBr or AgBr with an AgJ admixture) in a gelatin solution. After the microcrystals have been synthesized, a dye solution is added to the emulsion, and, depending on the concentration, it can be adsorbed on the microcrystals in the form of molecules (M), dimers (D), trimers (H-aggregates), and so-called J-aggregates. A J-aggregate consists of four molecules; when absorbing light, they play the role of a "spectroscopic cell".

For silver centers of latent photographic image to be formed following the two-stage electron-ion mechanism [13], it is necessary that light absorption by AgBr or the dye leads to the appearance of an electron in the conduction band or in a shallow AgBr trap. Therefore, polymethine dyes, which expand the photosensitivity region, are selected in such a way that the first excited singlet level (S_1) is in the conduction band or close to the band bottom, and the filled singlet ground level (S_0) is in the AgBr band gap.

In polymethine dyes, there are two π-electrons at the S_0 level, and when a light quantum is absorbed, one π-electron (without changing its spin) transits from the S_0 level to the S_1 level. To characterize the arrangement of those levels with respect to the energy levels in the valence and conduction bands of AgBr, the polygraphic method of determining the reduction, $V^{\rm red}$, and oxidation, $V^{\rm ox}$, potentials of the dye is used because they reflect the energies of the S_1 and S_0 levels, respectively. A comparison between the efficiency of the photographic influence of a set of dyes and the values of their potentials made it possible to determine the polygraphic potentials of the conduction band bottom, $V_{\rm CB}$, and the valence band top, $V_{\rm VB}$, in AgBr: in the scale of polygraphic potentials, $V_{\rm CB} = -1.34$ V and $V_{\rm VB} = +1.26$ V (relative to the saturated calomel electrode, SCE) [14].

When the temperature of spectrally sensitized layers is lowered, and they are irradiated with light that is absorbed by the AgBr microcrystals or the

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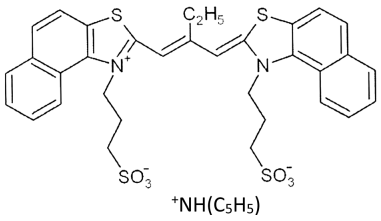
Table 1. **The name and formula of the dye, the polygraphic potentials, and the absorption band maxima in water**

Name and structural formula	Molecular weight, g/mol	λ_{max} in C_2H_5OH, nm	Polygraphic potentials (relative to SCE)	λ_{max} in H_2O, nm
Pyridinium salt 3,3′-di-(γ-sulfo-propyl)-4,5,4′,5′-dibenzo-9-ethylthiatri-methincyanine betaine 	759.9	581(M)	$V^{red} = -1.34$ $V^{ox} = +0.67$	535 (D) 575 (M) 637 (J_1) 657 (J_2)

adsorbed dye, their luminescence can be excited [15]. Furthermore, as was first shown in 1967 by Ovsyankin and Feofilov [16], if infrared light is used to irradiate specimens, radiation appears in the visible spectral interval (anti-Stokes luminescence, ASL). This phenomenon was studied and described in the review [17] and the research article [18]. For its explanation, various excitation schemes were proposed, which took into account the participation of two quanta in the absorption of long-wavelength light and the appearance of one luminescence quantum $h\nu_{Lum}$ ($h\nu_{IR} < h\nu_{Lum} < 2h\nu_{IR}$). For what follows, it is important to emphasize that J-aggregates, which are responsible for the ASL generation, consist of four geometrically identical molecules of the dye–sensitizer, which is introduced into the gelatin solution after the AgBr microcrystals have been synthesized.

2. Experiment

A fundamentally different scheme for obtaining specimens was used in this work [1]. Portions of 25 ml of 0.5-N KBr and $AgNO_3$ solutions were simultaneously introduced at equal rates and at room temperature into 50 ml of the water-gelatin (0.05% gelatin) solution of the dye (10^{-3} mg/l) (the name and formula of the dye, the polygraphic potentials, and the absorption band maxima in water are given in Table 1). Then, the solution temperature was raised to 35°C, and the gelatin concentration was increased to 5%. The gelatin concentration was increased in a special glass vessel with a magnetostrictor, which allowed the solution to be mixed under a permanent exposure to ultrasound (the exposure time and power were adjusted) at a frequency of 15 kHz. The magnetostrictor was powered by an ultrasonic generator, the power of which was regulated within the interval of 0.35–1.5 kW. The ultrasound favored better mixing of the solutions in the specimens.

The resulting emulsion was poured onto the surface of a glass substrate, and the specimen was placed in a helium cryostat. Luminescence spectra (in the interval of 300–720 nm) and the excitation spectra of various emission bands were registered using an experimental setup, which allowed measurements to be carried out at the temperature $T = 4.2$ K. In this case, when light was absorbed by AgBr, a luminescence band with $\lambda_{max} = 497$ nm was observed (it is associated with the presence of an uncontrolled (small) amount of iodine ions in the AgBr microcrystals), as well as bands generated by the adsorbed dye (see a more detailed consideration below).

The emergence of those bands occurs according to the following mechanism. A hole h from the valence band is trapped by an emission center and recombines with an electron e from the conduction band. As a result, the emission center becomes excited and generates a luminescence quantum (an exciton localized

[1] It should be noted that some of the experimental results presented in this work were obtained in 1991–1992, but they were not published.

Table 2. **Spectral maximum positions (λ and ν) and the vibronic frequency $\Delta\nu$ in the vibronic spectra of anti-Stokes luminescence excitation of iodine ions (the band with $\lambda_{max} = 497$ nm, columns A, B) and in the fragment of the luminescence excitation spectrum of the J_2-aggregate (column C)**

Transitions	J_1-aggregate band, $\lambda_{max} = 670$ nm (A)			J_2-aggregate band, $\lambda_{max} = 683$ nm (B)			J_2-aggregate luminescence band, $\lambda_{max} = 684$ nm (C)		
	λ, nm	ν, cm^{-1}	$\Delta\nu$, cm^{-1}	λ, nm	ν, см$^{-1}$	$\Delta\nu$, cm^{-1}	λ, nm	ν, cm^{-1}	$\Delta\nu$, cm^{-1}
0, 0′	670.0	14925	436	683.0	14641	732			
1, 0′	651.0	15361	437	650.5	15373	730	650.5	153.73	730
2, 0′	633.0	15798	436	621.0	16103		621.0	16103	732
3, 0′	615.0	16234					594.0	16835	

at the impurity center). From this scenario, it follows that in order to explain the appearance of anti-Stokes luminescence, it is necessary to find out how the absorption of two infrared light quanta by the adsorbed dye gives rise to the creation of an electron-hole pair in AgBr (the band gap of AgBr equals: $E_g = 2.6$ eV at $T = 300$ K, and $E_g = 2.7$ eV at $T = 4.2$ K).

For this purpose, the anti-Stokes luminescence excitation spectrum of AgBr microcrystals with the adsorbed dye was measured in the interval $\lambda < 720$ nm (Fig. 1). It was found that this excitation spectrum is identical for each band observed in the visible spectral interval; see Fig. 2 (the band of residual iodine ions with $\lambda_{max} = 497$ nm), the bands of molecules (M, with $\lambda_{max} = 581$ nm), dimers (D, with $\lambda_{max} = 546$ nm), and H-aggregates (H, with $\lambda_{max} = 536$ nm)). The same dye bands can also be excited by light in the AgBr absorption interval. As follows from Fig. 1, two overlapping bands are registered in the excitation spectrum: J_1 ($\lambda_{max} = 670$ nm) and J_2 ($\lambda_{max} = 683$ nm) with a vibronic structure (Table 2).

To clarify the nature of those bands, it is important to take into account the results of Ref. [19], where it was shown that in an aqueous solution of the dye used in this study, there appear the cis and trans stereoforms of the dye molecules; those forms, in turn, form the cis- and trans- forms of dimers, which participate in the formation of the cis- and trans- forms of J-aggregates. Schematic diagrams of those "enlargements" are shown in Fig. 3 (reproduced from Ref. [19]).

Electron microscopy showed that the sizes of the AgBr microcrystals varied from 22 to 24 nm. Since those microcrystals were synthesized in an aqueous-

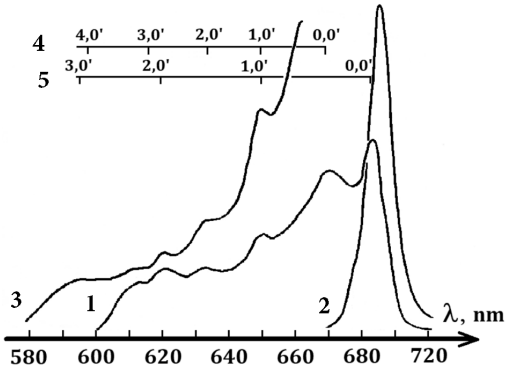


Fig. 1. Excitation spectrum of various anti-Stokes luminescence bands of the dye (the same for all bands) (*1*). Luminescence band of J_2-aggregate (*2*). Fragment of the luminescence excitation spectrum of J_2-aggregate (*3*). Horizontal auxiliary axes *4* and indicate the spectral position of the maxima in the vibronic spectrum *5* of the J_1- and J_2-aggregates, respectively. The intensity scale is arbitrary

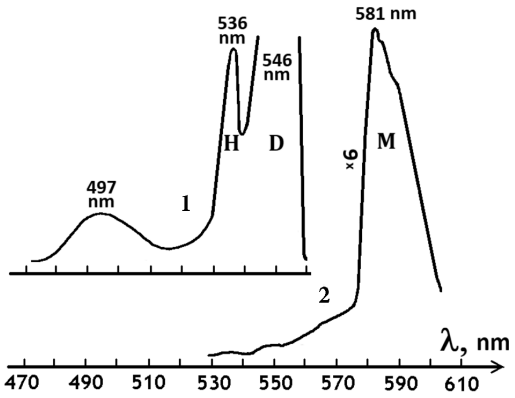


Fig. 2. Spectrum of anti-Stokes luminescence of AgBr microcrystals with adsorbed dye. The scale of curve *2* is 70 times smaller than that of curve *1*. The intensity scale is arbitrary

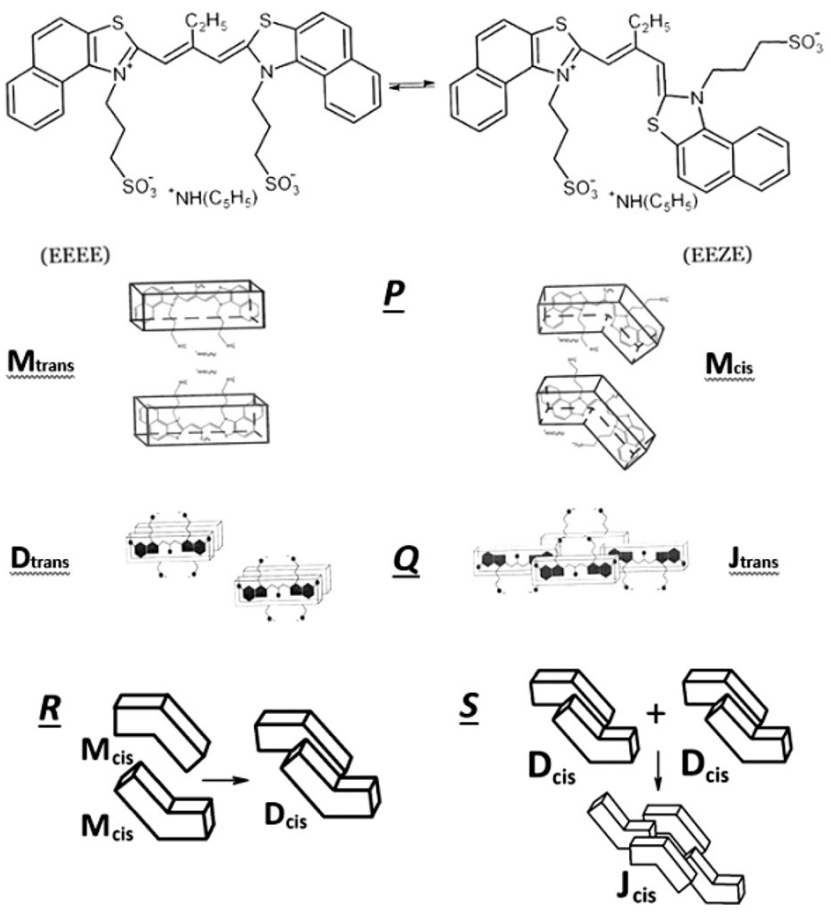


Fig. 3. Spatial view of stereoisomers, molecules, dimers, and J-aggregates of the dye. Panel *P*: the type and mutual reversibility of cis- and trans-isomers of the thiatrimethinecyanine dye molecule and their spatial-block representation (in the form of separate blocks); panel *Q*: schematic diagram of formation of J_{trans}-aggregates from trans dimers; panel *R*: schematic diagram of formation of cis dimers from cis monomers (dye molecules); panel *S*: schematic diagram of formation of J_{cis}-aggregates from cis dimers

gelatin solution of the dye, the dye layer adsorbed on the microcrystal surfaces consisted of molecular stereoisomers, dimers, and J-aggregates. This means that the short-wave J_1-aggregate and the long-wave J_2-aggregate are J_{cis}- and J_{trans}-aggregates, respectively. This conclusion is confirmed by the difference between the vibronic frequencies of 436 cm^{-1} [for the band J_1 (J_{cis})] and 732 cm^{-1} [for the band J_2 (J_{trans})] of the ASL excitation of AgBr microcrystals with adsorbed dye (Fig. 1, Table 2), because the indicated stereoisomers have different geometries. In addition, note that the luminescence excitation spectrum of the J_{trans} aggregate (Fig. 1, curves *2* and *3*; Table 2, column C) expectedly demonstrates a vibronic structure with a vibronic frequency of 732 cm^{-1}.

The presence of gelatin molecules and Ag^+ ions (introduced during the synthesis of AgBr) in the aqueous-gelatin solution of the dye favors the stabilization of J_{cis}- and J_{trans}-aggregates and prevents the transformation $J_{cis} \leftrightarrow J_{trans}$ upon specimen heating and cooling; see, for example, Ref. [20].

An analysis of the obtained luminescence spectra showed (Fig. 1, curve *2*) that the J_{trans}-aggregate emits (the band with $\lambda_{max} = 684$ nm), and the J_{cis}-aggregate does not emit, although emission by the J_{cis}-aggregate is observed in the aqueous-gelatin solution of the dye (the band with $\lambda_{max} = 673$ nm).

3. Results and Discussion

The obtained result allows us to conclude that in the adsorbed J_{trans} aggregate, the S_1 and S_0 levels are in the AgBr band gap. At the same time, in the J_{cis}-aggregate, which has an oxidation potential higher than that of J_{trans}, the ground S_0 level is in the valence band, so the radiative transition is excluded (Fig. 4). In this case, when the J_{cis}-aggregate absorbs an IR light quantum (the $S_0 \rightarrow S_1$ transition), there appears a vacancy at the S_0 level, which becomes

filled with an electron from the AgBr valence band, with the formation of an anion radical J^-_{cis}, stable at the temperature $T = 4.2$ K, and a hole h in the valence band (Fig. 4).

Based on the experimental results presented above, the following ASL mechanism can be proposed. Since the bands of the J_{cis}- and J_{trans}-aggregates overlap in the ASL excitation spectra, then, if the specimen is irradiated with IR light (with a frequency in the interval of those bands), there immediately appear a J^-_{cis} anion-radical, a hole in the valence band, and luminescence from the adsorbed J_{trans}-aggregate (Fig. 4). Absorption of a luminescence or IR light quantum by the J_{cis}-aggregate leads to an electron transition into the AgBr conduction band and a "reduction" of the J_{cis}-aggregate (it ceases to be a J^-_{cis} anion). Thus, the absorption of two IR light quanta ensures the appearance of an electron in the conduction band and a hole in the valence band. Later, the hole, when migrating through the valence band, is captured by the ground S_0 level of the M-molecule, the D-dimer, or the H-aggregate, converting them into the cations M^+, D^+, or H^+, respectively. The recombination of an electron from the conduction band with the indicated cations leads to the emergence of the excited M^*, D^*, and H^* states (the electron transition to the S_1 level and the formation of a localized exciton), with a subsequent emission of an ASL quantum.

The described mechanism of ASL emergence also explains the identity of the excitation spectra of all ASL bands. Measurements of the emission spectra of the adsorbed J_{trans}-aggregate on another experimental setup showed that a phosphorescence band (the $T \to S_0$ transition) with $\lambda_{max} = 800$ nm is also registered in the interval $\lambda > 720$ nm at $T = 77$ K.

According to the mechanism proposed above, the mentioned phosphorescence emission may also participate in the emergence of anti-Stokes luminescence. In addition, it is necessary to take into account a possible non-radiative energy transfer from the excited J_{trans}-aggregate to the anion-aggregate J^-_{cis}.

It should be emphasized that the ASL of the adsorbed dye aggregates has a number of features.

i) The band of the H-aggregate is quite narrow, which testifies to a dense packing of molecules in the aggregate. If, during their adsorption on the AgBr surface, the dye molecules are arranged in the aggregate in such a way that the angle α between the line

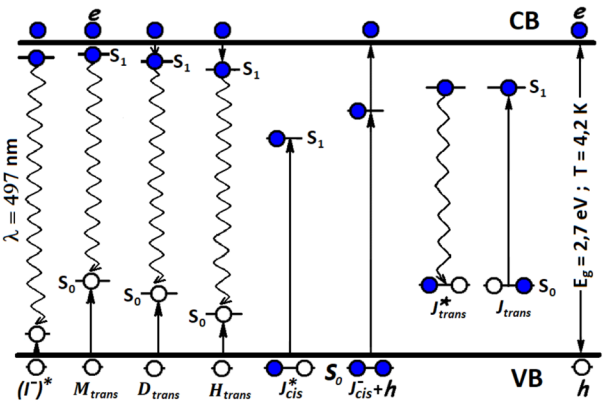


Fig. 4. Band diagram of the energy levels of the molecules, dimers, and J_{cis}- and J_{trans}-aggregates of the dye and iodine impurity ions in AgBr (the band with $\lambda_{max} = 497$ nm) adsorbed on AgBr, which explains the excitation ($S_0 \to S_1$) of anti-Stokes luminescence, VB denotes the AgBr valence band, CB denotes the AgBr conduction band, e and h are an electron and a hole in the corresponding bands, J^-_{cis} is the anion-radical, and J^*_{trans} is an excited J_{trans}-radical. The energy levels of the vibronic structure are not shown; wavy lines with arrows mark radiative transitions; straight lines with arrows mark absorption (excitation or capture)

connecting the centers of the molecules and the dipole direction in the molecule is 90°, then, as was shown in Ref. [21], the following equality holds:

$$\Delta\nu(\mathrm{H-M}) = 2\Delta\nu(\mathrm{D-M})\frac{N-1}{N}, \qquad (1)$$

where N is the number of molecules in the aggregate, $\Delta\nu$(H–M) is the frequency difference between the maxima of the absorption bands (the fluorescence resonance) for the aggregate and the molecule, and $\Delta\nu$(D–M) is a similar frequency difference for the dimer and the molecule. According to the obtained experimental results (Fig. 2), the maxima of the fluorescence bands of molecules (M), dimers (D), and H-aggregates are located at $\lambda_{max} = 581$ nm (17212 cm^{-1}), 546 nm (18298 cm^{-1}), and 536.5 nm (18657 cm^{-1}), respectively, and $\Delta\nu$(H–M) is equal to 1445 cm^{-1}. The same difference (1445 cm^{-1}) is obtained when calculating according to equality (1) with $N = 3$. This fact confirms our conclusion that the H-aggregate consists of three molecules, which are planarly adsorbed one above the other in the form of a ladder.

ii) There appears a weakly pronounced vibrational structure (steps) on the long-wavelength decay of the

ASL emission band of the excited dye molecule. As follows from Fig. 2 (curve *2*), the indicated steps are separated from the maximum of the molecular fluorescence band by one or two vibrational quanta. The spectral positions of this fluorescence band maximum, 581 nm (17211 cm^{-1}), and these steps, 584.5 nm (17108 cm^{-1}) and 587 nm (17007 cm^{-1}), allowed us to determine the frequency of the molecular vibrational quantum. It was found to equal 102 ± 1 cm^{-1}.

iii) As follows from the measurements (Fig. 2, curve *2*), the ASL of molecules is characterized by a very high intensity. Such an intensity growth at $T = 4.2$ K is explained by the following reason. As was noted above, the S_1 level in a molecule of the examined dye coincides with the AgBr conductivity band bottom at room temperature. A temperature reduction to 4.2 K leads to a broadening of the AgBr band gap, and the indicated S_1 level (taking into account the vibronic band structure of the transition $S_0 \to S_1$) is found below the bottom of the conduction band, where the band of AgBr exciton states is located. As was first shown by Rashba [22], if the impurity exciton level approaches the exciton band edge in the base substance, the strength of the impurity exciton oscillator increases drastically. It is this effect that is responsible for the observed sharp increase in the intensity of anti-Stokes photoluminescence from adsorbed molecules.

The explanation given above points to the specificity of the adsorption interaction between the polymethine dye molecule and a real (with defects) surface of silver bromide microcrystals. This hypothesis requires a more detailed experimental and theoretical justification.

4. Conclusions

To summarize, an important role of stereoisomers of thiatrimethinecyanine dye J-aggregates in the excitation of anti-Stokes luminescence of AgBr microcrystals 22–24 nm in size and with adsorbed dye has been shown for the first time. Also, for the first time, the ASL from iodine-ion impurities in AgBr, dye molecules (the band with the vibration frequency $\nu = 102$ cm^{-1}), dimers, and H-aggregates was registered. At $T = 4.2$ K, a vibronic structure was detected in the anti-Stokes luminescence excitation bands, and the difference between the vibronic frequencies of the J_{cis} and J_{trans}-aggregates (436 and 732 cm^{-1}, respectively) was found. The participation of cis and trans stereoisomers of polymethine dyes in the photophysical process may also be important for explaining other problems that arise in the practical application of these dyes, in particular, in the synthesis and spectral sensitization of fine-grained silver halide emulsions used in holography.

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В.М. Білоус, Г.В. Клішевич, В.І. Мельник

АНТИСТОКСОВА ЛЮМІНЕСЦЕНЦІЯ МІКРОКРИСТАЛІВ AgBr З АДСОРБОВАНИМ ПОЛІМЕТИНОВИМ БАРВНИКОМ: РОЛЬ цис- ТА транс-СТЕРЕОІЗОМЕРІВ J-АГРЕГАТІВ БАРВНИКА

Вивчено особливості адсорбції цис- та транс-стереоізомерів молекул і агрегатів тіатриметинціанінового барвника на поверхні мікрокристалів AgBr та участь цих ізомерів у виникненні антистоксової люмінесценції в системі "мікрокристал AgBr-адсорбований барвник".

Ключові слова: мікрокристал AgBr, стереоізомери молекул, агрегати барвника, антистоксова люмінесценція.