

# Aggregation of dyes in porous glass

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The research examines the interaction of dye molecules with their dimers (H aggregates) and the more complex formations (J aggregates) developing in porous glass. The use of porous glass when dealing with dye aggregation has resulted in obtaining photoluminescence dimers of the J aggregating dye, the formation of which is difficult under normal conditions. In addition, the porous glass matrix contributes to a substantial reduction in the interaction of photoexcited states of both a molecular and an aggregated dye, thus helping maximize the luminescence efficiency of porous glass-distributed dyes.

Keywords: dye, porous glass, luminescence, aggregates.

## 1. Introduction

Dyes absorb light selectively and efficiently, as they have a high quantum efficiency of irradiation. This property of dyes is used in spectral sensibilisation of halogen-silver emulsions [1], in photoelectric transducers based on nanoparticles and nanotubes (organic dye-sensitized solar cell, DSSC) [2], and in laser equipment [3–5].

The efficiency using dyes in laser equipment, during the emission of radiation from the first singlet level of dye molecules is significantly complicated by the interaction between particular dye molecules at high concentrations. This leads to the emergence of ordered dye dimers of the so-called H aggregates and polymolecular structure, the so-called J aggregates which absorb light in spectral ranges shifted relative to the absorption spectrum of the molecular dye [6], promoting a decrease in the transfer of photoelectrons to singlet levels of the dye molecules.

The emergence of associate dye molecules implies an opportunity for their interaction, which also has a significant impact on the efficiency using dye for laser equipment.

The research related to the possibility of controlling the process of aggregation of the dye is quite significant.

It is especially important that predominant emergence of H or J aggregates in a dye is not only related to the special structure and concentration of dye molecules in the solution, but also the dimension and state of the sorption surface, the special limitation and change of state of which may change the type of dye molecules aggregation, thus enabling one to control this process.

The latter point has served as the subject of this study dealing with peculiarities of the H and J aggregation of dyes, and photoexcitation of these aggregates interaction with dye molecules in case of spatial limitation and different states of the porous glass matrix which is a sorption surface.

## 2. Experiment

One of the techniques of studying the internal conversion of dye photoexcitation is the luminescent method with a temporal resolution of spectra [7]. The experimental facility that was used to perform low-temperature ( $T = 77$  K) luminescent studies is able to measure spectral values not only in continuous exciting light irradiation, but also in case of discontinuous (modulated) excitation, when the time of excitation of the specimen is equal to the time of registration of its luminescence, which is  $0.1 \times 10^{-4}$  s, while the interval between the end of irradiation and luminescence registration was  $1.1 \times 10^{-3}$  s.

This choice of the measuring technique was made because in continuous excitation, luminescence includes all major glows of luminescence centres: fluorescence, anomalously retarded fluorescence and phosphorescence. In modulated excitation, the glow is only caused by anomalously retarded fluorescence and phosphorescence of luminescence centres.

The luminescence studies were performed for two J aggregating dyes of cation and anion type, whose structural formulas are presented in Fig. 1.

A porous glass matrix was chosen to be the absorption surface with two predominant pore dimensions in the range of nanometres: matrix type A – mid-size pore diameters  $d_1 = 10$  nm and  $d_2 = 20$  nm (conditionally “small”) and matrix type C – mid-size pore diameters  $D_1 = 20$  nm and  $D_2 = 50$  nm (conditionally “large”), which appeared ideal in view of the limitation of the dimension of the sorption surface that displayed the aggregation and interaction of dyes. The procedure of measuring the medial sizes in the nanometre range of pores is given in [8].

The implantation of the dye into the porous glass was performed by dipping it in the dye solution and holding it there for 3 minutes, excessive dye being removed from the specimen surface by filter paper.

The dye solutions were made in isopropyl and polyvinyl alcohol (PVA) with a percentage of three weight percent and  $5 \times 10^{-4}$  g/mol/liter of dye concentration.

The choice of these solvents was made because in transition from isopropyl alcohol to polymeric solvent, PVA, apart from doing its primary job as a solvent, can contribute

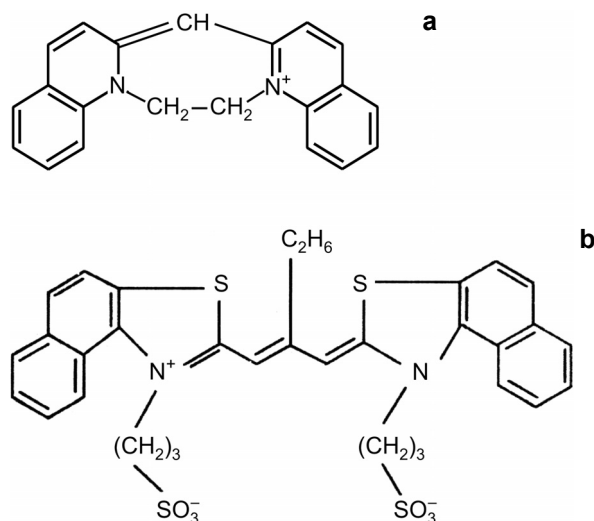


Fig. 1. The 1,1'-diethyl-2,2'-cyanineiodine, hereinafter referred to as Dye-I (a); pyridine salt 3,3'-( $\gamma$ -sulphopropyl)-4,5,4',5'-dibenzo-9-ethylthiacyaninebetaine, hereinafter referred to as Dye-II (b).

to a change to the state of the internal surface of pores of microporous glass, as was mentioned in the paper, thus changing the nature of dye aggregation in porous glass [9].

Pursuant to our studies and known experimental data [10–12] for the two selected dyes, we have designed a table presenting maxima of absorption and glow of molecules (M) of dimers (H) and aggregate dyes (J), in a solution of isopropyl (Tab. 1) and polyvinyl (Tab. 2) alcohol excited by light in the range of 400–700 nm.

Table 1. Position of band maxima (nm). Solution dyes in isopropyl alcohol. (A dash means no data available.)

	Luminescence											
	Absorption			Fluorescence			Anomalously retarded fluorescence			Phosphorescence		
	H	M	J	H	M	J	H	M	J	H	M	J
Dye-I	440	510	540	620	570	550	–	–	–	–	700	–
Dye-II	490	600	640	570	610	650	–	–	650	–	760	–

Table 2. Position of band maxima (nm). Solution dyes in PVA. (A dash means no data available.)

	Luminescence											
	Absorption			Fluorescence			Anomalously retarded fluorescence			Phosphorescence		
	H	M	J	H	M	J	H	M	J	H	M	J
Dye-I	470	510	540	650	570	550	–	–	–	–	700	–
Dye-II	490	600	640	570	620	650	570	–	650	–	780	–

### 3. Results

In excitation with either continuous (Fig. 2a, curves 1, 2) or modulated (Fig. 2c, curve 3) light at  $\lambda = 450$  nm, the luminescence spectra for the specimens of porous glass having Dye-I in isopropyl alcohol, regardless of the size of pores of the glass matrix, are only characterised by one glow maximum, the position of which fits the same wavelength, and which are only different from each other by the glow maximum intensity.

The luminescence maximum of the given band fits a wavelength of  $\lambda_{\max} \approx 610\text{--}620$  nm and, consequently, the glow can be referred to as fluorescence, also including anomalously retarded fluorescence of the H aggregates of Dye-I in the case of continuous excitation, otherwise to anomalously retarded fluorescence H aggregate of Dye-I in the case of modulated excitation (see Tab. 1).

Compared to the luminescence of the solution not distributed in porous glass, the following should be mentioned: in the glow of Dye-I in porous glass, there is no phosphorescence of Dye-I molecules ( $\lambda_{\max} \approx 700$  nm), with only anomalously retarded fluorescence of the H-aggregates of Dye-I ( $\lambda_{\max} \approx 610\text{--}620$  nm).

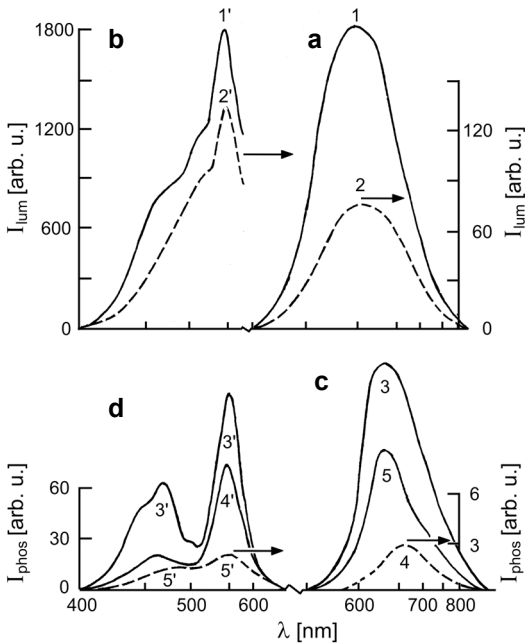


Fig. 2. Spectra of low-temperature ( $T=77$  K) luminescence (a, c), excitation of luminescence (b, d), in continuous (a, b) and modulated (c, d) excitation of isopropyl alcohol solution with Dye-I in porous glass matrix type A (solid line) and matrix type C (dashed line). Spectra of luminescence recorded in light excitation at  $\lambda = 450$  nm (curves 1, 2) (a). Spectra of excitation recorded for luminescence at  $\lambda = 610$  nm – curves 1', 2' (b). Spectra of luminescence recorded in light excitation at  $\lambda = 450$  nm – curve 3 and  $\lambda = 550$  nm – curves 4 and 5 (c). Spectra of excitation recorded for luminescence at  $\lambda = 650$  nm – curves 3', 5' and at  $\lambda = 750$  nm – curve 4' (d).

While the solution had phosphorescence of Dye-I molecules only, there is no anomalously retarded fluorescence of H-aggregates of Dye-I.

In the spectrum of continuous excitation of the fluorescence of H-aggregates of Dye-I at  $\lambda_{\max} \approx 610$  nm, only three overlapped bands are observed with maxima at  $\lambda_{\max} \approx 440$  nm,  $\lambda_{\max} \approx 510$  nm, and  $\lambda_{\max} \approx 540$  nm which are most distinct for porous glass matrix type A (Fig. 2b, curve 1'). These bands are related to the area of absorption of H dimers, molecular M and J-aggregated Dye-I, respectively (see Tab. 1).

In the spectrum of modulated excitation of anomalously retarded fluorescence of H aggregates of Dye-I ( $\lambda_{\max} \approx 610$  nm), four overlapped bands are observed at  $\lambda_{\max} \approx 440$  nm,  $\lambda_{\max} \approx 470$  nm, a low-intensity band at  $\lambda_{\max} \approx 510$  nm, and the most intensive amongst excitation bands at  $\lambda_{\max} \approx 540$  nm (Fig. 2d, curve 3').

These bands may be classified as follows: the bands at  $\lambda_{\max} \approx 510$  nm and  $\lambda_{\max} \approx 540$  nm, as in the case of continuous excitation, fit the absorption of molecular and J-aggregated Dye-I. Concerning the two bands at  $\lambda_{\max} \approx 440$  nm and  $\lambda_{\max} \approx 470$  nm which fit the Dye-I dimers' absorption region, it is for the first time that we have seen them in the excitation spectrum of anomalously retarded fluorescence of H-aggregates of Dye-I; their nature being ambiguous, we suggest dealing with this issue during a later discussion.

When looking at the spectrum of low-temperature ( $T = 77$  K) luminescence of a specimen of porous glass matrix type A having a solution of isopropyl alcohol at Dye-II in continuous excitation at  $\lambda = 500$  nm, an intensive glow band at  $\lambda_{\max} \approx 610$  nm (Fig. 3a, curve 2) is visible and typical of the fluorescence of Dye-II molecules. For the spectrum of continuous excitation of this glow band, a maximum is visible at  $\lambda_{\max} = 600$  nm (Fig. 3b, curve 2'), which coincides with the absorption maximum of molecular Dye-II (see Tab. 1).

On exposure to modulated light excitation at  $\lambda_{\max} = 600$  nm from the absorption region of molecular Dye-II, the above specimen's luminescence spectrum displays a vivid band at  $\lambda_{\max} = 760$  nm (Fig. 3c, curve 5) which relates to the phosphorescence of molecular Dye-II (see Tab. 1).

The spectrum of modulated excitation of the phosphorescence of molecular Dye-II ( $\lambda_{\max} = 760$  nm) does not form a vivid maximum unlike in the case of continuous excitation. Apart from the maximum related to the absorption of molecular Dye-II ( $\lambda_{\max} \approx 600$  nm), it also displays maxima in the absorption regions of Dye-II dimers (Fig. 3d, curve 4') (see Tab. 1).

On exposure to modulated light excitation from the absorption region of Dye-II dimers ( $\lambda_{\max} \approx 450$  nm), the luminescence displays a glow band at  $\lambda_{\max} \approx 610$  nm (Fig. 3c, curve 4) which coincides with that in continuous excitation (Fig. 3a, curve 2) and, consequently, in the case of modulated excitation, it pertains to anomalously retarded fluorescence of molecular Dye-II (see Tab. 1).

When taking a matrix of porous glass matrix type C having a solution of isopropyl alcohol with Dye-II, the luminescence spectrum in continuous light excitation at  $\lambda = 500$  nm displays an overlap of three bands of luminescence at  $\lambda_{\max} = 570$  nm,  $\lambda_{\max} = 610$  nm and  $\lambda_{\max} = 670$  nm (Fig. 3a, curve 1), which may be naturally related

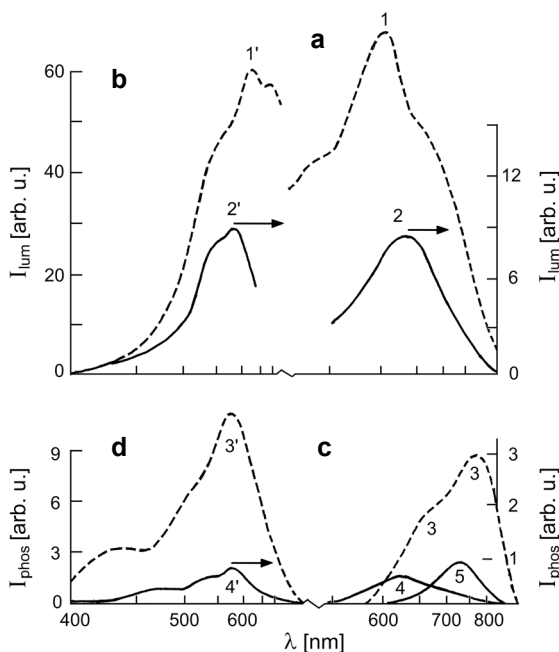


Fig. 3. Spectra of low-temperature ( $T = 77$  K) luminescence (a, c) and excitation of luminescence (b, d) in continuous (a, b) and modulated (c, d) excitation of a porous glass matrix type A (solid line) and matrix type C (dashed line), impregnated with a solution of isopropyl alcohol with Dye-II. Spectra of luminescence recorded in light excitation at  $\lambda = 500$  nm curves 1 and 2 (a). Spectra of excitation recorded for luminescence at  $\lambda = 700$  nm – curve 2' and at  $\lambda = 670$  nm – curve 1' (b). Spectra of luminescence recorded in light excitation at  $\lambda = 450$  nm – curves 4 and at  $\lambda = 600$  nm – curves 3 and 5 (c). Spectra of excitation recorded for luminescence at  $\lambda = 750$  nm – curves 3' and 4' (d).

to the luminescence of dimers, molecules and J aggregates of Dye-II, respectively. In the spectrum of continuous excitation for the long-wave glow band J aggregates of Dye-II ( $\lambda = 670$  nm), there are three bands at  $\lambda_{max} \approx 490$  nm,  $\lambda_{max} \approx 600$  nm and  $\lambda_{max} \approx 650$  nm (Fig. 3b, curve 1') which fit the absorption of dimers, molecules and J aggregates of Dye-II, respectively (see Tab. 1). This indicates that the photoexcitation of dimers and molecules of Dye-II is transferred to the J aggregates of Dye-II.

In modulated light excitation at  $\lambda = 490$  nm, the luminescence spectrum has two bands at  $\lambda_{max} \approx 670$  nm and  $\lambda_{max} \approx 760$  nm (Fig. 3c, curve 3) which pertain to the anomalously retarded fluorescence of J aggregates and phosphorescence of molecular Dye-II, respectively. In the spectrum of modulated excitation of these glow bands at  $\lambda_{max} \approx 760$  nm, bands are found at  $\lambda_{max} \approx 440$  nm,  $\lambda_{max} \approx 490$  nm,  $\lambda_{max} \approx 600$  nm and  $\lambda_{max} \approx 650$  nm (Fig. 3d, curve 3').

The nature of three bands with maxima  $\lambda_{max} \approx 490$  nm,  $\lambda_{max} \approx 600$  nm and  $\lambda_{max} \approx 650$  nm is clear and based on the absorption of dimers, molecules and J aggregates of Dye-II, respectively (see Tab. 1). As to the maximum at  $\lambda_{max} \approx 440$  nm from the absorption region of Dye-II dimers, we have seen this maximum for the first

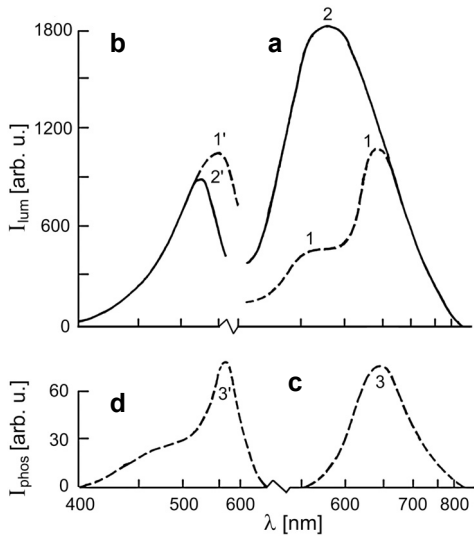


Fig. 4. Spectra of low-temperature ( $T = 77$  K) luminescence (**a, c**) and excitation of luminescence (**b, d**) in continuous (**a, b**) and modulated (**c, d**) light excitation of a porous glass matrix type A (solid line) and matrix type C (dashed line) impregnated with a PVA solution having Dye-I. Spectra of luminescence recorded in light excitation at  $\lambda = 450$  nm – curve 1, 2 (**a**). Spectra of continuous excitation recorded for luminescence at  $\lambda = 700$  nm – curves 1', 2' (**b**). Spectrum of luminescence recorded in excitation by the modulated light at  $\lambda = 550$  nm – curve 3 (**c**). Spectra of modulated excitation recorded for luminescence at  $\lambda = 750$  nm – curve 3' (**d**).

time displayed in the excitation spectrum of the phosphorescence of molecular Dye-II, so its origin requires classifications.

The luminescence spectrum of porous glass matrix type A holding a PVA solution with Dye-I in continuous light excitation at  $\lambda = 450$  nm is characterised by one glow band at  $\lambda_{\max} \approx 570$  nm (Fig. 4a, curve 2), which is typical of the fluorescence of molecular Dye-I (see Tab. 2). In the spectrum of continuous excitation of the above luminescence band, one band of glow excitation is seen with a maximum at  $\lambda_{\max} \approx 510$  nm which fits the absorption region of molecular Dye-I (see Tab. 2).

The specimen under study in modulated light excitation from the range of 400–700 nm does not glow and, consequently, there is no phosphorescence and anomalously retarded fluorescence of H aggregates of Dye-I.

The spectra of luminescence of porous glass matrix type C having Dye-I in PVA in continuous light excitation at  $\lambda = 450$  nm are based on two glow bands at  $\lambda_{\max} \approx 570$  nm and  $\lambda_{\max} \approx 650$  nm (Fig. 4a, curve 1) pertaining to the luminescence of molecular and H aggregated Dye-I, respectively (see Tab. 2).

In the spectrum of continuous excitation of a long-wave luminescence band at  $\lambda_{\max} \approx 650$  nm, there are three bands at  $\lambda_{\max} \approx 470$  nm,  $\lambda_{\max} \approx 510$  nm and  $\lambda_{\max} \approx 540$  nm, which in their spectral absorption coincide with the absorption range of dimers, molecular and J-aggregated Dye-I, respectively (see Tab. 2).

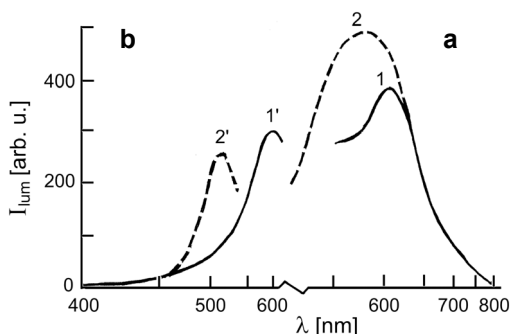


Fig. 5. Spectra of low-temperature ( $T = 77$  K) luminescence (a) and excitation of luminescence (b) with continuous light in porous glass matrix type A (solid line) and matrix type C (dashed line) impregnated with a PVA solution with Dye-II. Spectra of luminescence recorded in light excitation at  $\lambda = 450$  nm – curves 1 and 2 (a). Spectra of excitation recorded for luminescence at  $\lambda = 750$  nm – curve 1' and 2' (b).

In modulated light excitation from the range of 400–700 nm, unlike the porous glass matrix type A having a solution of Dye-I in PVA which does not glow, the specimen with matrix type C displays a glow at  $\lambda_{\text{max}} \approx 650$  nm (Fig. 4c, curve 3) which in its spectral position coincides with the fluorescence of Dye-I H aggregates in continuous excitation and, consequently, can be referred to as anomalously retarded fluorescence of Dye-I H aggregates. The spectrum of modulated excitation of the above glow ( $\lambda_{\text{max}} \approx 650$  nm) displays two bands at  $\lambda_{\text{max}} = 470$  nm and  $\lambda_{\text{max}} = 540$  nm (Fig. 4d, curve 3') which fit into the absorption region of H and J aggregates of Dye-I, respectively (see Tab. 2).

Eventually, on treating the porous glass matrixes of both types with a PVA solution having Dye-II, specimens were only luminescent in continuous excitation.

In the case of the specimen matrix type A in continuous light excitation at  $\lambda = 450$  nm, the luminescence spectrum displays a glow band at  $\lambda_{\text{max}} \approx 610$  nm (Fig. 5a, curve 1) which can be referred to as the fluorescence of molecular Dye-II (see Tab. 2). The spectrum of continuous excitation of the above luminescence band also comprises one band at  $\lambda_{\text{max}} \approx 600$  nm (Fig. 5b, curve 1') which fits the absorption of molecular Dye-II (see Tab. 2).

For the specimen matrix type C in continuous light excitation at  $\lambda = 450$  nm, the luminescence spectrum also displays one glow band at  $\lambda_{\text{max}} \approx 570$  nm (Fig. 5a, curve 2) which in its spectral position fits the fluorescence of Dye-II dimers (see Tab. 2). In the spectrum of continuous light excitation, this band displays one band excitation at  $\lambda_{\text{max}} \approx 490$  nm (Fig. 5b, curve 2') which in its spectral position fits the absorption of Dye-II dimers (see Tab. 2).

It is important that far more intensive glow is seen in the porous glass matrix of the PVA solution with Dye-II compared to the isopropyl alcohol solution with Dye-II (versus the intensity of luminescence in continuous excitation – Fig. 5a, curves 1, 2, and Fig. 3a, curves 1, 2).



## 4. Discussion

The luminescence spectrum of Dye-I in isopropyl alcohol and PVA, excited with modulated light from the range of 400–600 nm, displays a glow band caused by the phosphorescence of molecular Dye-I ( $\lambda_{\max} \approx 700$  nm, see Tab. 2). It is known [13] that such phosphorescence is only visible when dye molecules are adsorbed on H or J aggregates of the dye.

In the case of distribution of a Dye-I solution in isopropyl alcohol, the porous glass matrixes of both types does not display a phosphorescence of the Dye-I regardless of dimensions of pores – neither in the case of continuous nor modulated light excitation from the range of 400–600 nm, while displaying a fluorescence of H aggregated Dye-I, including that anomalously retarded ( $\lambda_{\max} \approx 640$  nm).

Besides, the point should be made that modulated excitation of luminescence of H aggregates from the absorption regions of molecular Dye-I ( $\lambda_{\max} \approx 510$  nm) has a minor efficiency and is accompanied by a low glow intensity.

Conversely, modulated excitation of luminescence of Dye-I H aggregates from the absorption regions of Dye-I dimers ( $\lambda = 450$  nm) is not only accompanied by a more intensive glow of H aggregates, compared to the excitation from the absorption bands of molecular Dye-I, but also – for the porous glass matrix type A (with predominantly “small” mid-size pores) – by the appearance of an extra maximum of excitation at  $\lambda_{\max} \approx 470$  nm. It is for the first time that we have recorded the appearance of a new maximum of luminescence excitation of Dye-I H aggregates, which perhaps implies the existence of two types of H aggregates in the glass matrix depending on the dimensions of pores, since, in transition to porous glass matrix type C (with predominantly “large” pores), the spectrum of modulated excitation of anomalously retarded fluorescence of Dye-I H aggregates does not display an extra maximum in the absorption regions of dimers.

These results indicate that porous glass matrix type A has a predominant formation of Dye-I H aggregates, while in the solution this dye mostly forms J aggregates. In addition, the interaction of molecular Dye-I and its H and J aggregates forming in porous glass is minimised.

In the case of distributing the Dye-I solution in PVA in a porous glass matrix type A, it displays a fluorescence of molecular Dye-I, whilst no fluorescence of dimers. Consequently, PVA has an impact on the character of Dye-I adsorption, which results in the fact that the formation of Dye-I dimers in a PVA solution in a porous glass matrix type A is hindered. For the glass matrix type C the formation of Dye-I dimers in PVA occurs, while not being predominant, unlike in the case of Dye-I solution in isopropyl alcohol in porous glass matrixes of both types.

The spectrum of luminescence of Dye-II dissolved in isopropyl alcohol out of a porous glass excited with continuous and modulated light from the range 400–700 nm displays two fluorescent glow bands, including the fluorescence of anomalously retarded J aggregates and Dye-II molecules. It is important that

the spectrum of excitation of phosphorescence of Dye-II molecules displays a maximum in the absorption region of dimers, molecules and J aggregates.

When distributing Dye-II dissolved in isopropyl alcohol in porous glass matrix type A no fluorescence of Dye-II J aggregates is found, while only phosphorescence occurs, including anomalously retarded fluorescence of Dye-II. Thus, porous glass matrix type A hinders the formation of Dye-II J aggregates, while they are only formed in porous glass matrix type C.

The glass matrix has a greater impact on the aggregation of Dye-II in a PVA solution. In this case, porous glass matrix type A does not form any aggregates, the luminescence spectrum only having a fluorescence of molecular Dye-II. In porous glass matrix type C only the dimerisation of Dye-II takes place, with no formation of J aggregates.

## 5. Conclusions

Based on our analysis, the following may be concluded:

1. Porous glass has a substantial impact on the aggregation of a dye and, depending on the size of pores, may lead to complete disappearance of aggregation, even at a high dye concentration in the solution, which greatly widens the possible category of the dye used in laser.

2. The use of the matrix of the porous glass contributes to a change of the features of predominant dye aggregation. An example is the 1,1'-diethyl-2,2'-cyanineiodine which mostly forms J aggregates in isopropyl alcohol solution and PVA. It is for the first time that we have seen predominant formation of dye dimers in porous glass, while the formation of J aggregates was difficult.

3. If a dye solution displays an interaction between aggregates and molecules of dye which reduces the intensity of luminescence of the dye solution, the use of porous glass will minimise this interaction, thus to a large extent enhancing the intensity of luminescence of the dye. This is substantial in the use of the dye in lasers.

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