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Laser radiation intensity limiter based on polymethine dyes

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***Abstract*. The nonlinear optical properties of a number of polymethine dyes (PD 7005, 7006, 7031, and 7098) with a fixed polymethine chain are studied upon their excitation by the focused second-harmonic radiation from a nanosecond Nd : YAG laser. It is found that PD 7098 has the minimum linear absorption over a greater part of the visible spectrum and the strongest nonlinear reverse saturable absorption. The attenuation coefficient of the 100-MW cm^{-2} laser radiation in the ethanol solution of this dye is $K = 14$. The characteristics of a single-stage high-power-laser-radiation limiter consisting of two confocal lenses with a focal distance of 5.5 cm are optimised. The maximum values of attenuation coefficients measured in experiments are 420 (for the initial transmission $T_0 \approx 50\,\%$) and 170 ($T_0 \approx 70\,\%$). The singlet – singlet absorption cross sections of the dyes are estimated from experiments. The efficient laser radiation limiter considered in the paper features a broad spectral range in the visible region and a high service time.**

Keywords: *laser, nonlinear absorption, radiation intensity limitation, single-stage limiter, polymethine dye, absorption cross section.*

1. Introduction

The rapid development of high-power pulsed lasers in the last decades and their wide applications in various fields of human activity require the solution of the problem of dynamical protection of organs of sight and optical sensors. This problem is of obvious current interest due to a noticeable increase in the radiation intensity of laser range finders, target pointers, and similar devices operating within a broad spectral range. Although the leading countries have ratified the International Conventional Protocol on the Prohibition of Laser Weapons of Blinding Action (5 November 1999), the applications of lasers can cause severe diseases of the personnel and the destruction of the sensitive elements of optical sensors.

The dynamic limitation of the high-power laser radiation can be achieved by many methods, in particular, by using the modified schemes of conventional laser gates. In practice, passive limiters are more convenient, which contain organic (metalloorganic) and inorganic materials darkening under the action of high-power laser radiation or drastically increasing the scattering (and also focusing or defocusing) of the transmitted light. The materials used in optical limiters are the solutions, polymer blocks and films of organic dyes and fullerenes, as well as the suspensions of carbon and metal micro- and nanoparticles and nanotubes [1, 2].

An advantage of a limiter based on a dye (fullerene) solution is the possibility to 'restore' comparatively rapidly the region of laser action, while its typical disadvantage is that the working substance is coloured in its ground state, which distorts the sighting of the operator of the optical device or narrows down the region of the spectral sensitivity of the optical sensor. At the same time, virtually non-selective suspensions of carbon particles can form aggregate (especially upon irradiation), thereby changing considerably their optical properties. Solid-state limiters, in which the 'restoration' mechanism of the irradiated region does not work, are less durable than liquid limiters.

The two-stage 532-nm laser radiation limiter provided high attenuation coefficients $K = T_0/T = (2-3) \times 10^4$ {where T_0 is the linear (initial) transmission of the limiter and T is the transmission for the specified incident radiation intensity [3]}. In the first stage of the limiter, laser radiation was attenuated in the optical breakdown plasma in a cell with carbon disulfide CS_2, and in the second stage – in a cell filled with the solution of lead phthalocyanine PcPb in $CHCl_3$ or with the suspension of carbon microparticles. In a broader spectral region from 485 to 680 nm, the coefficient K (with the suspension of microparticles in the second stage) did not exceed ~ 1000.

Another disadvantage of this limiter is that it is single-acting, which seems unacceptable for real applications. As a whole, despite the interest of researchers in the problem of laser radiation intensity limitation, this drawback has not been eliminated so far.

For this reason, the extensive search is now under way for new working materials for optical limiters, which would have the required nonlinear properties, and also for efficient schemes of these limiters. One of the most low-threshold and

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efficient physical mechanisms of nonlinear attenuation of the radiation intensity in the limiter material is nonlinear reverse saturable absorption (RSA), which is based on the induced absorption from excited singlet or triplet states of the medium [4]. The necessary condition for the existence of the RSA mechanism is the excess of the induced absorption cross section σ_{ik} over the linear ground-state absorption cross section σ_{01} at the laser wavelength.

Among organic and metalloorganic compounds, in which the laser radiation intensity is limited due to RSA, we will point out phthalocyanines, fullerenes, substituted dicyaminomethylenepyrans, and polymethine (cyanine) dyes (PDs) [1, 5–10]. The first two classes of compounds limit the laser radiation intensity mainly due to triplet–triplet absorption, and the others – due to singlet–singlet absorption. The singlet–singlet mechanism provides, as a rule, a faster response of a medium. For this reason, PDs find wide applications for mode locking ultrashort-pulse lasers.

PDs with a long polymethine chain (PC) have characteristic IR absorption bands. As the PC length and the effective length of the π-electron system in the chromophore end groups of molecules are increased, these bands shift to the red, which can be used to match the absorption band of a PD with the laser wavelength.

Because some PDs have no absorption bands in the visible region, they possess a considerable advantage over many other materials used in laser radiation limiters. A significant reduction of linear absorption in PD limiters, making them virtually colourless in the ground state, provides a good compatibility of limiters with other elements of optical instruments.

One of the first studies of nonlinear absorption in PDs was devoted to analysis of the properties of compounds of two types [5, 6]. PDs of the first type had a PC consisting of 1–3 units (CH=CH groups) with different end groups (the maxima λ_{max} of the long-wavelength absorption band of these PDs were in the 550–750-nm range), while PDs of the second type had a more complicated fixed PC containing 3–6 units ($\lambda_{max} = 640 - 840$ nm).

In experiments on the limitation of the 532-nm second-harmonic intensity of a Nd : YAG laser ($T_0 = 70\,\% - 80\,\%$, the pulse duration $\tau = 10$ ns, and the output power density $W \leqslant 10$ J cm^{-2}), the best results were obtained with the solutions of PDs of the second type PD1 and PD3 ($\lambda_{max} = 750 - 780$ nm) in ethanol and polyurethanacrylate. The maximum values of K were ~ 4 for picosecond pulses and 5–7 for nanosecond pulses.

In z-scan experiments, the values of K close to those presented above were obtained. Note that the same values of K were obtained for nanosecond and picosecond laser pulses when the energy of nanosecond pulses was approximately four times higher than that of picosecond pulses. This was explained by the transition of molecules to the system of excited singlet levels appearing upon conformation isomerisation of molecules.

The photostability of PDs of the second type was considerably higher than that of PDs of the first type (because the PC was fixed in the former case). The photostability of PDs depended on the polarisation of laser radiation, being higher for circular polarisation than for linear.

According to [5], the energy density W_d of radiation transmitted through a limiter layer of thickness d is described by the expression

$$W_d = \frac{W\exp(-\alpha d)}{1 + (\sigma_{12} - \sigma_{01})W[1 - \exp(-\alpha d)]/(2\hbar\omega)}, \qquad (1)$$

where α is the linear absorption coefficient; σ_{12} is the absorption cross section for the $S_1 - S_2$ singlet–singlet transitions; $\hbar$ is Planck's constant; and ω is the radiation frequency. Expression (1) is valid for moderate values of W, when the saturation effect can be neglected.

The highest ratio $\sigma_{12}/\sigma_{01} \approx 200$ among all the known limiter materials was obtained in [6]. The region of values $\sigma_{12}/\sigma_{01} > 1$ where the RSA mechanism is possible was in the spectral range from 440 to 650 nm. The excited-state lifetime of molecules studied in the paper was 1 ns in ethanol and 3 ns in polyurethanacrylate, so that the characteristic response time of limiters did not exceed 1 ns.

In [7], the values of σ_{eff}/σ_{01} (σ_{eff} is the effective excited-state absorption cross section of molecules) were determined for five dialkylcarbocyanine PDs ($\lambda_{max} = 420 - 775$ nm). They increased from 0.3 to 5.3 with increasing the PC length from 1 to 3 units. This was also accompanied by the increase in λ_{max} and K. The maximum value of $K \approx 15$ was achieved for the solution of tricarbocyanine in dimethyl sulfoxide ($\lambda_{max} \approx 775$ nm, three-unit PC).

The noticeably attenuation of visible laser radiation was achieved in solutions of tricarbocyanines ($\lambda_{max} \approx 680$ nm, three-unit PC) in ethanol, propylene glycol carbonate (PGC), and a polymethyl methacrylate matrix [9, 10], where the maximum value of K was ~ 15 ($\lambda = 532$ nm, $T_0 = 70\,\%$, $\tau = 6$ ns, $W \leqslant 2.5$ J cm^{-2}). In z-scan experiments (the $F/10$ scheme, where $F = 10$ cm is the focal distance of a lens) with the solution of the most efficient PD No. 5 in PGC, the value $K \approx 70$ was achieved. No induced triplet–triplet absorption was observed in these experiments, which confirmed the assumption about a weak influence of triplet states on the laser intensity limitation.

The molecular photostability γ (the ratio of the number of decomposed molecules to the number of absorbed photons) of PD solutions measured upon laser irradiation at 532 nm was $\sim 1.5 \times 10^{-5}$ mol phot^{-1} and $\sim 1.2 \times 10^{-3}$ mol phot^{-1} – upon UV irradiation at 308 nm. After irradiation of a polymer limiter based on PD No. 5 by 100 laser pulses at 532 nm ($T_0 = 70\,\%$, $\tau = 6$ ns, $W = 1.2$ J cm^{-2}) with a pulse repetition rate of 1 Hz, its spectral properties and appearance did not change, although microscopic defects appeared on its surface after further irradiation. If the value of W was reduced to 0.6 J cm^{-2} (the focus of a lens with $F = 6$ cm was located at a distance of 5 mm behind the rear surface of a sample), no damage of the limiter was observed even after irradiation by 500 laser pulses. In this scheme, $K = 22$.

2. Objects and experimental results

We studied the nonlinear absorption in four new PDs 7005, 7006, 7031, and 7098 with a fixed seven-unit PC. The structural formulas of these dyes are presented in Fig. 1, where for comparison the structures of the most efficient PDs (PD3 and No. 5) from [6, 9] are also shown. The structure of PDs studied here and of PD3 is more complicated than that of PD No. 5. All the dyes in Fig. 1 contain the same counterion ClO_4^-.

One can see from Fig. 2 that all the dyes presented in Fig. 1 exhibit intense absorption in the near-IR region. The maximum extinction coefficient ε_{max} in the long-wavelength

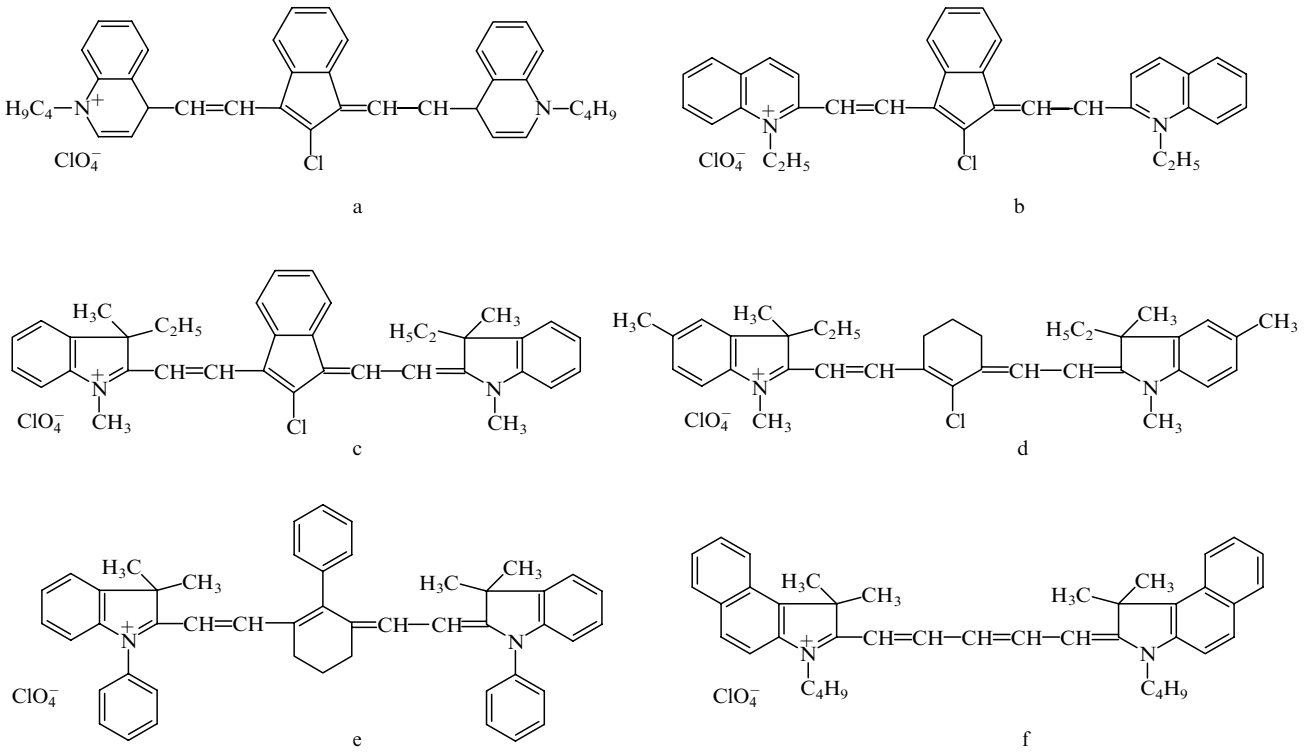

Figure 1. Structural formulas of PDs 7005 (a), 7006 (b), 7031 (c), 7098 (d), PD3 [5] (e), and PD No. 5 [9] (f).

absorption band is $\sim 2 \times 10^5$ L mol^{-1} cm^{-1}. The linear absorption for all the PDs (as for PD3) is weak over the entire visible wavelength range. The other intense absorption bands lie only in the spectral region below 400 nm.

A comparison of Figs 1 and 2 shows that the cyclisation of the central part of the PC with an orthophenylene bridge

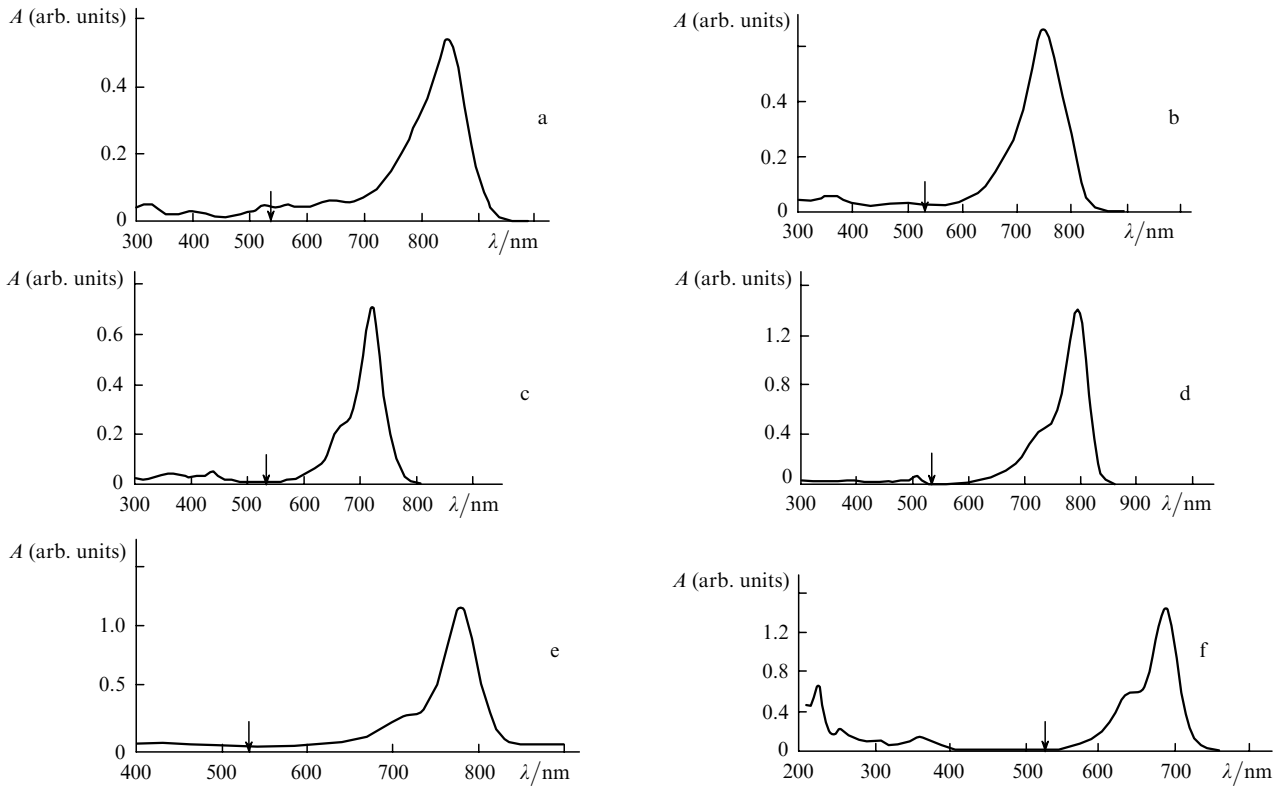

Figure 2. Absorption spectra of solutions of PDs 7005 (a), 7006 (b), 7031 (c), 7098 (d), PD3 [5] (e), and PD No. 5 [8] (f) in ethanol (a–d), polyurethanacrylate (e), and PGC (f). The arrows show the position of the exciting wavelength at 532 nm.

(PD 7006, 7008, and 7031) almost has no effect on the position of the absorption band of a PD with an unfixed PC. This is explained by the mutual compensation of the blue shift of the absorption band caused by the 3,5-substitution of the orthophenylene cycle and the red shift caused the 4-chlorine substituent. The substitution of a heterocycle at the nitrogen atom does not affect spectral characteristics, changing only the solubility of a dye. At the same time, the cyclisation of PDs with the saturated 3,5-trimethylene bridge caused a considerable red shift of the absorption band of PD 7098 with respect to the absorption band of PD 7098 having a close structure. Note also that dyes with the indolenine end groups are more stable photochemically than quinoline tricarbocyanines.

The nonlinear absorption in $\sim$ 1-mM ethanol solutions of PDs was studied in a 5-mm thick quartz cell upon excitation by 532-nm, 15-ns, 0.1-J second-harmonic pulses from a Nd : YAG laser ($T_0 = 70\,\%$). The laser radiation was focused with a lens with the focal length $F = 60$ cm. The aperture of a power meter measuring the laser radiation transmitted through the cell exceeded the laser beam diameter by a factor of ~ 4.

Figure 3 shows the dependences of the transmission of PD solutions on the exciting radiation intensity I. One can see that PD 7098, for which $K = 22$ for $I = 100$ MW cm^{-2}, has the highest nonlinear absorption, whereas $K = 2 - 3$ for the rest of the dyes. Note that a favourable situation for PD 7098 dye is weak linear absorption at the 532-nm excitation wavelength (the extinction coefficient is $\varepsilon =$ 400 L mol^{-1} cm^{-1}). Because the nonlinear attenuation of laser radiation by a molecule is determined by the ratio σ_{12}/σ_{01} of its absorption cross sections in the excited and ground states, in the case of small σ_{01} even weak induced absorption causes a considerable limitation of laser radiation.

For other dyes studied here (as for PDs investigated earlier in [9, 10]), linear absorption at 532 nm is substantially higher ($\varepsilon \geqslant 2000$ L mol^{-1} cm^{-1}). Therefore, to achieve efficient attenuation in these molecules, the excited-state absorption cross section σ_{12} for them should be considerably higher than that for PD 7098.

Table 1 presents the values of the molecular photostability γ of ethanol solutions of the dyes measured upon excitation at 532 nm. They vary from $\sim 4 \times 10^{-5}$ for PD 7005 to $\sim 10^{-7}$ mol phot^{-1} for PD 7031. Therefore, the photostability of the dyes increased with increasing the number of tightening groups in the molecular structure (Fig. 1). The stability of the dyes was noticeably higher after solution degassing. Note that all the PDs studied in the paper had the quantum yield of fluorescence below 0.1.

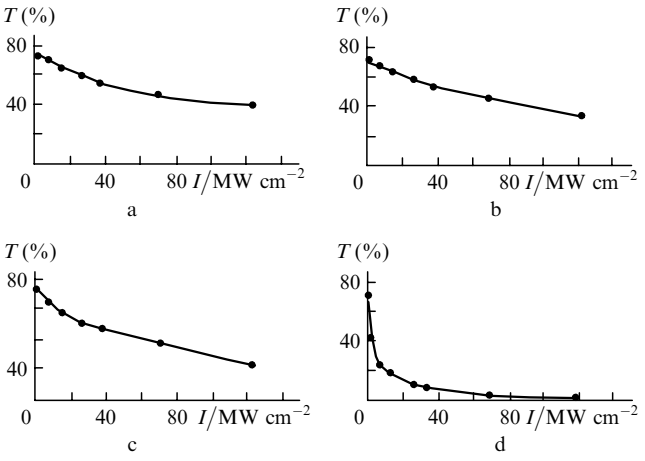

Figure 3. Dependences of the transmission T of the ethanol solutions of PDs 7005 (a), 7006 (b), 7031 (c), and 7098 (d) on the exciting radiation intensity I.

Table 1. Experimental molecular photostabilities γ and calculated absorption cross sections for transitions between the ground and the first excited singlet states (σ_{01}) and between the first and second singlet states (σ_{12}), and the ratio σ_{12}/σ_{01} for PDs ($\lambda = 532$ nm).

Dye	γ/mol phot^{-1}	$\sigma_{01}/10^{-18}$ cm^2	$\sigma_{12}/10^{-17}$ cm^2	σ_{12}/σ_{01}
PD 7005	4×10^{-5}	8	1.2	1.5
PD 7006	1.8×10^{-6}	8	1	1.25
PD 7031	10^{-7}	8	1.2	1.5
PD 7098	8×10^{-7} **1.2×10^{-8}**	1.5	4 (3)	25 (20)

Note. The value of γ in bold corresponds to a degassed solution; the values calculated by expression (4) are presented in parentheses.

3. One-stage limiter

Table 2 presents the characteristics of a single-stage confocal limiter, whose optical scheme is shown in Fig. 4, measured in our experiments. The limiter consisted of two confocal lenses L1 and L2 ($F = 5.5$ cm), an iris diaphragm D, and a quartz cell C with the ethanol solution of PD 7098. Quartz cells of thickness $h = 0.5 - 5$ cm could

Table 2. Attenuation coefficients K of the limiter based on PD 7098 obtained for different initial transmissions T_0, cell thicknesses h, and positions of the active medium with respect to lens L1 (the distance H).

T_0 (%)	h/cm	H/cm	K
48	1	0.8	37
		2	68
		3	152
		3.5	200
		4	405
50	2	0.8	49
		2	105
		3	210
		3.5	280
		4	420
51	5	0.8	86
		2	260
70	0.5	0.8	18
		2	33
		3	67
		3.5	109
		4	174
72	1	0.8	20
		2	36
		3	71
		3.5	103
		4	140
70	2	0.8	27
		2	49
		3	100
		3.5	126
		4	140

be moved along the optical axis of the limiter. Dye solutions were irradiated by 532-nm, 15-ns, 0.14-J pulses from a second harmonic Nd : YAG laser. The laser beam diameter was $d_b = 0.5$ cm. When the distance H between the cell and lens L1 was 4.5 cm, the input window of the cell was damaged, while in the absence of the cell the optical breakdown in air was observed in the lens focus.

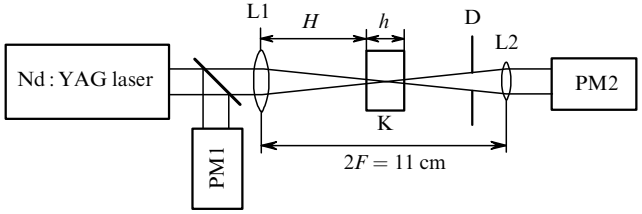

Figure 4. Scheme of a single-stage optical limiter: (L1, L2) spherical lenses; (D) iris diaphragm; (C) cell with the active medium of the limiter; (PM1 and PM2) calorimetric power metres KTP-2 or IMO-2N.

The maximum attenuation of laser radiation is achieved when the absorbing element of the limiter with the smallest thickness h is placed near the focus of lens L1. Such a limiter will have a low-energy operating threshold; however, the dynamic range of the limiter will be narrow because of its rapid damage at high incident radiation intensities. This effect can be enhanced at high pulse repetition rates due to thermal mechanisms already before the achievement of the radiation resistance threshold of quartz windows of the cell.

Therefore, the active element should be long enough to provide scattering of the absorbed energy in a large volume. At the same time, the radiation intensity over the entire length of the element should be sufficient to attenuate radiation efficiently.

One can see from Table 2 that for $T_0 = 70\,\%$, when $h = 0.5$ cm and a cell is located near the focal plane of lens L1 ($H = 4$ cm), the measured value of K is 174. This corresponds to rather stringent operation conditions of the limiter. The increase in the laser pulse energy by a factor of ~ 1.5 caused the damage of the limiter. In the case of less tight focusing, the limiter efficiency drastically decreased. As h was increased up to 1 cm, the operating conditions of the limiter slightly improved, the maximum value of K being reduced to 140 ($H = 4$ cm).

A limiter with $h = 2$ cm proved to be preferable. In this case, the attenuation coefficient increases somewhat and its maximum value was 140 ($H = 4$ cm). As H was decreased, the coefficient K decreased slower than in the first two cases, and for $H = 3$ cm it was equal to 100, which is a good value for a single-stage limiter with $T_0 = 70\,\%$. In this case, the laser energy could be increased by 3–4 times without the damage of the limiter.

A similar behaviour was observed for $T_0 \approx 50\,\%$. The best results were obtained for a cell with $h = 1 - 2$ cm. As h was increased up to 5 cm, the limitation efficiency considerably decreased because radiation was not sufficiently intense in a great part of the medium.

4. Discussion of results

The transmission T of a limiter due to nonlinear absorption averaged per pulse in the three-level scheme approximation upon nanosecond excitation can be written in the form

$$T(I) \approx \exp\{[-N_0(I)\sigma_{01} + N_1(I)\sigma_{12}]h\}, \quad (2)$$

where N_0 and N_1 are the concentrations of molecules in the ground S_0 and excited S_1 states, and $N_0 + N_1 = N_{tot}$ (where N_{tot} is the total concentration of molecules).

If collective effects are absent, the nonlinear transmission of the limiter material depends on the product of the concentration of molecules by the optical path. Therefore, in the case of a collimated laser beam, the transmission $T(I)$ for samples with different concentrations but the same T_0 (in a sufficiently broad range of concentrations N_{tot}) will be the same. Upon radiation focusing, the beam diameter d_b can change considerably along the path of radiation in an optical layer under study, resulting in a change in I. Therefore, nonlinear absorption can be substantially different for samples with the same values of T_0 but different h.

Taking into account that $T_0 = \exp(-\alpha d)$, expression (1) can be represented in a more convenient form

$$T(d) = \frac{T_0}{1 + (1 - T_0)(\sigma_{12} - \sigma_{01})W/(\hbar\omega)}. \quad (3)$$

For the case $\sigma_{12} \gg \sigma_{01}$ and $K \ll 1$, we obtain from (3) that

$$K \approx \frac{(1 - T_0)\sigma_{12}W}{2\hbar\omega}. \quad (4)$$

Therefore, the attenuation of laser radiation in the limiter material increases when its linear transmission is reduced, and the attenuation coefficient is proportional to the product of the absorption cross section for transitions between excited singlet levels and the incident radiation energy density.

Relations (3) and (4) can be used to estimate σ_{12} from the experimental dependences in Fig. 3, taking into accounts that $\sigma_{01} = 2.3 \times 10^3 \varepsilon / N_A$, where N_A is the Avogadro number. Table 1 presents the values σ_{01}, σ_{12} and σ_{12}/σ_{01} calculated by expression (3) for all the PDs studied here. For PD 7098, the values of σ_{12} and σ_{12}/σ_{01} calculated from (4), which differ little from those calculated from (3), are also given in parentheses.

One can see from Table 1 that the advantage of PD 7098 is explained by the fact that the values of σ_{12} and σ_{12}/σ_{01} for this dye are higher than for other dyes studied in the paper. Although the ratio σ_{12}/σ_{01} for PD 7098 is approximately an order of magnitude smaller than that for PD3, the values of K obtained for PD 7098 are considerably higher than those obtained for PD3 in [5, 6], despite the fact that we used substantially lower intensities I. Note that the values of T and K calculated from expressions (3) and (4) well agree with the experimental data presented above.

5. Conclusions

We have studied the nonlinear absorption of four photostable PDs excited by nanosecond second-harmonic pulses from a Nd : YAG laser. PD 7098 with minimal linear absorption in a greater part of the visible spectrum and the strongest nonlinear absorption has the best limiting properties. The attenuation coefficient K of laser radiation for the ethanol solution of PD 7098 is 14 for the incident radiation intensity $I = 100$ MW cm^{-2}. We have optimised the position and size of the medium of the efficient single-stage

limiter based on PD 7098. The maximum values of K for this limiter were 420 ($T_0 \approx 50\,\%$) and 170 ($T_0 \approx 70\,\%$). The singlet – singlet absorption cross sections were calculated for the PDs studied in the paper. The efficient limiter based on PD 7098 has a broad operating range in the visible spectral range and a long service life due to a high photostability of the dye.

PDs can be used for protecting biological tissues and organs of sight from the action of scattered (reflected) radiation of high-power medical IR lasers [11]. Some of these dyes can be virtually nontoxic and compatible with biological tissues, similarly to the known PD indocyanine green (cardogreen) [12].

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