An efficient solid-state laser based on a nanoporous glass – polymer composite doped with phenalemine dyes emitting in the 600 – 660-nm region

S.M. Dolotov, M.F. Koldunov, Ya.V. Kravchenko, V.B. Lugovoi, A.A. Manenkov, V.A. Petukhov, E.P. Ponomarenko, G.P. Roskova, T.S. Tsekhomskaya

Abstract. **The spectral and lasing parameters of solid laser elements made of a nanoporous glass – polymer composite doped with phenalemine dyes are studied. A high conversion efficiency (35 % – 40 %) of second-harmonic nanosecond pulses from a Nd^{3+} : YAG laser to the 600 – 660-nm region was obtained and a large operating life of the laser elements was demonstrated. A comparison of the parameters of solid laser elements with those of the liquid solutions of the same dyes showed that the conversion efficiency in solid laser elements is as good as that for the dye solutions.**

Keywords: *phenalemine dyes, nanoporous glass – polymer composite, solid laser elements.*

1. Introduction

The development of efficient solid-state organic dye lasers is one of the topical problems of laser physics and technology. To solve this problem, it is necessary to fabricate a solid matrix that possesses good mechanical, optical and other parameters and can be doped with dyes. It is also necessary to find dyes that can be easily introduced into the matrix and can efficiently convert the pump radiation to the radiation in the required wavelength region.

At present, optical polymers [1–3], sol–gel glasses [4–6], and a nanoporous glass–polymer (NPGP) composite [7–9] are used as solid matrices for doping with dyes.

It seems that the NPGP composite is an optimal matrix for using in laser elements [7–9]. It combines a high solubility of organic dyes in a polymer with a rigid quartz skeleton of the composite providing excellent mechanical properties. The composite has excellent thermo-optic and climatic parameters and can be easily machined by traditional glass polishing methods to obtain a high optical quality.

It was shown [9] that laser elements based on the NPGP composite doped with pyrromethene dyes efficiently convert the second harmonic of a Nd^{3+} : YAG laser to the spectral region between 540 and 600 nm. However, the conversion efficiency and the operating life of laser elements doped with these dyes decrease at wavelengths above 600 nm. For this reason, the search for dyes that can efficiently convert radiation at 532 nm to the spectral region above 600 nm is of great interest.

Note in connection with the choice of an efficient dye, that, according to the literature (see, for example, [2, 6]), the lasing efficiency of dyes in liquids is, as a rule, higher than that in a solid matrix. This fact is rather unexpected. The point is that a dye molecule acquires an additional 'rigidity' in a solid matrix, which results in the suppression of nonradiative relaxation and a corresponding increase in quantum yield of luminescence [10]. This suggests that the radiation conversion efficiency in solid matrices should be higher than that in liquids, whereas the reverse is observed in practice.

The aim of this paper is to fabricate a laser element based on the NPGP composite, which can efficiently convert the second harmonic of a Nd^{3+} : YAG laser to the 600–660-nm region, and also to compare the lasing efficiencies of dyes in liquid solutions and a solid composite.

2. Dyes studied

We studied here comparatively little-investigated phenalemine dyes P510, P512, and P640. These dyes exhibit intense luminescence in ethanol solutions and produce lasing at wavelengths above 600 nm upon pumping by the second harmonic of a Nd^{3+} : YAG laser [11]. The structural formula of a molecule of one of these dyes (P512) is shown in Fig. 1. Although the dyes belong to the same series, they are substantially different: the dyes P510 and P512 have a neutral form, whereas the dye P640 has an ionic form.

For comparison, we also studied the pyrromethene 650 dye (PM650), which exhibits luminescence and lasing at wavelengths above 600 nm.

S.M. Dolotov, E.P. Ponomarenko 'Alpha-Akonis' Research and Devices Association, Likhachevskii proezd 5, 141700 Dolgoprudnii, Moscow oblast, Russia;
M.F. Koldunov, V.B. Lugovoi 'Optronika' Research and Devices Enterprise, Post Box 3, 141700 Dolgoprudnii, Moscow oblast, Russia;
Ya.V. Kravchenko, A.A. Manenkov A.M. Prokhorov General Physics Institute, Russian Academy of Sciences, ul. Vavilova 38, 119991 Moscow, Russia;
V.A. Petukhov P.N. Lebedev Physics Institute, Russian Academy of Sciences, Leninskii prosp. 53, 119991 Moscow, Russia;
G.P. Roskova, T.S. Tsekhomskaya Institute of Silicates Chemistry, Russian Academy of Sciences, ul. Odoevskogo 24-2, 119155 St. Petersburg, Russia
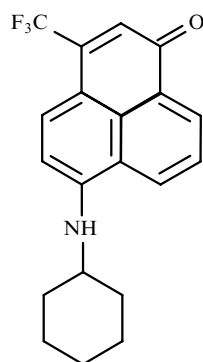
Figure 1. Structural formula of the phenalemine 512 dye molecule.

3. Spectroscopic studies

3.1 Experimental

The concentration of dyes in ethanol solutions and a monomer mixture was varied from 10^{-5} to 10^{-2} mol L^{-1} corresponding to the ultimate solubility. The absorption spectra of dyes were measured in the $190-900$-nm region with a Perkin-Elmer Lambda 5 spectrophotometer. The optical density of highly concentrated dye solutions was measured in thin Schott cells. Composite laser elements were of size $20 \times 10 \times 3.5$ mm, and their optical density was between 4 and 45 (at the maximum of the long-wavelength absorption band), so that we could not measure it directly. It was estimated by measuring the intensity of a weak short-wavelength absorption band.

The luminescence spectra were measured with a Perkin-Elmer 650-40 fluorimeter. At low concentrations of dyes in solutions (10^{-5} mol L^{-1}), luminescence was detected at a right angle to the exciting beam. At high concentrations of dyes in solutions and in the composite, luminescence was measured upon frontal excitation [10]. The luminescence of a dye was excited at the maximum of its main absorption band.

3.2 Results of spectral studies

The absorption spectra of each of the dyes did not change with increasing its concentration in solution up to the ultimate solubility. The careful measurements of the optical density at several wavelengths showed that the Bouguer–Lambert–Beer law

$$D(\lambda) = \lg(I_0/I) = \varepsilon(\lambda)CL,$$

is fulfilled in the entire concentration range, where $D(\lambda)$ is the optical density; λ is the wavelength; $\varepsilon(\lambda)$ is the extinction; C is the dye concentration; L is the sample thickness; I_0 and I are the intensities of the incident and transmitted radiation, respectively. This means that no dye associates are formed in ethanol and a monomer mixture over the entire concentration range up to the ultimate solubility. The maxima of the long-wavelength band of the dyes and some other parameters of their spectra in the monomer mixture are presented in Table 1.

The absorption spectrum of the P512 dye in a liquid monomer mixture is shown in Fig. 2, where the short-wavelength absorption band at 355 nm, used for estimating the concentration of the P512 dye in the NPGP composite is indicated by a letter A. As for other dyes, the ratio

Table 1. Spectral parameters of dyes in a monomer mixture.

Dye	λ_a/nm	λ_f/nm	λ_A/nm	$10^{-3}\varepsilon(\lambda_a)$	$10^{-3}\varepsilon(\lambda_p)$	$\varepsilon(\lambda_a)/\varepsilon(\lambda_A)$
P510	524	593	356	22	21	18
P512	533	578	355	25	25	42
P640	590	616	364	30	8,5	19
PM650	590	606	430	45	14	7.4

Note: λ_a, λ_f, λ_A and λ_p are the wavelengths of the maxima of the long-wavelength absorption band, luminescence band, of the short-wavelength absorption maxima and the pump, respectively; $\varepsilon(\lambda)$ is the extinction.

$\varepsilon(\lambda_a)/\varepsilon(\lambda_A) = 42$ for the P512 dye is constant over the entire range of concentrations. Assuming that this ratio is also constant for the composite, we calculated from the intensity of the A band the optical density of the composite at the maximum of the main absorption band at the pump wavelength 532 nm and the impregnation efficiency of the dye to the composite. The results of these calculations are presented in Table 2.
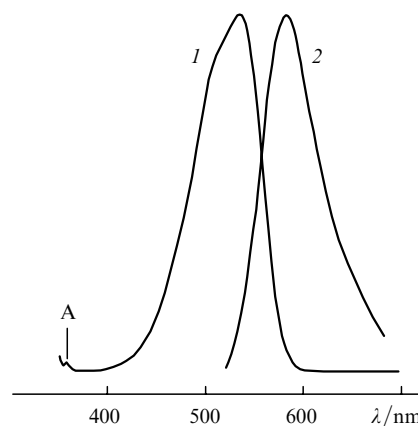

Figure 2. (_1_) Absorption and (_2_) luminescence spectra of the phenalemine 512 dye in the liquid monomer mixture. The spectral band used for estimating the dye concentration in the NPGP composite is denoted by A.

Unlike the absorption spectrum, the luminescence spectrum weakly depends on the dye concentration in solution. At high concentrations (about 10^{-3} mol L^{-1} for P512), a 'shoulder' appears at the long-wavelength wing of the luminescence spectrum, which shifts to the red with increasing the dye concentration. This shoulder is probably related to excimers appearing at high dye concentrations in solution [10].
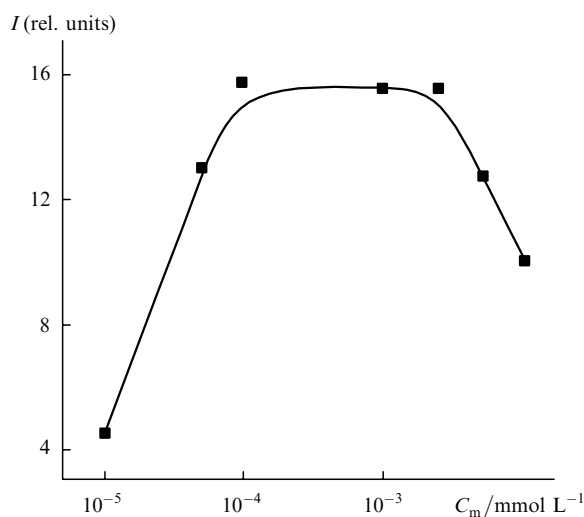
When the dye concentration in the monomer mixture exceeds 10^{-3} mol L^{-1} (3×10^{-3} mol L^{-1} for P512), the concentration quenching of luminescence is observed (Fig. 3). The picture is the same for the ethanol solution of the dye.

The luminescence spectrum in the NPGP composite is shifted to the red by $3-5$ nm compared to the spectrum in the monomer mixture, its shape being the same. At high dye concentrations in the composite, the luminescence spectrum exhibits a shoulder at the long-wavelength wing, as in the monomer mixture. The quenching of the dye luminescence in the composite occurs at the same concentration as in the monomer mixture; however, the luminescence intensity in the composite is higher.

Table 2. Parameters of the NPGP composite laser elements.

Dye	$C_m/$ mmol L^{-1}	$D(\lambda_A)$ measurement	$D(\lambda_p)$ estimate	$C_c/$ mmol L^{-1}	$\xi = C_c/C_m$
P510	6.8	1.24	22	8.0	1.2
	3.4	1.29	23	8.5	2.5
	1.7	0.89	16	5.8	3.4
	0.85	0.57	10	3.5	4.1
P512	10	1.01	43	11.8	1.2
	5	0.41	17	4.8	0.95
	2.5	0.24	10	2.8	1.1
	1.25	0.14	5.8	1.5	1.2
P640	2.5	4.2*	22	18.5	7.4
	1.25	3.1*	16	13.8	11
	0.5	1.46	7.8	6.5	13
	0.25	0.82	4.3	3.5	14
PM650	10	3.0*	12	5.8	0.6
	5	2.99	6.7	3.5	0.7
	2.5	1.26	2.9	1.5	0.60
	1.25	0.70	1.6	0.83	0.66

N o t e : C_m and C_c are the dye concentrations in the monomer mixture and a polymer embedded into glass pores; $D(\lambda_p)$ and $D(\lambda_A)$ are the optical densities at the pump wavelength and at the short-wavelength maximum; $\xi = C_c/C_m$ is the dye impregnation efficiency. The asterisks denote the values measured with a low accuracy.

Figure 3. Dependence of the luminescence intensity I on the concentration C_m of the phenalemine 512 dye in the monomer mixture.

4. Study of lasing parameters

4.1 Experimental setup

Laser elements were pumped by the second harmonic of the electrooptically Q-switched Nd^{3+} : YAG laser. We used longitudinal pumping: the pump radiation was directed to the laser element through a flat dichroic mirror whose transmission was 98 % at the pump wavelength 532 nm and

2 % in the range between 550 and 660 nm. The output mirror had the transmission 62 %. The pump laser beam diameter at the laser element was 1.3 mm, and the pulse repetition rate f was 3.67 and 33 Hz. At $f = 3.67$ Hz, the duration τ_p of the pump pulse, its energy E_p and intensity I_p were 5 ns, 2 mJ, and 30 M W cm^{-2}, and at $f = 33$ Hz – 7 ns, 0,7 mJ and 7.5 M W cm^{-2}, respectively. The pulse energy was measured with an accuracy of 10 %. The lasing wavelength λ_g was measured with an MCD-1 monochromator with an accuracy of ±1 nm.

4.2 Measurements performed and methods for data processing

We measured the lasing threshold J_g and the radiation conversion efficiency η of dye solutions and composite laser elements, as well as the operating life $N_{0.7}$ of laser elements. The conversion efficiency was defined by the expression $\eta = (E_g/E_p) \times 100\%$ (E_g is the laser pulse energy). It was measured by irradiating one region of the laser element by a small number of pulses ($N \ll N_{0.7}$). The operating life $N_{0.7}$ was measured as the number of pump pulses irradiating one region of the laser element after which the value of η became equal to 70 % of its initial level.

We calculated η by neglecting the loss of radiation energy caused by Fresnel reflection from the laser element surface. When the optical density of a laser element was low, a part of the pump-pulse energy was transmitted through the element, and we calculated η based on the absorbed energy rather than on the incident energy of the pump pulse.

4.3 Compositions and laser elements studied. Results of measurements

We studied a monomer mixture consisting of methyl methacrylate, a low-molecular additive, which is commonly used to enhance the stability of dyes in a bulk polymer and the polymer resistance to laser radiation damage [1], and one of the dyes (P510, P512, P640, and PM650). Laser elements made of the NPGP composite had a high optical quality with a homogeneous spatial distribution of a dye.

The parameters η and J_g for dye solutions in ethanol and the monomer mixture are presented in Table 3, and the parameters of composite laser elements are presented in Table 4.

5. Analysis of results

5.1 Doping of the composite with dyes

As follows from the data presented in Table 2, the impregnation efficiency of a dye to the composite $\xi = C_c/C_m$ (where C_m and C_c are the dye concentrations in the monomer mixture and a polymer embedded into the composite, respectively) changes in a broad range. It increases in a row of dyes P512, P510, and P640 as $0.95 - 1.2$, $1.2 - 4.1$, and $7.4 - 14$, respectively.

Nanoporous glass has a well-developed surface. Its specific value (for nanoporous glass, we used) is within $300 - 500$ m^2 cm^{-3}. The physicochemical properties of the nanoporous-glass surface are determined to a great extent by the presence of many hydroxyl groups [12]. These groups play an important role in adsorption processes proceeding during penetration of dye molecules into the glass. In addition, the strength of adsorption interaction between hydroxyl groups on the glass surface and the dye depends on

Table 3. Lasing efficiency of dye solutions.

Dye and solvent	C_m/mmol L^{-1}	L = 3.3 mm			L = 1.0 mm			L = 0.51 mm		
		$D(\lambda_p)$	η (%)	J_g/MW cm^{-2}	$D(\lambda_p)$	η (%)	J_g/MW cm^{-2}	$D(\lambda_p)$	η (%)	J_g/MW cm^{-2}
P512, monomer mixture	10	83	–	–	25	2.1	29	13	2.1	16
	5.0	41	T	–	13	15	4.2	6.3	23	3.7
	2.5	21	7	4.4	6.3	36	1.8	3.2	38*	1.8
	1.25	10	36	2.8	3.1	46	1.2	1.6	42*	1.3
PM650, monomer mixture	10	46	–	–	14	–	–	7.1	–	–
	5.0	23	–	–	7	5.2	14	3.5	11*	8.0
	2.5	12	–	–	3.5	11	7.5	1.8	13*	7.8
	1.25	5.8	19	7.5	1.8	21*	5.6	0.9	19*	2.8
P512, ethanol	10	86	–	–	26	–	–	13	–	–
	5.0	43	–	–	13	18	5.6	7	23	3.7
	2.5	21	19	6.7	7	35	2.2	3	40	2.0
	1.25	11	35	2.2	3	44	1.3	1.6	36*	1.8
PM650, ethanol	2.6	16	–	–	5	3.8	19	2.5	7.9*	16
	1.3	8	–	–	2.5	7.1*	12	1.2	4.4*	21

N o t e: C_m is the dye concentration in the monomer mixture; L is the cell thickness; $D(\lambda_p)$ is the optical density at the pump wavelength; the asterisks denote the samples through which the pump radiation is transmitted partially; a letter T indicates the appearance of parasitic radiation in the transverse direction; the dash denotes the absence of lasing.

Table 4. Lasing efficiency and the operating life of composite laser elements for different operating regimes.

Dye	C_m/mmol L^{-1}	$D(\lambda_p)$ ($\lambda = 532$ nm)	λ_g/nm	η (%)		J_g/MW cm^{-2}	$10^{-3}N_{0.7}$
				$f = 3.67$ Hz $E_p = 2$ mJ	$f = 33$ Hz $E_p = 0.7$ mJ		$f = 33$ Hz $E_p = 0.7$ mJ
P512	10	43	615	11	8.8	5.0	40
	5	17	604	34	29	1.7	34
	2.5	10	600	40	37	1.3	55
	1.25	5.8	595	42	38	1.1	30
P510	6.8	22	630	24	12	3.2	24
	3.4	23	628	23	11	2.8	4
	1.7	16	622	30	13	2.0	10
	0.85	10	614	31	14	2.0	4
P640	2.5	22	–	–	–	–	–
	1.25	16	658	16	6.8	7.5	115
	0.5	7.8	647	27	13	2.2	50
	0.25	4.3	642	35	20	1.8	16
PM650	10	12	630	10	6	10	6
	5	6.7	628	30	18	5.3	20
	2.5	2.9	625	27*	13*	3.8	13
	1.25	1.6	620	33*	16*	2.9	8

N o t e: C_m is the dye concentration in the monomer mixture; $D(\lambda_p)$ is the optical density at the pump wavelength; the asterisks denote samples through which the pump radiation is transmitted partially; the dash denotes the absence of lasing.

the polarity of the dye itself. For example, the ionic dye P640 has the maximum impregnation efficiency, as other ionic dyes (for example, rhodamine 11B, which we studied earlier [9]). Weakly polar dyes, such as PM650, P510, and P512, are not disposed to adsorption, which explains the lower degree of their penetration into the composite.

Upon doping of the composite with dyes, adsorption plays a role that is ambiguous from the point of view of the operation of the laser element. Adsorption prevents the formation of aggregates and, hence, prevents the luminescence quenching. Probably, this takes place in the case of P640 and rhodamine 11B. However, when a strong bond is

formed, a surface-dye complex can appear, whose properties drastically differ from those of the dye.

5.2 Lasing efficiency in solutions and composite

Comparison of the data presented in Tables 3 and 4 shows that the lasing efficiencies of composite laser elements and dye solutions coincide with the accuracy of measurements. One can see this from a comparison of the optimal values of η and the efficiencies obtained for liquid and composite laser elements with the same optical density.

It was reported many times in the literature that the radiation conversion efficiency of dyes in solutions is higher

than in polymer [2], sol−gel [6], or polycom [6, 13] matrices. It was found in [2] that $\eta = 30\%$ and 50% for rhodamine 6G in a polyurethanecrylate matrix and ethanol, respectively. The radiation conversion efficiency in the PM567 dye in liquids, modified polymethyl methacrylate, and a polycom matrix was $30\% - 40\%$, less than 10%, and less than 20%, respectively [13].

A decrease in the conversion efficiency was attributed to the formation of associates upon polymerisation, a decrease in the Stokes shift, and to the inhomogeneous broadening of the dye spectrum in matrices [2]. These changes are caused by a lower polarity of the matrix material and by the matrix shrinkage during polymerisation.

A lower polarity of the solution components facilitates the formation of aggregates. This process should affect most strongly ionic dyes (for example, P640). At the same time, associates will be formed only when the concentration of the dye in a monomer mixture is close to the ultimate solubility. The spectral studies of dye solutions showed that no associates were formed over the entire region of solubility, the concentration quenching beginning before the achievement of the ultimate solubility. Because the dye concentration in the monomer mixture intended for the fabrication of a composite was lower than the ultimate solubility, the formation of associates was probably ruled out. It is also possible that the formation of associates was prevented by adsorption of the dye on the nanoporous glass surface, as mentioned above.

A decrease in the lasing efficiency with increasing pulse repetition rate (Table 4) is caused by a change in the pulse energy. The lasing efficiency for a 0.7-mJ pulse and a pulse repetition rate of 3.67 Hz coincides, within the accuracy of measurements, to the lasing efficiency for a pulse repetition rate of 33 Hz presented in Table 4. Therefore, the increase in the pulse repetition rate from 3.67 to 33 Hz does not change the lasing efficiency.

5.3 Lasing thresholds in solutions and composite, and the operating life of laser elements

It follows from Tables 3 and 4 that the lasing threshold of all the dyes in the composite is lower than that in monomer mixtures and ethanol at the same optical density of laser elements. This is explained by the fact that the intensity of luminescence in the composite is higher than that in solution, i.e., the quantum yield ϕ of luminescence in the composite increases. It is known [14] that $J_p \sim \phi^{-1}$, in accordance with the experimental data.

Table 4 presents the results of the study of the operating life of laser elements for $f = 33$ Hz. According to these data, the dependence $N_{0.7}(C_m)$ has a maximum. It appears due to the presence of two different regimes of the dye degradation. In laser elements with low dye concentrations (less than $3 - 5$ mmol L^{-1}), the irradiated region was bleached, whereas in laser elements with high dye concentrations (above $3 - 5$ mmol L^{-1}), the irradiated region slowly darkened during the operation. Note that the dye was bleached at any concentration in the irradiated region of the laser element when the pulse repetition rate was 3.67 Hz. This suggests that irradiation of laser elements with high dye concentrations by laser pulses with a pulse repetition rate of 33 Hz results in the thermal decomposition of the dye and polymer because of a great heat release.

The operating life of laser elements presented in Table 4 is a technical parameter. A physical quantity characterising the stability of a dye in a matrix is the energy resource $W_{0.7}$ equal to the energy absorbed by a mole of the dye at which the radiation conversion efficiency is reduced down to 70% of its initial level. The estimate of $W_{0.7}$ from the data presented in Table 4 (for $f = 33$ Hz and $I_p = 7.5$ MW cm^{-2}) gives $W_{0.7} \simeq 15 - 10$ GJ mol^{-1} for all the dyes studied. The parameter $W_{0.7}$ was calculated for the dye concentration at which the value of $N_{0.7}$ is maximal. The random study of the operating life of some laser elements at a pulse repetition rate of 3.67 Hz showed that the value of $N_{0.7}$ and, hence, $W_{0.7}$ was $3 - 5$ times greater than that for $f = 33$ Hz.

5.4 Differences in the radiation conversion efficiency in liquid and solid laser elements

It follows from Tables 3 and 4 that composite laser elements produce laser emission at a very high optical density (although, of course, with a low efficiency). Thus, P512 has the conversion efficiency $\eta = 11\%$ in the laser element with the optical density $D(\lambda_p) = 43$ pumped by 2-mJ pulses, whereas the liquid laser element based on P512 in ethanol does not produce laser emission at $D(\lambda_p) = 43$, and in a monomer mixture at $D(\lambda_p) = 43$. At such high optical densities, liquid laser elements exhibit superluminescence (or, possibly, lasing) in the direction perpendicular to the pump beam.

5.5 Comparison of the lasing parameters of phenalemine dyes and PM650 dye

Pyrromethene dyes operate most efficiently upon pumping at 532 nm. However, this concerns lasing in the region between 550 and 570 nm. The conversion efficiency decreases at longer wavelengths. Thus, PM650 lasing at 650 nm has (under comparable conditions) the conversion efficiency of 30%. Phenalemine dyes produce laser emission at wavelengths above 590 nm: P512 $(595 - 615$ nm), P510 $(614 - 630$ nm), and P640 $(630 - 658$ nm). The lasing efficiency of a laser element doped with the P512 and P640 dyes is 40% and 35%, respectively. Therefore, the efficiency of phenalemine dyes is greater than that of PM650 lasing in the same spectral region.

6. Conclusions

Our study showed that phenalemine dyes embedded into a solid NPGP composite matrix produce efficient lasing in the region from 600 to 660 nm upon pumping by the second-harmonic nanosecond pulses from a Nd^{3+} : YAG laser. A comparison of the lasing parameters of these dyes in liquid solutions and a solid NPGP composite matrix showed that the lasing efficiency in composite laser elements is the same as that in liquids. However, the NPGP composite has better thermo-optic properties, which permit the operation at higher pulse repetition rates. An important feature of phenalemine dyes is their high solubility without the formation of associates in all the media studied in this work. This property allows one to fabricate efficient thin laser elements.

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