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Influence of various coumarin dyes on the laser performance of laser dyes co-doped into ORMOSILs

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ABSTRACT Several kinds of coumarin dyes (C440, C460, C500, C503 and C540A) were co-doped with perylene red (p-red) or pyrromethene 567 (p567) into VTES- and MTESderived organically modified silicates (ORMOSILs) respectively, by the sol-gel process. The effects of coumarin dyes and their concentration on the laser properties and photostabilities of p-red and p567 were studied and the mechanisms involved were discussed. At optimized coumarin dye concentration, the slope efficiency of co-doped p-red increased by a factor of 2 while only minor increases in the laser efficiency of p567 were observed. A broader tunable range with increased conversion efficiency and at least a 3-fold improvement in the photostabilities of p-red and p567 in the presence of coumarin family dyes have also been achieved. The mechanism responsible for the improvement in the laser performances and photostabilities of p-red and p567 resulted from the energy transfer between p-red/567 and coumarin dyes.

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1 Introduction

Compared with other commercially available multiwavelength laser sources, the dye laser has its obvious advantages such as a low-cost and a high conversion efficiency for a variety of applications, which require tunable high-power pulsed beams [1, 2]. Since the mid 1980s, intensive research efforts have been devoted to the solidification of organic laser dyes, which resolved problems such as toxicity and difficulty in maintenance of traditional liquid dye lasers [1–9]. Up to now, great progress had been made on the laser performance of the solid-state dye laser (SSDL) materials [7–13]. However, one of the main obstacles which has prevented the practical use of SSDL are parameters such as the laser efficiency, longevity and tunable range. It had been found previously that controlling the interactions between various dyes co-doped in solid hosts, i.e. the energy transfer between dyes, improvement in laser efficiency, laser lifetime and tunable range might be realized [14, 15]. In recent years, the interaction between dyes co-doped in solid hosts has generated much

interest. T.H. Nhung et al. and A.K. Sheridan et al. had observed the energy transfer from RB or P-orange to P-red in ORMOSILs or in organic thin films, respectively [16, 17]. M. Ahmad et al. had obtained normalized photostability as high as 350 GJ/cm^2 by co-doping p567 with C540A into PMMA, which increased the photostability of p567 by five times [7]. However, the interaction between dyes might also bring negative impacts on the performance of SSDL materials. W.N. Sisk et al. found that P546 accelerates the photodegradation rate of P597 and P567 [18]. In our pervious research, a 2 fold of enhancement in the laser efficiency of p-red and a four times improvement in the photostability of p567 had been observed in the presence of coumarin dyes, respectively [14]. In the case where coumarin, p567 and p-red were co-doped into MTES-derived ORMOSILs, a narrow linewidth laser output tunable in a spectral range as wide as 60 nm with at least a seven times higher energy conversion efficiency was observed [15], which validates the effectiveness of the energytransfer dye lasers (ETDL). The energy transfer mechanism between coumarin dyes and p567 or p-red under the excitation of a 532 nm laser was suggested to be the triplet-state quenching of p567 or p-red by coumarin dyes, thereby the excited coumarin dye molecules could transfer energy to the groundstate p567 or p-red dye molecules, resulting in increased fluorescence intensity, laser efficiency and laser lifetime of the latter two dyes.

However, in our previous works, only two kinds of coumarin dyes, C440 and C500 had been co-doped with p567 or p-red, nor had the concentration of the coumarin dyes been optimized, which had a large influence on the performances of ETDL. In this work, five kinds of coumarin dyes, C440, C460, C500, C503 and C540A were adopted to co-dope with p-red or p567 into solid-state matrices by the sol-gel process. The laser efficiency, photostability and tunable properties of p-red and p567 were also investigated. The effects of coumarin dye concentration on the laser performances of p-red and p567 were studied and the mechanisms involved were also discussed.

2 Experimental details

2.1 *Preparation of the dye doped ORMOSILs*

The laser dye doped methyltriethoxysilane (MTES)- and vinyltriethoxysilane (VTES)-derived samples were prepared by hydrolysis-condensation of the MTES and VTES precursors under acid-catalyzed hydrolysis and basic-catalyzed condensation. The laser dyes, C440, C460, C500, C503 and C540A were co-doped with p567 or p-red into MTES- or VTES-derived ORMOSILs at various initial concentrations respectively. The initial concentration of p567 and p-red in all the samples were kept constant at 1.0×10^{-4} mol/l and 5.0×10^{-5} mol/l respectively whereas the initial concentration of the coumarin dyes varied. When co-doped with p567, the initial concentration of coumarin dyes was given the values 2.5×10^{-4} mol/l, 5.0×10^{-4} mol/l and 7.5×10^{-4} mol/l, corresponding to molar ratios between coumarin and p567 of $2.5:1$, $5:1$, and $7.5:1$ respectively. These samples were labelled as coumarin : $p567$ (2.5 : 1), coumarin : $p567$ (5.0 : 1) and coumarin : $p567$ (7.5 : 1), respectively. The coumarin and p-red co-doped samples were labelled following a similar rule. Hydrolysis was performed under acid catalysis with HCl as the acid catalyst and ethanol as the solvent. The pH value of the mixed solution was adjusted to approximately 2. The initial molar ratio of MTES (or VTES) : ethanol : water was 1 : 3 : 3. After several hours of hydrolysis, a small amount of amine-modified silane N-(3- (triethoxysily)-propyl)-ethylenediamine) was added to neutralize the acidity of the sol thereby increasing the condensation reaction rate. After several more hours of stirring, the obtained sol was sealed into cuvettes and placed into an oven at 40° C for drying and aging. The dye doped MTES- and VTES-derived ORMOSILs could be machine or hand polished. The net thickness of all the samples was kept nearly the same, at about 7 mm.

2.2 *Measurements*

The laser performances such as the slope efficiency and the laser threshold of the dye doped ORMOSILs were investigated with the laser cavity which has been reported previously in detail [6]. A longitudinal plano-plano configuration was adopted, consisting of a dichroic mirror with a high transmission at 532 nm and a high reflectance from 570 nm to 650 nm, and an output coupler with 60%–70% transmission at the laser wavelength. The cavity length was 3.5 cm. The laser profiles were taken by a SP 750 monochromator followed by a CCD camera (Princeton) with the resolution at about 0.1 nm.

In this work, the photostabilities of p567 and p-red in the presence and absence of various coumarin dyes were investigated by a method that has been reported previously [19]. In detail, a 30 W continuous ultraviolet (UV) lamp with a peak wavelength at 254 nm was adopted as the excitation source and the fluorescence spectra of p567 or p-red were measured after various exposure times, and thus the photostabilities of the laser dyes in each case could be determined and compared. The photoluminescence (PL) spectra were taken by a Hitachi 850 fluorescence spectrometer. The scan speed was 60 nm/min and the slit width was 1.0 nm.

3 Results and discussions

The main purpose of this work was to develop a dye laser with a higher efficiency or an extended tunable range. The laser performances such as slope efficiency and the laser threshold of p-red doped in VTES-derived ORMOSILs in

the absence and presence of various kinds of coumarin dyes, and their dependence on the concentration of each kind of coumarin dye were also investigated, as shown in Table 1. Higher laser efficiencies and a lower laser threshold of p-red in the presence of coumarin dyes were observed in nearly all cases as compared with those of p-red in the absence of coumarin dyes.

However, the improvement in the laser efficiency of p-red by the presence of coumarin dyes varied sharply, depending on the kind of coumarin dyes and their concentration. In the case of C460, the laser efficiency of p-red increased with the increase of C460 concentration and reached its maximum at the C460 concentration of 1.25×10^{-4} mol/l, as is shown in Fig. 1. Beyond this C460 concentration, the laser efficiency of p-red decreased. In other words, increasing the coumarin concentration up to a particular value will not in any way enhance the laser efficiency of p-red.

The highest laser efficiency of p-red obtained in this work was 15.5% when C540A was introduced at the concentration of 2.5×10^{-4} mol/l, as shown in Fig. 2. Compared with that of p-red in the absence of coumarin dyes, at least a 1-fold improvement in the slope efficiency of the p-red was observed in this case.

FIGURE 1 The slope efficiency of p-red doped in VTES-derived OR-MOSILs in the absence and presence of C460 dye. (\blacklozenge) : C460 $(5.0 \times 10^{-5} \text{ mol/l})+$ p-red; \triangle : C460(1.25 × 10⁻⁴ mol/l)+p-red; \triangle : C460 $(2.5 \times 10^{-4} \text{ mol/l})+$ p-red; ■: C460(3.75 × 10⁻⁴ mol/l)+p-red; □: p-red)

Dye pair (molar ratio)	Slope efficiency (%)	Laser threshold (μJ)	Tunable range (nm)	Conversion efficiency (%)
$C440:$ p-red $(10:1)$	11.8	74.8		
$C440:$ p-red $(20:1)$	11.7	70.4		
$C440: p-red (40:1)$	5.1	131.9		
$C460: p-red (1:1)$	7.8	91.3	$583.82 \sim 613.43$	
$C460$: p-red $(2.5:1)$	12.6	59.6	$585.10 \sim 613.12$	
$C460:$ p-red $(5:1)$	11.8	53.3	$587.65 \sim 611.34$	
$C460$: p-red $(7.5:1)$	11.0	53.0	$586.43 \sim 613.08$	
$C503 : p-red (1 : 1)$	14.0	49.6	$585.30 \sim 614.13$	0.52%
$C503:$ p-red $(2.5:1)$	12.2	49.6	$589.66 \sim 613.90$	
$C503 : p-red (5 : 1)$	11.4	61.8	$588.79 \sim 613.78$	
$C503:$ p-red $(7.5:1)$	10.6	67	$591.66 \sim 611.95$	
$C540A : p-red (5 : 1)$	15.5	46.8		
$C540A : p-red (10 : 1)$	13.7	54.5		
$C540A : p-red (15 : 1)$	10.1	65.2		
p-red	7.3	118.9	$599.49 \sim 613.25$	$< 0.2\%$

TABLE 1 Laser performance of p-red doped into VTES-derived OR-MOSILs in the absence and presence of various coumarin dyes

FIGURE 2 The slope efficiency of p-red doped in VTES-derived OR-MOSILs in the absence and presence of C540A dye. (A: C540A $(2.5 \times 10^{-4} \text{ mol/l})+p\text{-red}$; \triangle : C540A(5.0 × 10⁻⁴ mol/l)+p-red; **■**: C540A $(7.5 \times 10^{-4} \text{ mol/l})+p\text{-red}; \square$: p-red)

The laser performances of p567 doped in MTES-derived ORMOSILs in the absence and presence of various kinds of coumarin dyes and their dependence on the concentration of each kind of coumarin dye were also investigated, as shown in Table 2. However, unlike the p-red, enhanced laser efficiencies of p567 in the presence of coumarin dyes were only observed in some cases. As was reported in our previous research, a marginal improvement in the slope efficiency of p567 had been observed in the presence of C500 [14]. It was found that the slope efficiency of p567 could be increased by introducing C440, C500 and C540A, while no obvious positive effect of C460 and C503 on the laser performance of p567 was observed. The highest laser efficiency of p567 obtained in this work was 76.3% when C540A was introduced at a concentration of 2.5×10^{-4} mol/l, as shown in Fig. 3. Compared with that of p567 in the absence of coumarin dyes, only about 20% improvement in the slope efficiency of the p567 has been observed in this case.

The mechanism responsible for the improvement in the laser efficiencies of p-red and p567 in the presence of coumarin dyes has been revealed in our previous report [14, 15]. In detail, the triplet-state of p-red and/or p567 was quenched by ground-state coumarin dyes by a multi-

Dye pair (molar ratio)	Slope efficiency threshold (%)	Laser (μJ)	Tunable range (nm)	Conversion efficiency (%)
C440: p567 (2.5:1)	72.4	53.7	$542.35 \sim 567.10$	2.320
C440: p567(5:1)	74.7	47.0	$540.95 \sim 572.16$	2.200
C440: p567(7.5:1)	65.6	66.0	$540.34 \sim 578.25$	2.824
C460: p567(2.5:1)	62.4	52.0		
C460: p567(5:1)	63.2	56.7		
C460: p567(7.5:1)	58.4	66.3		
C500: p567(5:1)	\prime	\prime	$541.73 \sim 566.40$	
C503: p567(2.5:1)	63.7	49.0		
C503: p567(5:1)	60.1	52.7		
C503: p567(7.5:1)	55.7	55.2		
C540A: p567(2.5:1)	76.3	50.0	$541.47 \sim 571.63$	
C540A: p567(5:1)	52.3	51.4	$541.03 \sim 572.77$	
C540A: p567(7.5:1)	48.4	54.0	$540.77 \sim 574.07$	
P ₅₆₇	64.2	57.8	$542.34 \sim 566.93$	1.427

TABLE 2 Laser performance of p567 doped into MTES-derived OR-MOSILs in the absence and presence of various coumarin dyes

FIGURE 3 The slope efficiency of p567 doped in MTES-derived OR-MOSILs in the absence and presence of coumarin dyes. (A: C540A $(2.5 \times 10^{-4} \text{ mol/l})+p567$; \triangle : C540A(5.0 × 10⁻⁴ mol/l)+p567; **■**: C540A $(7.5 \times 10^{-4} \text{ mol/l})+p567; \square$: p567)

photon process. Thus the excited-state coumarin dyes could transfer energy to the ground state p-red and/or p567, which reduced the triplet state absorption loss and increased the absorption cross section of p-red and/or p567, and as a result, improved the laser efficiency of p-red and/or p567. It was found that the effect of various coumarin dyes on the laser performances of p-red and p567 varied sharply, depending on the dye pair combination, i.e. higher laser efficiencies of p-red in the presence of coumarin dyes were observed in nearly all cases while the slope efficiency of p567 could only be increased by introducing C440, C500 and C540A. It should also be noted that the laser output wavelength of p-red blue-shifted $6 \sim 11$ nm in the presence of coumarin dyes as compared with that of p-red in the absence of coumarin dyes, while less than 1nm blue-shifts of the laser output wavelength of p567 were only observed in the cases in which the laser efficiencies of p567 were enhanced. This proof was one of the typical phenomena of energy transfer in dye mixtures that had been extensively reported before [20–22].

It was suggested that the variation in the effect on the laser performances of p-red and p567 by various coumarin dyes could be illustrated from two aspects, based on the above mechanism. Firstly, the energy transfer efficiency between dye pairs and the improvement in the laser performances of p-red and p567 were dependent on the energy level compatibility between the dyes, i.e. the energy level compatibility between the triplet-state p-red/p567 and the ground state coumarin dyes together with the energy level compatibility between excited state coumarin dyes and ground-state p-red/p567. For instance, it had been observed in our previous report that a small absorption peak of C500 at about 920 nm was found which coincided with the triplet-state energy of p567 thus the possibility of triplet-state quenching of p567 by C500 could be confirmed [14]. It was suggested that the triplet-state quenching of p-red/p567 by coumarin dyes might be varied, depending on the absorption of coumarin dyes in the longer wavelength and the triplet-state energy of p-red/p567, which resulted in the energy level compatibility between the triplet-state p-red/p567 and ground state coumarin dyes. It should also be noted that the energy level compatibility between excited state coumarin dyes and ground-state p-red/p567 depended on the overlap between the emission spectra of coumarin dyes and the absorption spectra of p-red/p567, which had been illustrated extensively by other authors. It should be mentioned that the triplet-state yield p567 was much less that of p-red, thereby resulting in less excited-state coumarin dyes via the multi-photon process, and the relative absorption of p-red in $400 \sim 500$ nm was larger than that of p567 in the same region, thereby resulting in a larger overlap between the emission spectra of the coumarin dyes, as revealed in our previous report [14].

Secondly, the dye concentration was still one of the critical parameters which decided the energy transfer efficiency between dye pairs and the improvement in the laser performances of p-red and p567. This could be explained by the intermolecular distance between dye molecules and the longrange dipole-dipole energy transfer mechanism which was diffusion controlled. In other words, increasing the donor concentration up to a particular value might cause the reduction in the effective distance between the coumarin molecules and thereby cause an energy transfer to take place within the donor system itself. And it should also be noted that too high a coumarin concentration might influence the hydrolysis and poly-condensation process of the precursors and lead to more light scattering and optical loss. In this work, the optimized concentration of each coumarin dye relative to p-red and p567 was given, as shown in Tables 1 and 2.

In our previous research, tunable narrow linewidth laser output from dye doped ORMOSILs had been achieved by introducing a holographic grating as the intra-cavity dispersive element [6, 15, 23]. In this work, a similar longitudinal pumped laser cavity was also adopted to investigate the tunable laser properties of laser dye co-doped ORMOSILs. In detail, the optical arrangement of the cavity consists of a dichromic mirror, an 1800 lines/mm holographic grating set at a grazing incidence for beam expanding, and an output coupler for tuning. The cavity length was about 12 cm. Tuning is achieved by rotating the output coupler, which feeds back a specific wavelength for laser oscillation.

When solely doped, the tunable range of p-red was somewhat narrow, from 599.49 nm to 613.25 nm, with a conversion efficiency lower than 0.2%. In the presence of C460 and C503, the tunable ranges of p-red were broadened in the blue side, together with the increase in energy conversion efficiency, from 0.2% to 0.52%. It was also found that the tunable ranges of p-red were broadened in the blue side in the presence of C440, C500 and C540A, which were largely depended on the concentration of C440. In the case where the C440 concentration was 7.5×10^{-4} mol/l, about a 1-fold increase in the conversion efficiency of p567 was observed, as shown in Fig. 4. The full-width-at-half-maximum (FWHM) of the tunable laser output profile was 0.07 nm, which had approached the resolution limit of the detecting system, as shown in Fig. 4. It is believed that the linewidth of the tunable laser obtained in this work was comparable to that had which has been achieved in our previous work [22], i.e. 0.014 nm, which was measured by an F-P interferometer.

Although much progress had been made during the last few years, one of the main problems of solid-state dye lasers is still the photo-degradation of the laser dyes under irradiation [6–12]. In this work, the photostabilities of p567 and p-red in the presence and absence of coumarin dyes were in-

FIGURE 4 The tunable range of p567 doped in MTES- derived OR-MOSILs in the absence and presence of coumarin dyes (*inset*: laser output profile of the sample labelled as $C440: p567(2.5:1)$ at the wavelength of 563.96 nm). (▲: C440(2.5 × 10⁻⁴ mol/l)+p567; △: C440(5.0 × 10⁻⁴ mol/l) +p567; \blacksquare : C440(7.5 × 10⁻⁴ mol/l)+p567; □: p567)

vestigated by measuring the exposure time dependence of the fluorescence intensity of laser dyes under the excitation of UV irradiation. The fluorescence intensity of p567 in the absence and presence of coumarin dyes as a function of the irradiation time were obtained, as shown in Fig. 5. In the case of p567 solely doped in ORMOSIL, the PL intensity decreased with the increase of irradiation time, reflecting the permanent photo-degradation of p567. After 200 h of exposure, the PL intensity of p567 decreased to half of its initial value. After 1200 h of irradiation, the excited region was nearly colorless and its PL intensity approached zero. However, in the presence of C440, the PL intensity of p567 began to decrease after 600 h of irradiation and approached 50% after 821 h of irradiation. It is evident that the addition of C440 leads to at least a 3-fold increase in the photostability of p567. Similarly, the presence of C540A also improved the photostability of p567 about 3-fold, as shown in Fig. 5. The presence of C460 and C503 only brought minor improvement to the photostability of p567, less than 40%. These results were in accordance with that obtained from the laser properties investigation.

In this work, the photostabilities of p-red in the presence and absence of coumarin dyes were also investigated.

FIGURE 5 Exposure time dependence of the fluorescence intensity of p567 doped in MTES-derived ORMOSILs in the absence and presence of coumarin dyes. (\blacklozenge : C440(5.0 × 10⁻⁵ mol/l)+p-red; **△**: C460 $(5.0 \times 10^{-4} \text{ mol/l})+p567$; \triangle : C503(2.5 × 10⁻⁴ mol/l)+p567; **■**: C540A $(2.5 \times 10^{-4} \text{ mol}/1) + p567$; \Box : p567)

However, only marginal enhancement (less than 25%) of the photostability of p-red by co-doping coumarin dyes was observed, indicating that the presence of coumarin dyes would not largely improve the photostability of p-red. Even at optimum donor dye concentrations, the laser lifetime of p-red in the presence of p567 and C440 was only improved by 50%, as revealed in our previous report [15]. This phenomenon might be explained by the photo-degradation mechanism of p-red in which the photo-chemical reaction related to the triplet-state of p-red did not play a major role. This was also in accordance with the results that reversible photoreduction was suggested to be the major component of the photo-degradation mechanism for perylene di-imide dyes, which was reported recently by other authors [24].

4 Conclusion

Laser dyes p-red and p567 were co-doped with various coumarin dyes into MTES- and VTES-derived OR-MOSILs by the sol-gel process, respectively. Energy transfer from coumarin dyes to p-red and p-567 had been observed and led to improvement in the laser performances of p-red and p567. The effects of coumarin concentration on the laser performances of p-red and p567 were investigated and the mechanism involved was discussed. At least a 1-fold increase in the slope efficiency of p-red and a marginal increase in the slope efficiency of p567 in the presence of coumarin dyes were observed. The tunable laser properties of p-red and p567 in the presence of coumarin dyes were also improved resulting in a broader tunable range and higher conversion efficiency. The laser dyes co-doped materials as ETDL are very promising for practical use.

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