

Laser performance of some polymeric-state laser dyes in transversely resonating cavity

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Abstract-Pyromethene laser dyes (PM567 and PM597) and Rhodamine 6G (R6G) were doped in 2-hydroxy ethyl methacrylate (HEMA) and methyl methacrylate (MMA) to form copolymer host of composition (1:1; v/v). optical and photo-physical properties such as; the oscillator strength (f), the transition dipole moment μ_{12} , the attenuation length $\Lambda(\lambda)$, the energy of fluorescence (Ef), radiative decay rate (Kr) and intersystem crossing rate (Kisc) showed that, R6G in polymeric host has the highest values which gave indication of its higher laser performance than other PM's dyes. 2×10^{-3} mol/L was the optimum concentration of the laser dyes in this polymer host. At this concentration, PM567, PM597 and R6G have laser performance values (by transversely resonating cavity) 51.1%, 52.0% and 54.3% respectively. Also, the photostability of the optimum concentration of the three laser dyes in solid host was studied.

Key words: PM567, PM597, R6G, transversely resonating cavity, laser performance, photostability.

1 INTRODUCTION

An advantage of a solid-state dye laser over its liquid counterpart is the ability to engineer a rugged and compact system by eliminating the dye flow hardware that is necessary in a liquid system. In addition, by inserting different samples, the laser wavelength range can be altered quickly without the risk of contamination with previous dye residuals. The storage and disposal is also easier to accomplish with a solid gain media than with the liquid counterpart. In recent years, the implementation of organic dye molecules into the solid matrix have resulted in significant advances towards the development of practical tunable solid-state dye lasers [1-15]. An ideal host matrix should possess good optical transparency in the region of absorption and emission of the dopant dye.

It should possess thermal and mechanical stability and time durability (longevity) and it should not have chemical and photo-physical interaction with the dye. On the other hand, solid state dye lasers have important applications especially in cardiology, dermatology, urology, underwater communications and remote sensing of atmospheric contaminants devices [16], a lot of work was devoted to incorporate different dyes into solid matrices in order to obtain solid-state laser emission from UV to the near IR light range [17-21].

confined in the cavity reflects multiple times producing standing waves for certain resonance frequencies. The standing wave patterns produced are called modes which are longitudinal modes and transverse modes. Laser performance of the most solid state laser dyes was carried out by using resonating cavity or optical resonator which is an arrangement of mirrors that surrounding the gain medium and providing feedback of the laser. Rhodamine and Pyromethene laser dye families are considered the benchmark in laser performance because they show very interesting and encouraging

photo-physical properties, high molar absorption coefficient and a fluorescence quantum yield, in some cases, near unity. Several factors contribute to their good laser performance such as: low triplet-triplet absorption capacity at the lasing spectral region, which reduces the losses in the resonator cavity; a poor tendency to self-aggregate in organic solvents, avoiding the fluorescence quenching of the monomer emission by the presence of H-aggregates in highly concentrated solutions. Also their high photostabilities improve the lifetime of the laser action. Owing to these properties, rhodamines and PM dyes have been successfully incorporated into different solid matrices specially to develop solid-state syntonizable dye lasers [22]. In this paper, We have carried out comparative laser performances study of PM567, PM597 and R6G dyes, doped in HEMA/MMA solid host matrices, using pulsed Nd:YAG (532 nm) as pumping laser in homemade transversely resonating cavity. Also, a comparison and description of the optical and photo-physical properties of the three dyes doped in HEMA/MMA solid host matrices were assessed.

2 EXPERIMENTAL PARTS

2.1 Materials

Rhodamine 6G (M. Wt. 479.01, Aldrich), PM567 (M. Wt. 318.22, Exciton Inc.), PM597 (M. Wt. 374.32, Exciton Inc.) were used without further purification. The purity of the dyes were found to be >99%, as determined by spectroscopic and chromatographic methods. Methyl methacrylate (MMA, M. Wt. 100.12, Aldrich) and 2-hydroxy ethyl methacrylate (HEMA, M. Wt. 130.14, Aldrich) were chosen as the monomer component of polymer host. These monomers were purified from their inhibitors by standard methods. 2,2-azobisisobutyronitrile (AIBN, 98%, Aldrich) was recrystallized in ethanol before use.

2.2 Preparation of polymeric laser dye samples:

The polymer samples were prepared as follows: 3g/L of AIBN was dissolved in freshly purified HEMA/MMA (1:1

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v/v) comonomer. The solution was placed in an ultrasonic bath for a period ~10 min until the initiator completely dissolved. Then laser dyes (PM567, PM597 and R6G) were added to obtain the desired concentrations and allowed to dissolve in the ultrasonic bath for up to 20 min. The solution then was poured in Pre-cleaned polymerizing tubes. Bulk polymerization was carried out in a temperature controlled oven; the temperature is maintained at 60°C for 7 days and then it was reduced in steps of 10°C per day until room temperature was reached. Doped copolymer host material of HEMA/MMA with laser dyes (PM567, PM597 and Rh 6G) of different concentrations (2x10⁻⁴, 4x10⁻⁴, 6x10⁻⁴, 8x10⁻⁴, 1x10⁻³, 2x10⁻³ mol/L), (4x10⁻⁴, 6x10⁻⁴, 1x10⁻³, 2x10⁻³, 4x10⁻³ mol/L) and (2x10⁻⁴, 6x10⁻⁴, 1x10⁻³, 2x10⁻³ mol/L) respectively were prepared. The sliced (2mm thickness) and the rods (2 cm length, 1 cm diameter) were ground and polished to the optical quality required.

2.3 MEASUREMENTS

2.3.1 OPTICAL MEASUREMENTS:

Absorption and excitation–emission spectra were measured by Camspec M501 (UV–Vis) Spectrophotometer and PF-6300 Spectrofluorometer respectively.

2.3.2 HOMEMADE TRANSVERSELY RESONATING CAVITY EXPERIMENT:

Optical laser cavity consisted of a flat aluminum mirror with 90% reflectivity at the laser wavelength and the output coupler with 50% transmission of the laser wavelength between (550-650 nm). The optical cavity length was about 6cm. Nd: YAG laser beam was expanded by quartz concave lens with focal length f=-10cm, and then line focused onto the laser dye rod using a quartz cylindrical lens of focal length f= 10 cm (2 cm line shape was focused on the length of the dye laser rod). The fluorescence emitted from rod was resonated transversely in optical laser cavity. The input and output energy of laser was measured by the Gentec power meter (model: QE50).

2.3.3 PHOTOSTABILITY MEASUREMENT:

The photostability effect of 2x10⁻³mol/L (PM567, PM597 and R6G) dyes doped (HEMA/MMA) polymeric host material were studied by focusing the pumping Nd-YAG laser beam, by quartz cylindrical lens, onto the dye rod. The laser repetition rate was kept at 10Hz and the energy was kept constant at 10 mJ . The output energy was measured continuously after number of pulses (~ 1800 pulses from Nd-YAG laser incident on dyes, using Gentec energy meter (model: QE50).

2.4 METHODS OF CALCULATIONS:

Photo-physical parameters of the three laser dyes in polymer host were determined such as; the molar extinction coefficient (ε) as given by BEER's law:

$$\log(I_0/I) = \epsilon.c.d \tag{1}$$

Where, I₀ is the intensity of the light before it encounters the sample. I is the intensity of the light emerging from the sample, c is the concentration in moles per liter, and d is the path length in centimeters. The absorption and emission cross sections σ_a and σ_e (cm²) respectively, are then calculated according to the following formula [23]:

$$\sigma_a = 0.385 \times 10^{-20} \epsilon \tag{2}$$

and

$$\sigma_e = \frac{\phi_f \lambda_e^4 E(\lambda)}{\pi 8 c \tau_f n^2 \int E(\lambda) d(\lambda)} \tag{3}$$

Where λ_e is the emission wavelength, n the refractive index of the solvent, c is the velocity of light, τ_f is the fluorescence life time, ∫E(λ) dλ is the normalized fluorescence spectrum and φ_f is the quantum yield of the dyes (defined as the ratio of the number of photons emitted to the photons absorbed). φ_f was estimated by comparing the absorption and emission spectra to those of a known standard φ_x according to the relation [24].

$$\frac{\phi}{\phi_x} = \frac{S h_x}{h S_x} \tag{4}$$

Where, h is the absorption peak height; S is the area enclosed by the emission curve and wavelength axis. In determination of fluorescence quantum yields φ_f(s) (determined relative to the R6G laser dye in ethanol as reference) care was taken to keep the concentration of all the samples at levels low enough to avoid re-absorption of the emitted photons [25,26].

From the quantum yield, absorption and emission spectra, fluorescence lifetime τ were calculated and tabulated using expression [27]:

$$\frac{1}{\tau} = 2.88 \times 10^{-9} n^2 \phi_f^{-1} \frac{\int F(\nu) d\nu}{\int \nu^{-3} F(\nu) d\nu} \int \frac{\epsilon(\nu)}{\nu} d\nu \tag{5}$$

Where ν is wave number (in m⁻¹), F(ν) is the emitted fluorescence intensity, and n is sample refractive index. Based on absorption and emission spectra, in which the values of wavelength (λ) were converted in wave number (ν: λ⁻¹). Values of oscillator strength are calculated using equation (6) [28]:

$$f = 4.32 \times 10^{-9} \int \epsilon(\nu) d\nu \tag{6}$$

The transition dipole moment (μ₁₂) from ground to excited state was calculated by using the equation (7), f is related to the transition moment and the Einstein coefficient by the following expressions:

$$f = \frac{4\pi\nu m_e \mu_{12}^2}{3\hbar e^2}$$

or

$$\mu_{12}^2 = 2.36 \times 10^{-51} \times f \times \lambda \tag{7}$$

Where ν is the optical frequency at the maximum absorption, m_e and e are the mass and electrical charge of an electron, all in The International System of Units (SI). A Debye (D) is the traditional non-SI unit of dipole moment. The conversion between D and SI units is

$$1D_p = 2.36 \times 10^{-30} \quad (\text{coulomb.meter})$$

The energy yield of fluorescence (E_f) calculated by:

$$E_f = \phi_f \lambda_A / \lambda_f \quad (8)$$

where ϕ_f is the fluorescence quantum yield, λ_A, λ_f the maximum absorption and fluorescence wavelength. The attenuation length $\Lambda(\lambda)$: the distance at which the original light intensity reduced to $(I=I_0 / e)$ given by the equation (9) where (e) Euler's number ($e \approx 2.7$) [29, 30]:

$$\Lambda(\lambda) = \frac{1}{\epsilon(\lambda)c \ln 10} \quad (9)$$

Where, $\epsilon(\lambda)$ is the molar extinction coefficient, (c) the molar concentration.

The intersystem crossing rate constant (k_{isc}) is related to the quantum fluorescence yield ϕ_f for $(\phi_f \sim 1)$ by the approximate relationship [31].

$$K_{isc} = \frac{(1-\phi_f)}{\tau_f} \quad (10)$$

3 RESULTS AND DISCUSSION:

The study of the electronic spectra and photo-physical parameters of PM567, PM597 and R6G laser dyes in polymeric environment provide useful informations about the intramolecular interactions of the dye molecules and the intermolecular interactions between dye molecules and host material. Figures (1a-c) represents the absorption spectra of the three laser dyes as a function of dye concentrations.

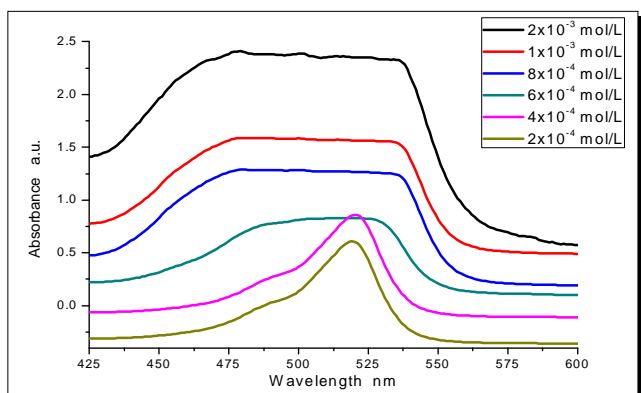


Figure (1a): Absorption spectra of PM-567 in HEMA/MMA copolymer as a function of dye concentration.

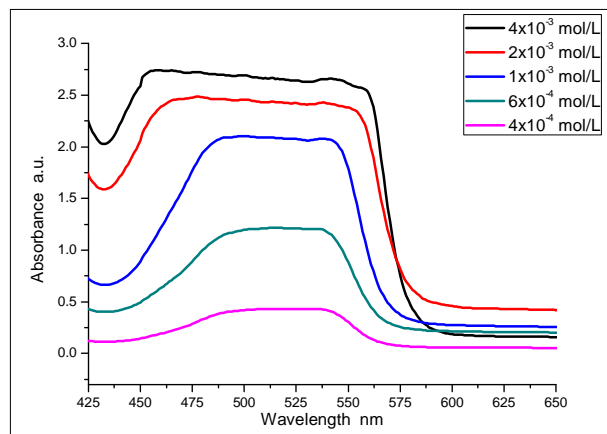


Figure (1b): Absorption spectra of PM-597 in HEMA/MMA copolymer as a function of dye concentration.

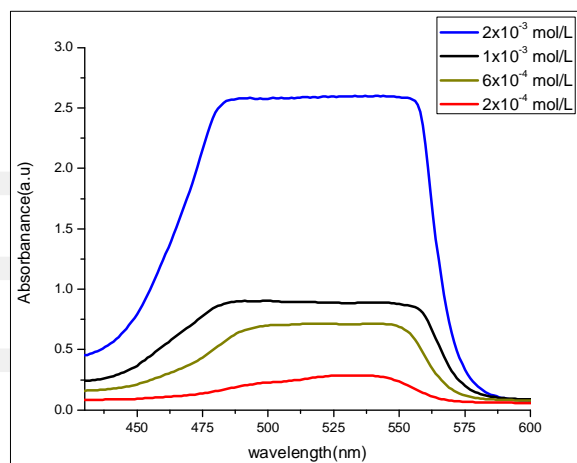


Figure (1c): Absorption spectra of R6G in HEMA/MMA copolymer as a function of dye concentration.

It was clear from absorption spectra of the three dyes as a function of dye concentration that, increasing the dye concentrations leads to cluster of dye molecules as dimer and excimer resulting in broadening in absorption spectra. These dimer and excimer formations have inherent effect on nonlinearity of fluorescence spectra of dyes as a function of dye concentration as shown in figures (2a-c and 3). This nonlinearity of fluorescence spectra is attributed to inner filter effect. Inner filter effect as a function of high concentration of dyes is divided into the "primary inner filter effect", which is caused by the absorption of exciting light such that a less intense light flux reaches each subsequent layer of the host than the previous one, and the "secondary inner filter effect", which is caused by the reabsorption of fluorescence [32]

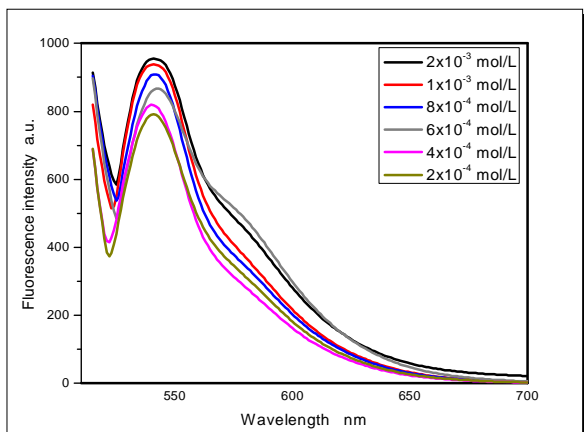


Figure (2a): Emission spectra of PM567 in HEMA/MMA copolymer as a function of dye concentrations.

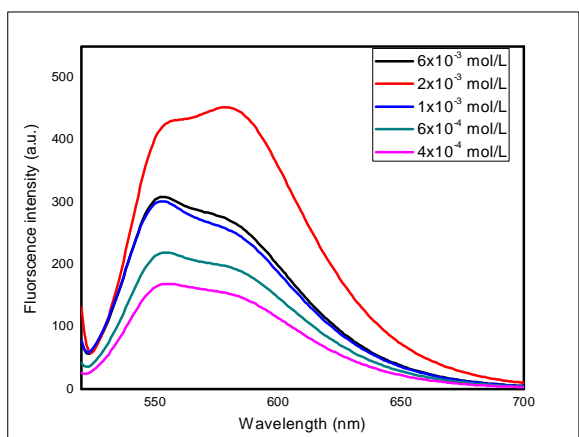


Figure (2b): Emission spectra of PM597 in HEMA/MMA copolymer as a function of dye concentrations.

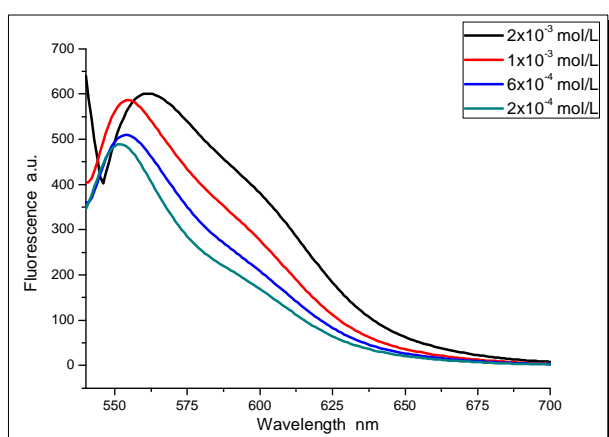


Figure (2c): Emission spectra of R6G in HEMA/MMA copolymer as a function of dye concentrations.

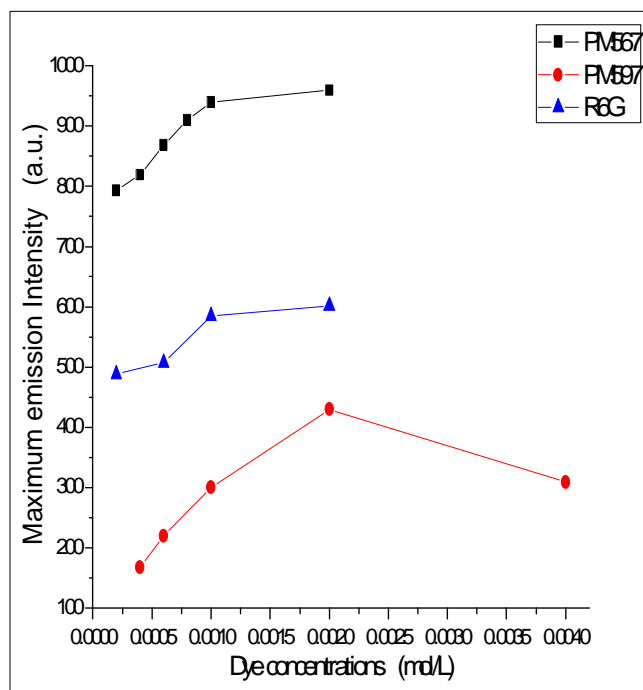
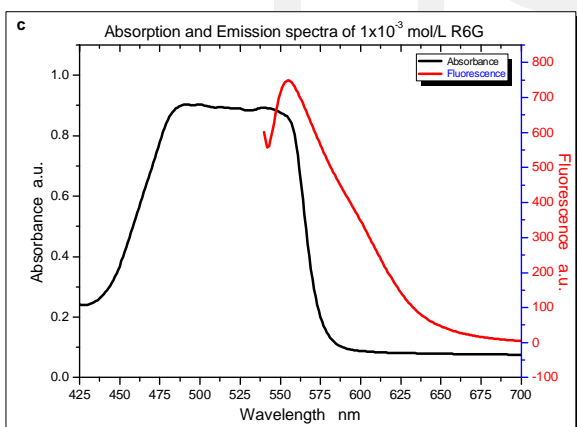
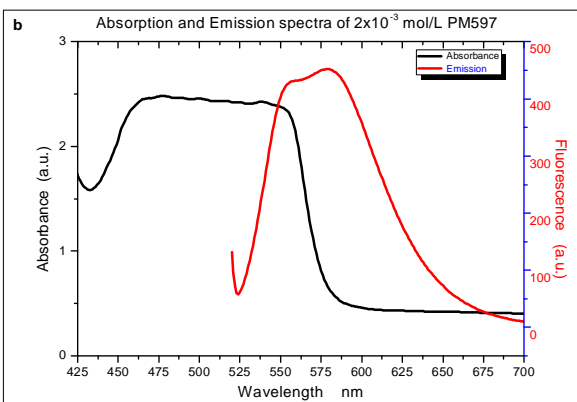
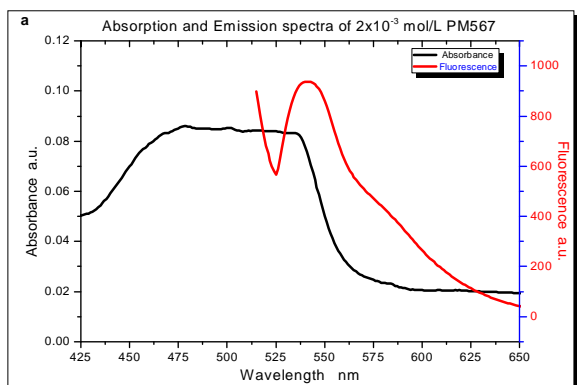


Figure (3): Emission Intensities as a function of dyes concentrations in HEMA/MMA copolymer host.

The emission spectra of the optimum concentration of the three laser dyes are shown in (figures 4a-c) which represent a spontaneous radiative process (fluorescence) from excited state S1 to ground state S0. The emission spectra shifted to longer wavelength than absorption spectra owing to Franck-Condon effect. Since the electronic charge distribution in excited state is usually different from that in the ground state, reorientations occur. Therefore, the resulting energy loss is manifested in a shift of the emission to longer wavelength.

The effect of different dye concentrations on fluorescence spectra for all dyes under study (PM-567, PM-597 and R6G) indicated a persistent red-shifting of emission peak for all dyes. In general, the red shift observed from increasing the dye concentration arises from increasing the number of unexcited dye molecules. These unexcited dye molecules will absorb the radiation emitted by the excited dye molecules (re-absorption effect). [33]



Figures (4a-c): Absorption and Emission spectra of the optimum concentration of the three laser dyes.

According to the absorption and emission spectra of the three laser dyes (PM567, PM597 and R6G) doped in HEMA/MMA polymer host, some important photo physical parameters were assessed and tabulated in table (1). These properties such as the oscillator strength (f), the transition dipole moment μ_{12} , the attenuation length $\Lambda(\lambda)$, the energy of fluorescence (E_f), radiative decay rate (K_r) and intersystem crossing rate (K_{isc}). These optical parameters give good information about laser performance of the three laser dyes in this polymeric host. It noticed from table (1) that, the extinction coefficient " ϵ " of the three laser dyes is ~ 104 which mean that,

$\pi-\pi^*$ transition takes place by singlet transition and not triplet transition because in the last case " ϵ " $\sim 10^{-1}-10^{-2}$. Also, R6G has the highest quantum yield compared with other two dyes.

The value of oscillator strength (f), which shows the effective number of electrons whose transition from ground to excited state, gives the absorption area in the electron spectrum, showed from table (1) that, Rhodamine 6G has the highest effective value. Also, Rhodamine 6G has the highest transition dipole moment value which is a vector describe the electric charge within a molecule of a fluorophore shifts during excitation through absorption of a photon (or during emission of a photon of fluorescence). we noticed also from table (1) that, Rhodamine 6G in HEMA/MMA host has the highest radiative decay rate value and the lowest intersystem crossing rate value compared with the other two laser dyes in the same polymeric host which indicated that, Rhodamine 6G will expect to have higher laser performance than the other laser dyes.

Different samples with 2cm length and 1cm diameter for each of (PM567, PM597 and Rh6G) with concentrations ranging from (2×10^{-3} to 2×10^{-4} mol/L), (4×10^{-3} to 4×10^{-4} mol/L) and (2×10^{-3} to 2×10^{-4} mol/L) respectively doped in polymeric host material were pumped transversely in resonating cavity to examine their laser performance. The laser performance efficiency is defined as the total output laser energy divided by the total input pumping energy:

$$\eta = \text{Edge-laser} / E_{\text{pump}} \quad (11)$$

Therefore the efficiency was calculated by plotting the input energy of Nd-YAG laser against the output energy of the emitted radiation of the dye laser. The slope efficiency of each dye of different concentrations was measured on a single pulse basis and averaging five pulses provided each data point. The average energy efficiencies extracted from the above input-output energy measurements are presented in figures (5a-c) and table (2). In general, laser performance is affected by methodology and morphology of polymer host and setup alignment of resonating cavity. According to our results the concentration (2×10^{-3} mol/L) is the approximately optimum concentration for PM567, PM597 and R6G laser dyes doped in (HEMA: MMA) polymer which gave a highest laser performance values of 51.1 %, 52.0% and 54.3% respectively.

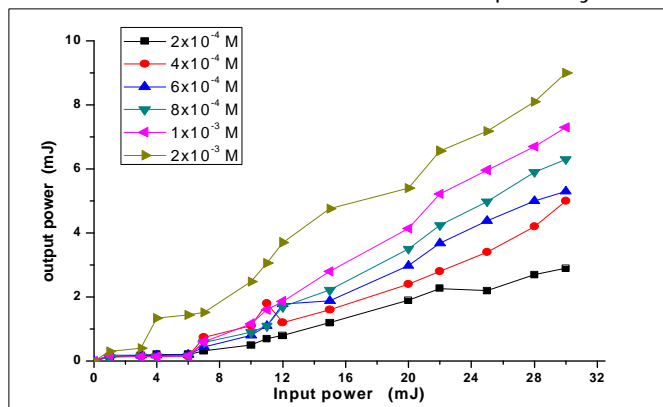


Figure (5a): output energy of PM567 dye doped in (HEMA: MMA) as a function of pumping energy for different concentrations.

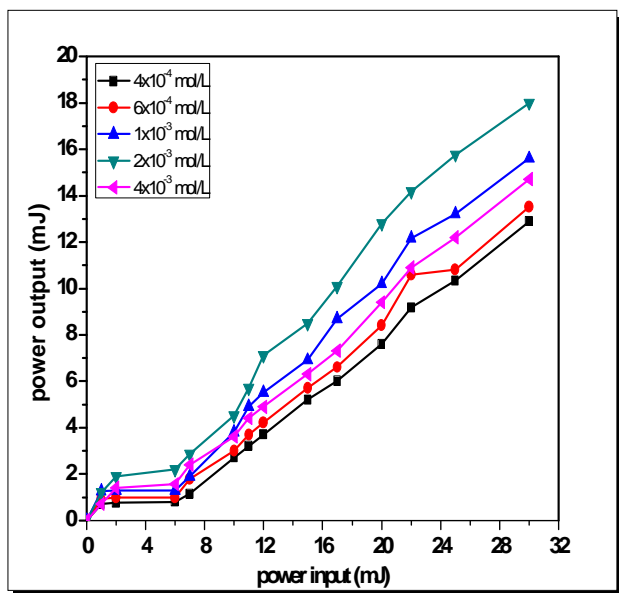


Figure 5(b): output energy of PM597 dye doped in (HEM:MMA) as a function of pumping energy for different concentrations.

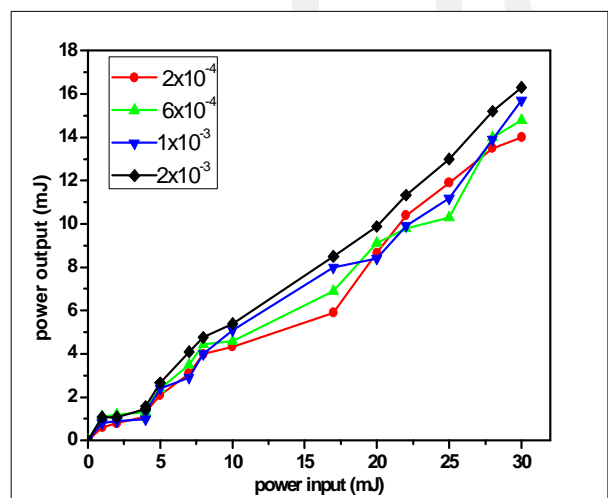


Figure 5(c): Output energy of R6G dye doped in(HEMA:MMA) as a function of pumping energy for different concentrations.

Since one of the most important and fundamental properties of solid state dye lasers is photochemical degradation occurs through the interaction of the dye molecules in the excited state with other species such as impurities, other dye molecules and singlet state oxygen, Through the process of interaction between laser dyes and a host medium. These photochemical pathways can be suppressed by caging or trapping the dyes within a solid host, and the photostability of the dye molecules in solid host is remarkably improved.

The improved photostability of the dye within solid hosts has been attributed to caging and immobilizing the dye molecules, thereby preventing excited-state interaction with other species. So in a solid host, the photo-degradation of the laser dye depends on the nature of dye molecule, the nature of interaction between dye molecules and solid host, the composition and structure of the host, and the impurities presented in the host. Other factors that may affect the thermal and optical stability of dye molecules are peak and average pump fluence, excitation wavelength, pulse rate, sample thickness, geometry, and experimental setup and conditions [34]. The nature of these photochemical degradation mechanisms means that the complete stabilization of organic dye molecules is not possible.

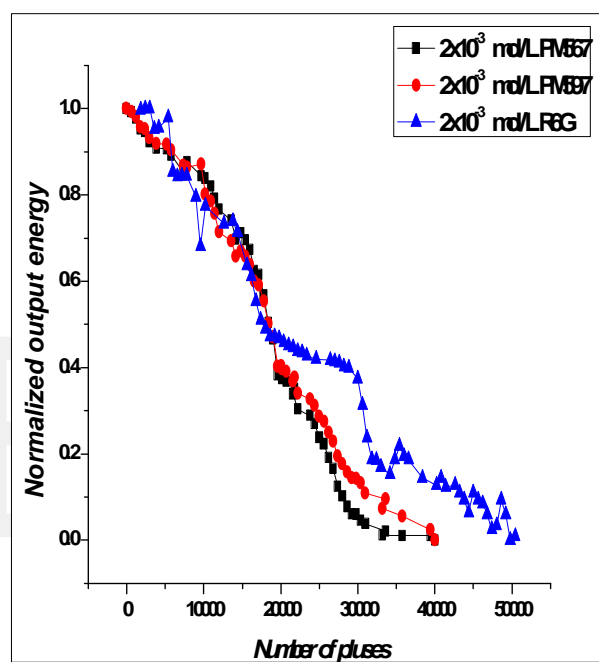


Figure 6): Normalized laser output as a function of the number of pumping pulses at repetition rates of 10Hz. Pumping energy 10mJ.

The photostability of the polymeric state laser dyes were carried out for optimum concentration (2×10^{-3} mol/L) of laser performance efficiency. It was observed that, although the laser output energy decreased to nearly 50% of its initial after approximately the same number of pulses (~ 18000) for all dyes. After about 20000 pulses, R6G showed higher stability than the other PM dyes as shown in figure (6).

TABLE (1) PHOTO-PHYSICAL PARAMETERS OF (2X10⁻³MOL/L) PM567, PM597 AND R6G DOPED IN HEMA/MMA POLYMER HOST; (E) MOLECULAR EXTINCTION COEFFICIENT; ΣA AND ΣE: ABSORPTION AND EMISSION CROSS SECTION; (Λ) THE ATTENUATION LENGTH,(TCALL)CALCULATEDFLUORESCENCE LIFE TIME, M12(D) THE TRANSITION DIPOLE MOMENT, (Ef)ENERGY YIELD OF FLUORESCENCE, (KR)THE RADIATIVE DECAY RATE ,(KISC) THE INTERSYSTEM CROSSING RATE, (F) OSCILLATOR STRENGTH, ΦFLUORESCENCE QUANTUM YIELD.

Dye	ϵ (L.M ⁻¹ .Cm ⁻¹) (10 ⁴)	σ_a (10 ⁻¹⁷) Ccm ²	σ_e (10 ⁻¹⁷) Ccm ²	Λ (cm)	τ_{call} (ns)	μ_{12} (D)	E_f	K_r (10 ⁹) s ⁻¹	K_{isc} (10 ⁹) s ⁻¹	f	ϕ_f
PM567	6.6	27.2	8.3	0.08	7.2	4.2	0.32	0.73	2.5	0.45	0.62
PM597	6.1	22.5	7.4	0.11	7.7	4.8	0.35	1.0	2.1	0.62	0.65
R6G	6.9	30.1	9.7	0.32	6.6	5.3	0.38	2.3	0.8	0.73	0.73

TABLE (2) LASER PERFORMANCE OF (PM567, PM597 AND R6G) DOPED IN HEMA/MMA POLYMERIC STATE AS A FUNCTION OF DYE CONCENTRATION:

Host materials	Laser dyes	Dye concentration (mol/L)	Laser performance (%)
HEMA:MMA copolymer	PM567	2x10 ⁻⁴	35.6
		4x10 ⁻⁴	41.6
		6x10 ⁻⁴	43.0
		8x10 ⁻⁴	44.0
		1x10 ⁻³	49.9
		2x10 ⁻³	51.1
	PM597	4x10 ⁻⁴	24.3
		6x10 ⁻⁴	33.3
		1x10 ⁻³	49.0
		2x10 ⁻³	52.0
		4x10 ⁻³	40.6
	R6G	2x10 ⁻⁴	46.6
		6x10 ⁻⁴	49.3
		1x10 ⁻³	52.3
		2x10 ⁻³	54.3

4 CONCLUSION

According to optical spectroscopy spectra, photo-physical parameters, laser performance and photostability of PM567, PM597 and R6G in HEMA/MMA (1:1 v/v) copolymer host, R6G had higher laser performance and photostability than PM's dyes which is promising for wide solid state dye laser applications in 550-650 nm range specially in underwater communications and remote sensing of atmosphere contaminants devices.

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