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Study of Linear and Non-Linear Optical Properties for the Thin Films of Laser Dye-Fe_3O_4 Nanoparticles Doped Pmma Thin Films

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Abstract: Four different mixtures of 10^{-3} mol/L Rhodamine B laser dye, 0.1 g PMMA and one of Fe_3O_4 nanoparticles weights (0.02, 0.04, 0.06 and 0.08 g) and other four mixtures for the Styryl 9M laser dye with the same contents had been prepared using chloroform solvent. Eight different thin films had been made from these mixtures using drop casting method. The absorption spectrums of these thin films had been taken. The absorption spectral properties as (peak wavelength, peak absorbance and frequency difference at half absorbance maximum) had been measured depending on the absorption spectrums for thin films. The non-linear optical properties as (Transmittance difference ΔT_{p-v}), non-linear phase shift $\Delta\Phi_0$, non-linear refractive index n_2, Minimum normalized transmittance T(Z) and non-linear absorption coefficient β) were calculated according to normalized transmittance data obtained from Z-scan setup with closed and open aperture. The effect of the laser dye type changing in thin films of the same contents had been studied. The main conclusion is that the Rhodamine B laser dye give highest linear and non-linear optical properties than Styryl 9M laser dye. This can be attributed to that the first has more complete Benzene rings than for second and causes more absorption. So, this helps in choosing the medium and Fe_3O_4 nanoparticles with appropriate excitation wavelengths.

Key words: Rhodamine B and Styryl 9M laser dye, Fe_3O_4, nanoparticles, linear, non-linear, properties, excitation

INTRODUCTION

Iron oxide nanoparticles is a crystal magnetite configuration and had particle size <128 nm (El-Ghandoor *et al*., 2012). They have the super paramagnetic properties, since, they magnetic behave only in the existence of applied magnetic field prevents self-accumulation. They have a high magnetic moment depending on the number of nanoparticles in super paramagnetic cluster which is called as magnetic nano bead (El-Ghandoor *et al*., 2012; Tadic *et al*., 2014; Nicklaus, 2015). The physical and optical properties of Fe_3O_4 nanoparticles had been illustrated in Table 1.

Fe_3O_4 nanoparticles had been used in the magnetic recording applications as: the audio recording and the manufacturing of videotape or disks.In medicine, they may be utilized as a drug transport structures, radiofrequency and microwave devices, the magnetic system of resonance imaging (MRI) in cancer therapy and finally, in the construction of sensors (Lu *et al*., 2007). Rhodamine B laser dye has red to violet colour. It had the molecular formula of $C_{28}H_{31}ClN_2O_3$ and 479.02 gm/mol molecular weight. The laser emission by Rhodamine B is tunable about 610 nm while it has quantum yield of about

Table 1: The physical and optical properties of $F_{e3}O_4$ nanoparticles (El-Ghandoor *et al*., 2012; Tadic *et al*., 2014; Nicklaus, 2015)

Properties	Values
Chemical formula	$F_{e3}O_4$
Molar mass	231.533 (g/moL)
Appearance	Solid black powder
Density	5 (g/cm^3)
Melting point	1.597°C
Boiling point	2.623°C
Refractive index (n_D)	2.42

(0.49-1.0) dependent on the temperature. When it is excited by a 514 nm of argon laser and 532 nm of Q-switched Nd:YAG laser, the quantum yield of absolute fluorescence had been appeared 5 (Yang *et al*., 2006; Du *et al*., 1998; Kubin and Fletcher, 1982; Casey and Quitevis, 1988; Kellogg and Bennett, 1964). The chemical structure of a Rhodamine B dye had been drawn in Fig. 1 (Fikry *et al*., 2009).

Styryl 9M is a 2-(6-(4-Dimethylaminophenyl)-2,4-neopentylene-1,3,5-hexatrienyl)-3-methyl-benzothiazolium Perchlorat and the molecular structure $C_{27}H_{31}N_2O_4SCl$. Styryl 9M had the green, crystalline solid appearance and the absorption maximum (in ethanol) is 585 nm and the molar absorptivity is 5.05×10^4 L moL/cm (Lin and Marshall, 1984; Smith *et al*., 1984; Mulders and

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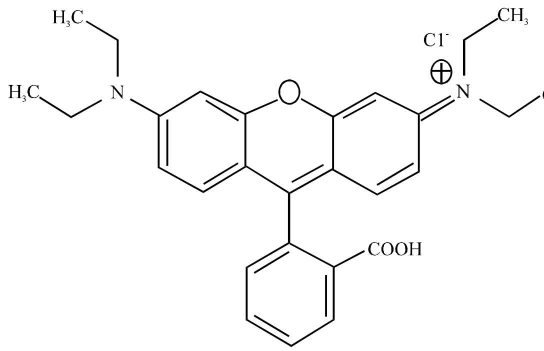

Fig. 1: The molecular structure of Rhodamine B laser dye (Fikry *et al*., 2009)

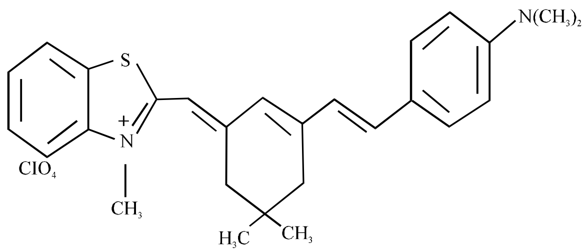

Fig. 2: The molecular structure of the Styryl 9M laser dye (Piccotti, 2016; Brackmann, 2000)

Steenhuysen, 1985; Makarov *et al*., 2008, 2011 Berezin *et al*., 2011; Piccotti, 2016). The diagram of the molecular structure for the Styryl 9M laser dye had been illustrated in Fig. 2.

Poly (methyl2-methylpropenoate) is the Polymer of Methacrylic Acids Esters (PMMA) which has $C_5H_8O_2$ form. It is rear, colorless polymer. It had been used in many fields as (dust covers, sunglasses, lenses, rear lights in vehicles, lamp covers and switch parts in electrical circuits, pills and capsules in medicine, …, etc. (Qian *et al*., 2010). The molecular structure of PMMA polymer had been shown in Fig. 3.

PMMA has the long, thinner as well as smoother chains of essentially carbon atom. It is hardest polymer which has highly mechanical resistant. Moreover, the highly strength of it doesn't sever more changes when it exposure to UV radiation. It had less absorption peak capacity in water, high dielectric constant and thermal stability of PMMA, for that it can be used in electrical fields.

Because of it has a good transmittance for visible light, highly appropriate with uman tissue (Antonio, 2007). An important properties of PMMA had been shown in Table 2.

The study of non-linear optical properties for a non-linear medium is very important due to it provides a knowledge of molecular response to incident laser beam energy and the possible changes related with it, so, this project aims to study the non-linear optical properties for a four different thin films of 10^{-3} mol/L Rhodamine B laser dye, 0.1 g PMMA and one of Ag nanoparticles weights (0.02, 0.04, 0.06 and 0.08 g).

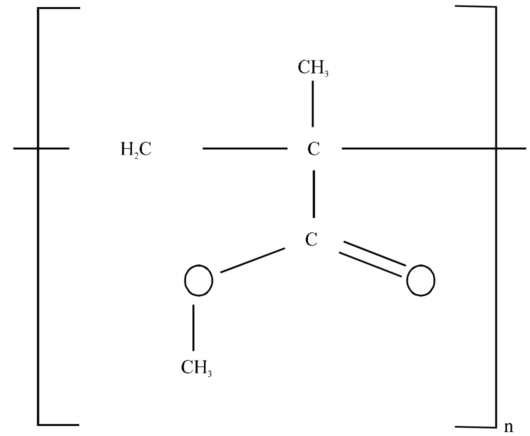

Fig. 3: The molecular structure of PMMA polymer (Qian *et al*., 2010)

Table 2: An important properties for PMMA polymer (Antonio, 2007)

Properties	Values
Density	(1.15-1.19) (g/cm^3)
Peak absorption in water	(0.3-2)%
Transmission, visible	(80-93)%
Refractive index	(1.49-1.498)
Electrical resistivity	(10^{14}-10^{15}) Ω. cm
Dielectric constant	(2.8-4)
Specific heat capacity	(1.46-1.47) (J/g.°C)
Thermal conductivity	(0.19-0.24) (W/m.K)
Melting point	130°C

MATERIALS AND METHODS

Absorption spectral properties: Absorption process can be happened when an incident photon crash the dye molecule in its lower energy state, the dye will be excited due to that the photon is absorbed and its energy is utilized in the dye excitation. This process can be occurred only if the incident photon energy equals to the energy difference between the two states the absorption take place at them.

Absorbance A can be defined as the logarithmic relative decrease of intensity. So, the wavelength at the maximum absorbance may be called as peak wavelength (nm) while the maximum absorbance can be named as peak absorbance (arb.unit) and the width of absorption (curve at the half value of maximum absorbance is termed as $(\Delta v)1/2$ (sec^{-1}) which is calculated from the absorption spectrum (Elmer, 2000).

Linear optical properties: The linear optical properties related with linear optical response had been formulated as:

The absorbance (A) describes the amount of absorbed photons by molecules can be written as:

$$A = 1\text{-}\log\left(\frac{1}{T}\right) \quad (1)$$

where, T is the transmittance of medium which is related with refractive index n as:

$$T = \frac{2n}{n^2+1} \quad (2)$$

The absorption coefficient α of an optical medium can be related to the Aabsorbance according to Eq. 3 (Gun and Chong, 2009; Ara *et al*., 2010):

$$\alpha = \frac{1}{2.302A} \quad (3)$$

The effective length L_{eff} of an optical medium can be calculated by Eq. 4 as Gun and Choang (2009) and Ara *et al*. (2010):

$$L_{eff} = \frac{\left(1\text{-}\exp^{-\alpha L}\right)}{\alpha} \quad (4)$$

Where:

L = The length of a sample

α = The absorption coefficient

Non-linear optical properties: Some of materials severe from the non-linear effects as Kerr non-linearity which is a change in the refractive index of a material in response to an applied electric field.

The Z-scan technique is used to measure the non-linear refractive index and the non-linear absorption coefficient using the closed and open aperture, respectively. The closed aperture form helps in determining the small distortions in the incident beam on the non-linear medium which behaves as a small non-linear lens and in measuring the non-linear refractive index as shown in Fig. 4a where illustrates the construction of a closed aperture and the diagram of the possible measured data of the normalized transmittance using it.

The open aperture Z-scan is used for measuring the non-linear absorption coefficient as the whole laser beam may be allowed to incident on the detector and the small distortions can be neglect. Figure 4b illustrates the construction of a Z-scan with open aperture and the diagram of the possible measured data of the normalized transmittance using it.

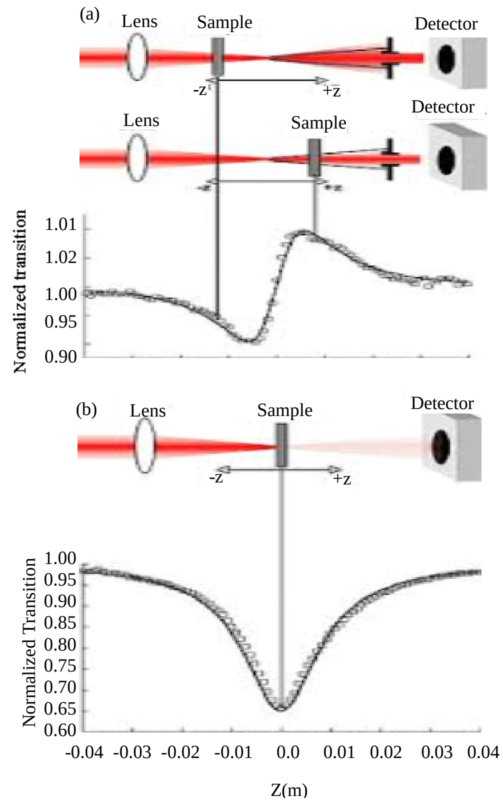

Fig. 4: The construction of a Z-scan with two different designs and the diagram of the possible measured data of the normalized transmittance using them: a) Closed aperture and b) Open aperture (Ali and Mahdi, 2012)

There are some of the changes must be taken into account in the study of the non-linear coefficients as: the non-linear response of the medium due to the laser beam in limited region (laser spot area on the medium) may be affected by the laser beam intensity in the surrounding regions. This effect can be named as (non local response).

The other effect appears in the liquid samples of the colloid non-linear medium in the dielectric solution ,the photonic incident field in the non-local points of a medium induces many changes as reorientation of the molecular dipoles due to the changes in the electric fields in different parts of a non-linear medium (Ali and Mahdi, 2012).

The non-linear refractive index can be calculated using Eq. 5 as follow (Gun and Chong, 2009):

$$n_2 = \frac{\Delta\Phi_o}{I_o L_{eff} K} \quad (5)$$

Where:

- I_o = The incident laser intensity
- K = The wavenumber of the incident laser beam, equals to $K = 2\pi/\lambda$, since λ is the wavelength of the incident laser beam
- $\Delta\Phi_o$ = Non-linear phase shift and equals to Gun and Chong (2009) and Ao *et al*. (2015)

$$\Delta T_{p\text{-}v} = 0.406\,|\,\Delta\Phi_o\,| \tag{6}$$

where, $\Delta T_{p\text{-}v}$ is the normalized transmittance difference between the top and the valley transmittance values which can be calculated using closed aperture Z-scan setup.

The non-linear absorption coefficient β may be calculated using Eq. 7 as follow (Gun and Chong, 2009; Ao *et al*., 2015):

$$\beta = \frac{2\sqrt{2}T(Z)}{I_o L_{eff}} \tag{7}$$

Since, T(Z) means the minimum normalized transmittance obtained using open aperture Z-scan setup.

Experimental setup: A 10^{-3} mol/L molar concentration of Rhodamine B laser dye (R 6626-25G, product of USA, Sigma-Aldrich) and of Styryl 9M laser dye (MW = 515.06 g/moL, product of USA, Sigma-Aldrich) had been prepared by weighting (0.001 g) using digital balance (model-HR-200, made in Japan) from laser dye. This weight had been dissolved in (10 mL) of chloroform solvent (D-30926 seelze, Sigma-Aldrich Laborchemikalien Gmbh, Germany). A 0.1 g of PMMA polymer (Sc-255435, Santa Cruz Biotechnology, Dallas) had been added to the dye solution. A four different weights of Fe_3O_4 nanoparticles the Fe_3O_4 nanoparticles used in this thesis is of (particle size of 20-30 nm, SkySpring Nanomaterials, Inc, SSNANO, made in USA) as (0.02, 0.04, 0.06 and 0.08 g) had been weighted using the same balance and added to the dye mixtures. So, a four different mixtures were obtained, contain (10^{-3} mol/L Rhodamine B or 10^{-3} mol/L Styryl 9M laser dye, 0.1 g PMMA and Fe_3O_4 nanoparticles). To perform a homogeneous solution, A magnetic stirrer (Model-HP-3000, Lab. companion) had been used.

These mixtures had been prepared as a thin films using drop casting method. Figure 5 shows the pictures for the prepared thin films.

The absorption spectrum for thin films samples had been recorded using UV-Vis spectrophotometer (Scinco, Mega-2100, made in Korea) and Fig. 6 shows the absorption spectrum for these samples.

The used Z-scan set up as described in paragraph (2-c), contained from SHG-Nd:YAG laser at 532 nm (green light) as a laser source had been focused to a sample using a lens of (15 cm) focal length where the radius of laser spot was (0.05 cm) and the incident laser intensity on the sample of 778 W/m^2. The normalized transmittance can be measured using a laser power meter (LP1-Mobiken).

The normalized transmittance data for the prepared thin film samples of 4 cm length and 1 mm thickness, contained from (10^{-3} Rhodamine B, 0.1 g PMMA and different weights of Fe_3O_4 nanoparticles) had been measured using Z-scan setup of closed aperture and pen

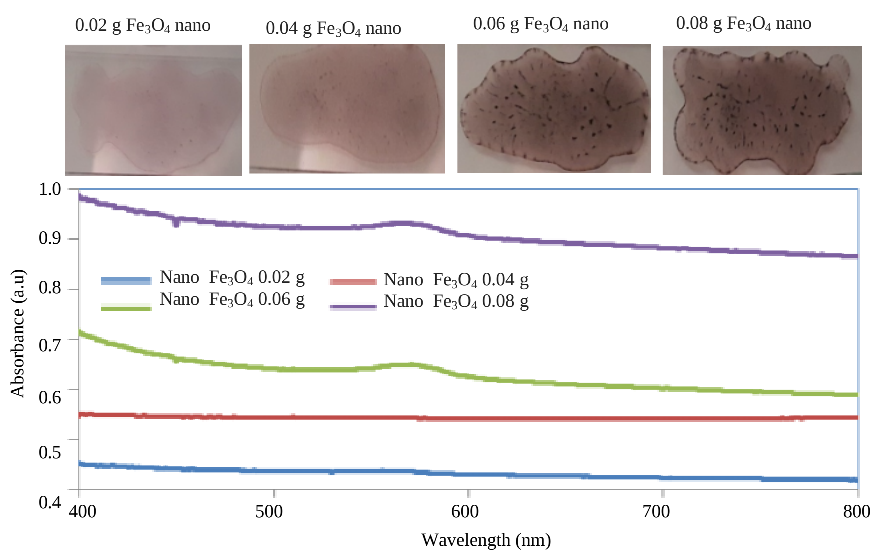

Fig. 5: The pictures for the prepared thin films of 10^{-3} mol/L Rhodamine B, 0.1 g PMMA and Fe_3O_4 nanoparticles) and the absorbance spectrum for them

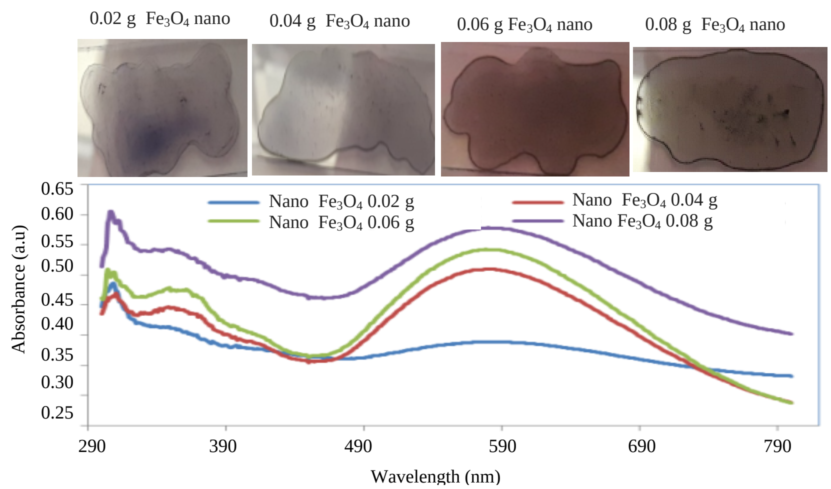

Fig. 6: The pictures for the prepared thin films of 10^{-3} mol/L Styryl 9M, 0.1 g PMMA and Fe_3O_4 nanoparticles) and the absorbance spectrum for them

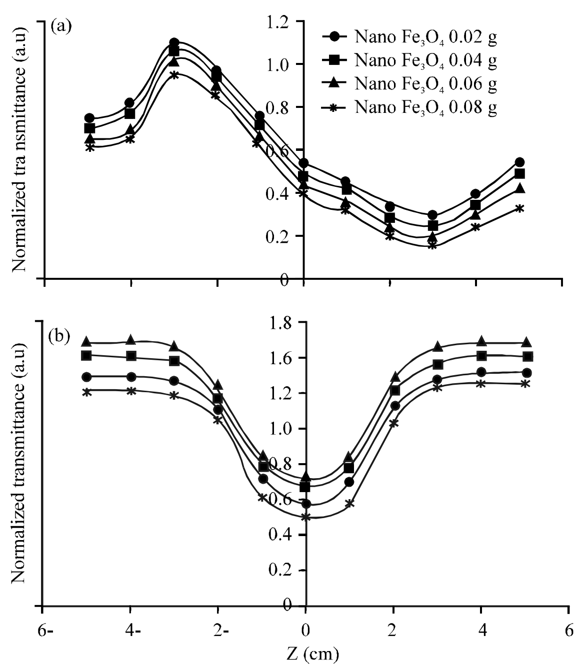

Fig. 7: The normalized transmittance for thin film samples of 10^{-3} Rhodamine B, 0.1 g PMMA and different weights of Fe_3O_4 nanoparticles) using Z-Scan setup of: a) Closed aperture and b) Open aperture

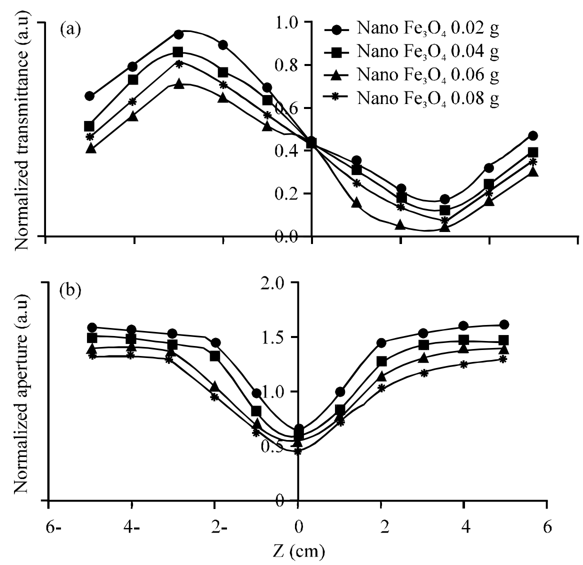

Fig. 8: The normalized transmittance for thin film samples of 10^{-3} Styryl 9M, 0.1 g PMMA and different weights of Fe_3O_4 nanoparticles using Z-scan setup of: a) Closed aperture and b) Open aperture

aperture with aid of laser power meter (LP1-mobiken) and had been plotted in Fig 7a closed aperture and b open aperture. The normalized transmittance data for the prepared thin film samples of 10^{-3} Styryl 9M, 0.1 g PMMA and different weights of Fe_3O_4 nanoparticles are measured using Z-Scan setup had been plotted in Fig. 8a closed aperture and b open aperture.

RESULTS AND DISCUSSION

According to the absorption spectrums shown in Fig. 4, the absorption spectrum characteristics for thin films of (10^{-3} Rhodamine B, 0.1 g PMMA and different types of nanoparticles), like (Peak wavelength nm, Peak absorbance (arb.unit) and $(\Delta v)1/2$ (sec^{-1}) had been listed in Table 3.

According to the absorption spectrums taken to the prepared thin films and recorded in Fig. 5 and 6 and in Table 3, the linear optical properties of that films had been calculated and recorded in Table 4.

Table 3: Absorption spectrum characteristics for different Rhodamine B and Styryl 9M laser dyes thin films doped with Fe_3O_4 nanoparticles

Laser dye	Weight of $F_{e3}O_4$ nanoparticles (g)	Peak wavelength (nm)	Peak absorbance arb.unit	$(\Delta v)_{1/2}$ sec^{-1}
Rhodamine B	0.02	555	0.1090	9×10^{15}
	0.04	567	0.1350	10.7×10^{15}
	0.06	570	0.1620	12×10^{15}
	0.08	574	0.2330	120×10^{15}
Styryl 9M	0.02	590	0.0975	$1.7\times10^{+15}$
	0.04	592	0.1270	$1.85\times10^{+15}$
	0.06	593	0.1350	$2\times10+15^{+15}$
	0.08	594	0.1450	$2.3\times10^{+15}$

Table 4: The linear optical parameters for thin film samples of (10-3 Rhodamine B and Styryl 9M laser dyes, 0.1 g PMMA and Fe_3O_4 nanoparticles at four different weights

Laser dye/Weight of $F_{e3}O_4$ nanoparticles	Absorbance A	Transmittance T	Absorption coefficient α	Refractive index n	Effective length L_{eff} (m)
Rhodamine B					
0.02	0.1090	0.778	3.985	2.092	0.0395
0.04	0.1350	0.732	3.217	2.296	0.0395
0.06	0.1620	0.687	2.673	2.513	0.0394
0.08	0.2330	0.584	1.864	3.102	0.0394
Styryl 9M					
0.02	0.0975	0.798	4.455	2.008	0.0396
0.04	0.1270	0.746	3.420	2.233	0.0394
0.06	0.1350	0.732	3.217	2.296	0.0395
0.08	0.1450	0.716	2.995	2.371	0.0396

Table 5: The non-linear optical parameters for thin film samples of (10-3 Rhodamine B and Styryl 9M laser dyes, 0.1 g PMMA and Fe_3O_4 nanoparticles) using Z-scan setup of both closed aperture and open aperture

Nanoparticle/Weights of particles (g)	Transmittance difference ΔT_{P-v}	Non-linear phase shift $\Delta\phi_o$	Non-linear refractive index $n_2(x10^{-9})$	Minimum normalized transmittance T(Z)	Non-linear absorption coefficient β
Rhodamine B					
0.02	0.94	2.3152	6.3849	0.81	0.0745
0.04	0.96	2.3645	6.5209	0.76	0.0699
0.06	0.96	2.3645	6.5389	0.65	0.0599
0.08	0.93	2.2906	6.3346	0.56	0.0516
Styryl 9M					
0.02	0.93	2.2900	6.3010	0.64	0.0590
0.04	0.89	2.1920	6.0620	0.60	0.0550
0.06	0.86	2.1180	5.8410	0.54	0.0490
0.08	0.81	1.9950	5.4880	0.46	0.0420

The non-linear optical parameters such as (Transmittance difference (ΔT_{p-v}), non-linear phase shift ($\Delta\Phi_o$), non-linear refractive index (n_2), minimum normalized transmittance T(Z) and non-linear absorption coefficient (β) for thin film samples of 10^{-3} Rhodamine B or 10^{-3} mol/L Styryl 9M laser dye, 0.1 g PMMA and different weights of Fe_3O_4 nanoparticles) had been calculated according to the measured data of normalized transmittance using Z-scan setup of both closed aperture and open aperture and the equations of non-linear optical properties mentioned in paragraph (2-Theory). These non-linear optical coefficients had been listed in Table 5.

It can be shown that, the increasing of Rhodamine B dye concentration in thin films makes the film more transmittance and less absorbance and the prepared films have more absorption coefficient and less refractive index. This behavior can be attributed to the double effect for the micro and nanoparticles with the PMMA polymer existence in films, since, the PMMA polymer is the transparent component in the film and Rhodamine B molecules are the absorbant component and the micro and nanoparticles are the scattering centers in films.

The results in Table 5 illustrates that the increasing in the nanoparticles weights increases both of absorbance and refractive index while it decreases both of transmittance and absorption coefficient. This behavior is attributed to that the highest number of scattering particles consolidates the absorption process with scattering process and makes this medium has more optical density which causes highest refraction.

It can be shown from Table 5 that the Rhodamine B laser dye has more absorbance than Styryl 9M laser dye because of that the first has more molecular weight than Styryl 9M (Yang *et al*., 2006). It is clear from the results in above table that the Rhodamine B laser dye has more absorption characteristics than Styryl 9M laser dye.This is true in different nanoparticles are used and in case of liquid or solid (thin film) phase. The normalized transmittance for thin film samples of 10^{-3} Rhodamine

B, 0.1 g PMMA and different weights of Fe_3O_4 nanoparticles) with a good agreement with the published data (Iranizad *et al*., 2014; Al-Sultani, 2017).

Figure 7 and 8 show that the increasing of Fe_3O_4 nanoparticles is the main reason of reduction the top-valley normalized transmittance in closed aperture and decreasing the minimum transmittance using open aperture. This can be interpreted by the increasing of scattered photons through sample of high nanoparticles concentration. The normalized transmittance data for thin film samples of 10^{-3}Styryl 9M, 0.1 g PMMA and Fe_3O_4 nanoparticles with a good agreement with the published data (Zhang *et al*., 2011).

CONCLUSION

It can be concluded that the nanoparticles are the main reason to more non-linear response to laser fields. The two photon or reverse saturation absorption or the saturation absorption are main non-linear effects controlled the less values of non-linear absorption coefficient. The main reason to possess Rhodamine B laser dye more non-linear refractive index and absorption coefficient than for Styryl 9M laser dye attributed to that the first. While the negative non-linearity is clearly appeared in the samples contained Fe_3O_4 nanoparticles which is the main indicator to occur the self-defocusing effect as shown in less values of non-linear refractive index. The main conclusion is that the Rhodamine B laser dye give highest linear and non-linear optical properties than Styryl 9M laser dye. This can be attributed to that the first has more complete Benzene rings than for second and causes more absorption. So, this helps in choosing the medium and Fe_3O_4 nanoparticles with appropriate excitation wavelengths.

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