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Investigation of nitrogen plasma effect on the nonlinear optical properties of PMMA

Davoud Dorrnian*, Yasaman Golian and Alireza Hojabri

Abstract

Effects of low power direct current nitrogen plasma on linear and nonlinear optical properties of red BS dye-doped poly(methyl methacrylate) (PMMA) film are studied employing different optical techniques. From spectrometry data, it is shown that applying plasma in such a low range of power does not affect the linear absorption coefficient, linear refractive index, and optical bandgap of this dye-doped polymer; however, nonlinear parameters are changed. Experiments are performed using the second harmonic of a continuous Nd-Yag laser beam at a 532-nm wavelength and 20-mW power. The effect of nonlinear refractive index of red BS dye-doped PMMA film in broadening the laser beam is observed. The optical bleaching behavior is investigated by measurement of the intensity of laser beam through the sample. Its third-order nonlinearity is measured using close and open Z-scan data. Nonlinear absorption coefficient and refractive index, as well as real and imaginary parts of the third-order nonlinear optical susceptibility χ^3 , are changed significantly due to plasma treatment.

Keywords: Nonlinear optics: Refractive index: Nonlinear absorption: Z-scan: Beam broadening: Two-photon absorption: Optical bleaching: Plasma

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Background

Nonlinear optical properties have been the subject of numerous investigations by both theoreticians and experimentalists during recent years due to potential applications in optical signal processing and computing. The detailed investigation of linear and nonlinear optical coefficients enables us to fabricate materials, which are appropriately designed at the molecular level for a specific device [1]. Materials with third-order optical nonlinearities and optical limiting have been investigated extensively for their applications in optical phase conjugation, high-speed all optical switches, optical bistability, optical limiting devices, and so on [2].

Poly(methyl methacrylate) (PMMA) is a hard, rigid, and transparent polymer with a glass transition temperature of 125°C. Its average molecular weight is 6×10^{-4} . It is tougher than polystyrene. PMMA is a polar material and has a large dielectric constant. PMMA matrix is most

preferred for designing components because of its better resistance to hydrolysis and its good outdoor weather resistance. It is a thermoplastic and can be molten and molded into anything we want [1,3,4].

It is one of the most interesting polymeric media for integrated optics and for optical fiber fabrication. PMMA is a representative polymer with excellent optical transparency. In addition to its optical transparency, it is found that it can produce a large refractive index difference with acrylamide-based photopolymer. Indeed, passive polymeric waveguides have been successfully obtained using PMMA or PMMA-based media, by means of different techniques, and PMMA optical fibers for the visible spectral range are commercially available. Despite its advantages, PMMA by itself cannot be used as an NLO medium because of its low nonlinear refractive index n_2 .

Plasma treatment of polymer has been utilized broadly in surface modification to increase material adhesion and improve compatibility. It involves the interaction of the plasma-generated excited species with a solid interface and results in a physical and/or chemical modification of the

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first few molecular layers of the surface, while maintaining the properties of the bulk. It is known that by conventional ion beam and plasma treatments, the polymer samples can obtain resistance to chemical attacks; in addition, there is an increase in surface hardness, and a metal oxide layer is formed on the surface [5]. Plasma generated in a vacuum environment influences the surface of the polymer to make it suitable for a specific application. It has sufficiently high energy to break the covalent bonds of polymers exposed to the plasma. In the case of polymers, the surface should be compatible to the biological system, which can be effectively modified by the plasma. Plasma treatment can improve wettability, oxidize the surface, and enhance cell growth and adhesion. Absolute treatment affects the optical properties of the polymer. The various effects of plasma on a polymer surface may be categorized as follows: (a) surface modification, (b) grafting, and (c) film deposition [6].

In this work, the effect of plasma treatment on the optical properties of PMMA is studied. This effect on non-linear optical parameters and optical bleaching properties of this polymer is investigated.

This paper is organized as follows. After this short background, the experimental setup is presented in section 2. Section 3 is devoted to results and discussion and section 4 discusses the by conclusion.

Experimental details

In our experiments, the red BS dye-doped PMMA film is prepared by solving PMMA powder in dichloromethane solvent. PMMA powder is provided by Yazd Polymer Talaei (YPT) Co., Tehran, Iran. Twenty grams of PMMA powder with 1 g red BS dye is solved in 200 ml dichloromethane.

The viscous solvent is poured on a plane glass in a closed box. Solution dries in a closed atmosphere at room temperature for 24 h to obtain a 0.2-mm thickness of red BS dye-doped PMMA film.

After the preparation process, PMMA films are cleaned in alcohol ultrasonically, and one sample is treated in direct current (dc) nitrogen plasma of 70 W (50-mA current) for 8 min. Nitrogen plasma is generated in a cylindrical plasma reactor. Distance between electrodes was set at 8 cm, and the pressure in the chamber after introducing nitrogen was 8×10^{-2} Torr. PMMA films are placed near the cathode of the reactor during the treatment.

Structural and topological characters of modified films are investigated in another paper, and in this article, the optical characterization of treated film is presented. The chemical structures of PMMA and red BS dye are presented in Figure 1a,b, respectively. Change in absorbance spectra of the samples is measured by a Varian Cary-500 spectrophotometer (Varian Medical Systems, Palo Alto, CA, USA) at room temperature, and a 532-nm wavelength continuous Nd-Yag laser of 20-mW power is used to study

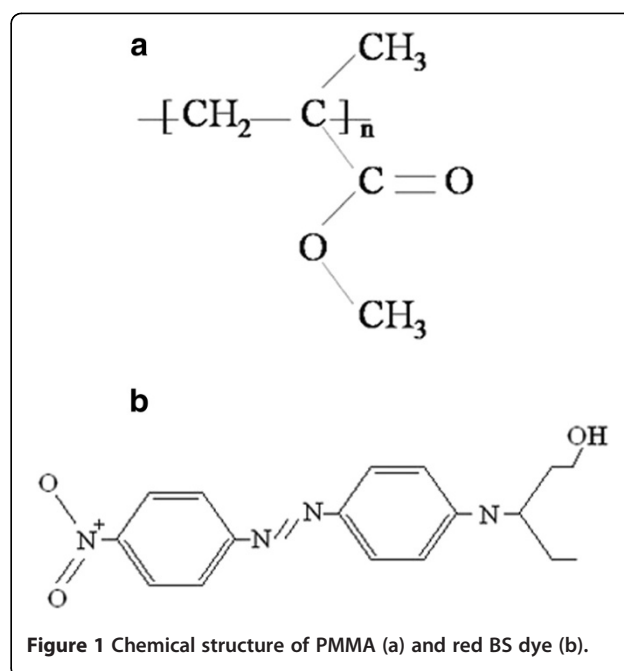


Figure 1 Chemical structure of PMMA (a) and red BS dye (b).

the nonlinear behavior of pristine and plasma-treated films. The experimental setup is shown in Figure 2. In this setup, the laser beam of 2 mm in diameter, after propagating through the 50 % beam splitter, is focused by a 6.5-cm focal length lens, which leads to a Rayleigh length of $z_0 = 1.7$ mm. The distance between the lens focal point and power meter 1 is changed to 6 cm during the experiment. The pinhole diameter is 0.8mm.

Results and discussion

Optical measurement

Reflectance $R(\lambda)$, transmittance $T(\lambda)$, and absorbance $A(\lambda)$ spectra of samples are presented in Figure 3 in the wavelength range of 200 to 900 nm. These basic parameters are not changed by the plasma treatment in this range of power, and in the visible spectrum, treated and pristine samples cannot be distinguished as well. The optical absorption coefficients of samples are evaluated from the transmittance and reflectance data using the following relation which can be used for polymeric samples [6,7]:

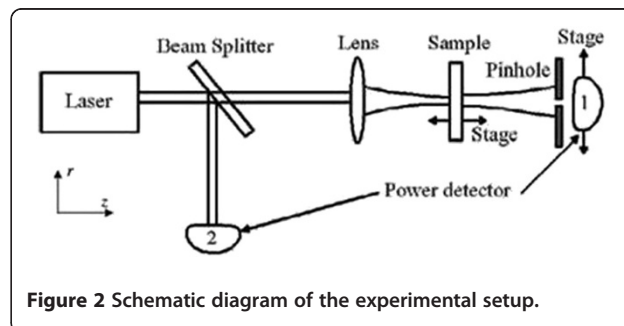


Figure 2 Schematic diagram of the experimental setup.

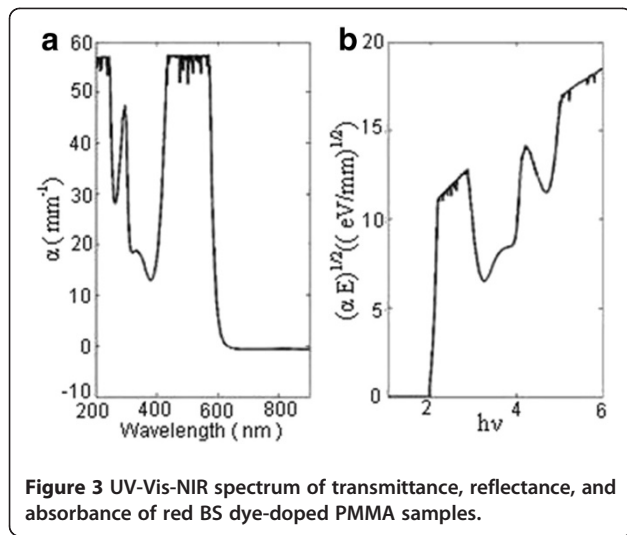


Figure 3 UV-Vis-NIR spectrum of transmittance, reflectance, and absorbance of red BS dye-doped PMMA samples.

$$a = \frac{1}{d} \ln \left[\frac{(1-R)^2}{2T} + \sqrt{\frac{(1-R)^4}{4T^2} + R^2} \right] \quad (1)$$

where $d=0.2$ mm and is the thickness of the samples. Figure 4a shows the absorption spectrum.

The optical bandgap is the value of optical energy gap between the valence band and the conduction band. The optical bandgap of the samples is determined from the absorption spectra near the absorption edges. The photon absorption in many amorphous materials is found to obey the Tauc relation [7], which is of the following form:

$$ahv = B(hv - E_g)^m \quad (2)$$

where hv is the photon energy; factor B depends on the transition probability and can be assumed to be constant

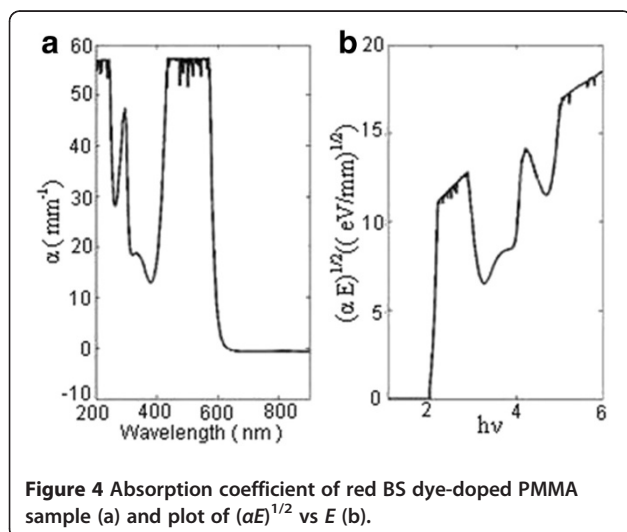


Figure 4 Absorption coefficient of red BS dye-doped PMMA sample (a) and plot of $(\alpha E)^{1/2}$ vs E (b).

within the optical frequency range [8], and index m is related to the distribution of the density of states. Index m has discrete values like $1/2$, $3/2$, 2 , or more depending on whether the transition is direct or indirect and allowed or forbidden, respectively.

In the direct and allowed cases, index $m = 1/2$, whereas for the direct but forbidden cases, it is $3/2$. However, for the indirect and allowed cases, $m = 2$, and for the forbidden cases, $m = 3$ or more. The value of m for the samples is estimated from the slope of the $\log(\alpha)$ vs $\log(hv)$ plots by taking a linear fit, which is found to be 2 [8,9]. To calculate E_g , the usual method is plotting $(ahv)^{1/2}$ versus hv as is done in Figure 4b. The values of the triple optical bandgap energy are calculated to be 1.92, 3.7, and 4.3 eV for both samples. The value of bandgap energy is not changed by the plasma treatment. It is useful to mention that for a pure PMMA (without dye doping), the magnitude of the bandgap is found to be 4.4 eV by our group in another experiment, so two other magnitudes for E_g are the effects of red BS dye. PMMA is naturally transparent in the visible range of electromagnetic radiation, and this high absorbance in this range of electromagnetic spectrum is due to the red BS dye which is a very common UV absorber in the polymers industry [10].

The real part of linear refractive index n for the samples can be calculated using the following relation:

$$n = \left(\frac{1+R}{1-R} \right) + \sqrt{\frac{4R}{(1-R)^2} - k^2}, \quad (3)$$

in which k is the extinction coefficient given by $k = \alpha\lambda/4\pi$. The data of refractive index as a function of incident wavelength are displayed in Figure 5. It can be observed that for red BS dye-doped PMMA film, there is an anomalous dispersion at $\lambda < 600$ nm as well as normal dispersion at $\lambda > 600$ nm. This is a transition behavior between

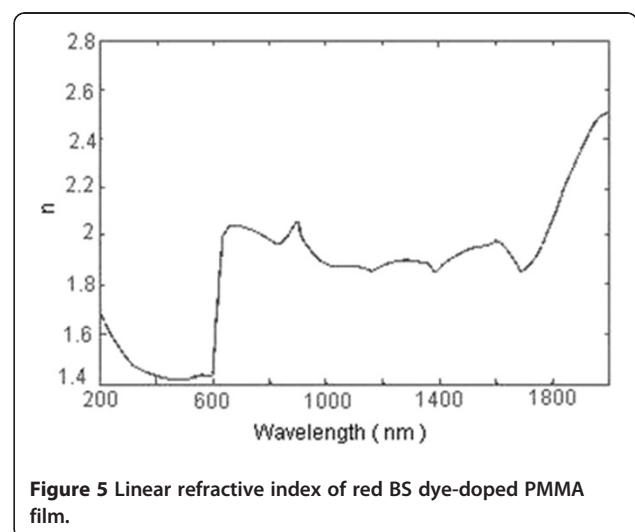


Figure 5 Linear refractive index of red BS dye-doped PMMA film.

the multi-oscillator and single oscillator models developed by Wemple and Didomenico [11]. The dispersion plays an important role in the research for optical materials because it is a significant factor in optical communication and in designing devices for spectral dispersion.

Different experiments were carried out to study the effect of plasma treatment on the nonlinear optical properties of red BS dye-doped PMMA film, including beam broadening measurement, optical bleaching measurement, and open and closed Z-scan experiments, which are discussed in the following sections.

Beam broadening

Self focusing and beam broadening of laser radiation in nonlinear media is a well-known effect that occurs due to the nonlinear index of refraction n_2 . In this experiment, the beam splitter and power meter 2 are not included in the setup. The lens collects the entire laser beam, and the intensity of the beam after transmission through the sample and passing through the pinhole of 0.8 mm in diameter is measured by a photo detector fed to the digital power meter. To measure the beam profile, the detector and pinhole move in the r direction which is the transversal direction perpendicular to the direction of laser beam propagation.

The spatial intensity distribution of the laser beam sent into the red BS dye-doped PMMA film is measured to have a Gaussian shape profile with a full width at half maximum (FWHM) of 2.4 mm when it is first measured without having any sample. The profiles of laser beam propagated through air, and pristine and plasma-treated samples are shown in Figure 6. As can be seen, the magnitudes of FWHM of profiles are 2.75 and 2 mm after propagating through pristine and plasma-treated samples, respectively.

This is the effect of minus n_2 of red BS dye-doped PMMA film. The nonlinear refractive index for the

pristine and plasma-treated samples are -3.4288×10^{-9} and $-2.3937 \times 10^{-9} \text{ cm}^2/\text{W}$, respectively, which confirms that the laser beam should be focused in the case of propagating through the treated sample.

Optical bleaching

By optical bleaching measurement, the critical power of laser beam at which the nonlinearity starts to affect the transmission can be measured. For this experiment, the sample was positioned near the focal plane of the lens, and the input power was changed using different optical filters. A 50 % beam splitter divides the initial power into two halves. The input power was measured by power meter 2, and power meter 1 was used to measure the output power of the focused transmitted beam through the red BS dye-doped PMMA film, as can be seen in Figure 7. The pin-hole was not used in this experiment.

In Figure 7, the output power of the laser beam is plotted as a function of incident power. There is the same result for all treated samples as well as for the pristine sample. The input power was in the range of 0 to 4.5 mW. At a low incident power up to 3.5 mW, the output power varies linearly, but for incident beam power above 3.5 mW, the slope of the line increases. Optical bleaching is the increase in transition of laser beam through the sample. At this point, nonlinearity of polymeric materials appears in two forms. Some of them show optical limiting, while for others, optical bleaching occurs. When the sample contains polar molecules, the intermolecular electron transfer is more efficient in both ground and excited single states, and charge transfer and quenching between the polar molecules and the carbonyl group in the polymer are most likely to reduce the optical limiting action. However, when the sample contains nonpolar molecules, neither quenching nor charge transfer takes place, and optical limiting can be observed [12].

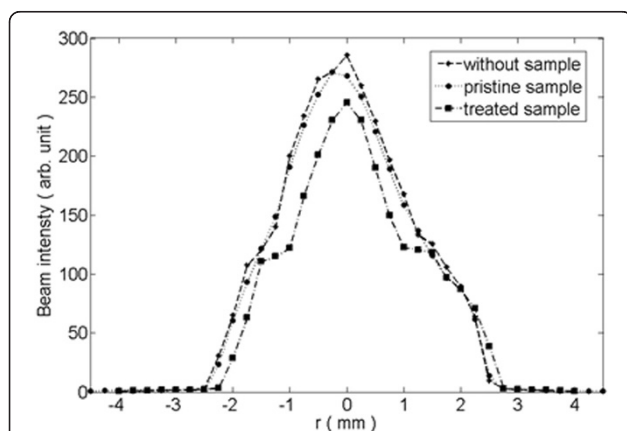


Figure 6 Intensity profiles of the laser beam intensity profiles transmitted through air, pristine, and plasma-treated red BS dye-doped PMMA films.

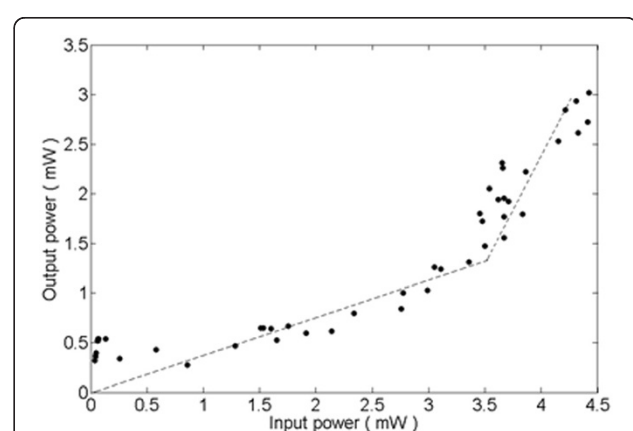


Figure 7 The nonlinear transmission behavior of red BS dye-doped PMMA films. Dashed line is the fitted line that shows the linear transmission samples.

In the samples of this experiment, C-O and C=O polar group bands lead to optical bleaching effect.

Z-scan experiment

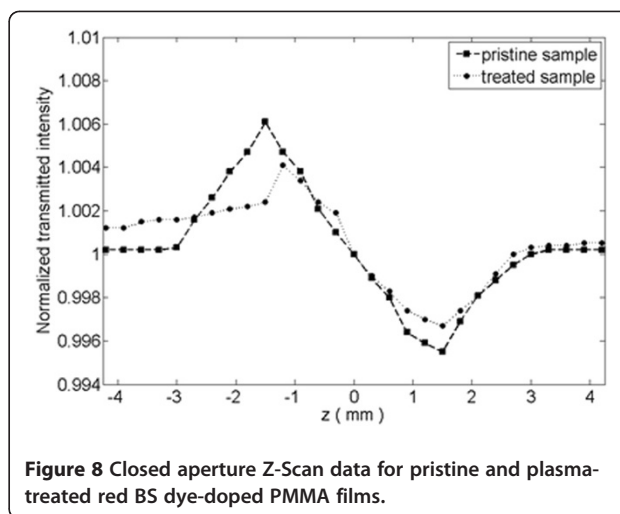
The Z-scan technique is a simple and effective tool for determining the nonlinear properties of various kinds of materials because it provides not only the magnitude of the real and imaginary parts of the third-order nonlinear susceptibility χ^3 , but also their sign. Nonlinear index of refraction n_2 is proportional to the real part of the third-order susceptibility, and the nonlinear absorption coefficient β is proportional to its imaginary part [13].

As the sample approaches the beam focus, irradiance increases, leading to self-lensing in the sample. A negative (positive) self-lens tends to collimate (diverge) the beam on the aperture in the far field before the focal plane, so the measured transmittance increases (decreases). After the focal plane, the inverse effect occurs. In fact, the nonlinear refractive index of the sample, when its thickness is smaller than the diffraction length of the focused beam, makes it to act as a thin lens with variable focal length. In the case of open aperture Z-scan, when the aperture is removed, any nonlinear absorption present in the sample can be found in the measurement. In this case, the sample transmittance is measured as a function of intensity, once the sample is scanned through the laser beam focal plane.

In the nonlinear regime, the absorption will be a nonlinear function of irradiance at a given point. If the nonlinear absorption coefficient β is positive, it increases with increasing the input power irradiance (two-photon absorption), but if β is negative, it decreases with increasing the input power irradiance (saturation absorption).

The experimental setup of Figure 2 used in this experiment while the sample is translated along the z direction and the transmitted intensity is measured through a pinhole in the far field as a function of the sample position z , measured with respect to the focal plane. As the sample moves through the beam focus ($z=0$), self-focusing or defocusing modifies the detected beam intensity. For an open aperture Z-scan, the pinhole is removed, and the transmitted beam touch the detector without any limitation.

In Figure 8, the closed Z-scan data for pristine and plasma-treated red BS dye-doped PMMA films at a laser beam intensity of $I_0 = 4.26 \text{ kW/cm}^2$ are presented. The thickness of the sample is 0.2 mm in this experiment. The peak, followed by a valley-normalized transmittance obtained from the closed aperture Z-scan data, indicates that the sign of the refraction index nonlinearity is negative, i.e., self-defocusing. In this case, Δz_{p-v} decreases from 3 mm to 2.7 mm for the plasma-treated sample compared with the pristine one. The typical Z-scan data with fully opened aperture are insensitive to nonlinear refraction; therefore, the data are expected to be symmetric with respect to the focus, but absorption in the sample



enhances the peak and decreases the valley in the closed aperture Z-scan curve, which results in the distortions in the symmetry of the Z-scan curve about $z=0$. The observed effect shown in Figure 9 is attributed to a thermal nonlinearity that resulted from the amount of absorption of radiation at 532 nm. Localized absorption of a tightly focused beam propagating through an absorbing dye medium in PMMA produces different spatial distribution of temperature in the pristine and plasma-treated samples, and consequently, difference of spatial variation of refractive index acts as a thermal lens, resulting in severe phase distortion of the propagating beam. In the case of the pristine sample, Δz_{p-v} is about $1.76z_0$, while in the case of the plasma-treated sample, this magnitude is $1.59z_0$, which leads to focusing the beam by this sample in comparison with the pristine one. In other word, plasma treatment changes the thermal effects in the material, so n_2 is more positive.

In Figure 9, the results of open aperture Z-scan experiment due to two-photon absorption phenomena are

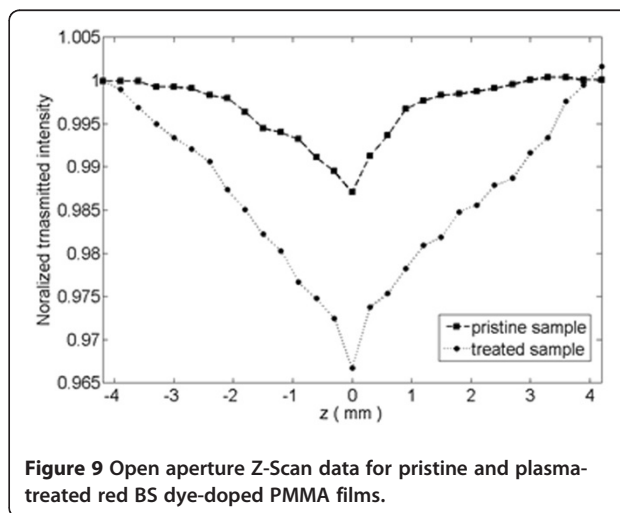


Figure 9 Open aperture Z-scan data for pristine and plasma-treated red BS dye-doped PMMA films.

presented. As can be seen, although the intensity of the transmitted beam at the peak is more than three times larger for the plasma-treated sample in comparison with the pristine one, the intensity of the transmitted beam is changed effectively, and two-photon absorption occurred. According to Sheik-Bahae's theory [13], multi-photon absorption takes place in the composite films. Since the incident photon energy $h\nu$ (2.33 eV) is less than the band-gap energy of the PMMA polymer (4.3 eV) but greater than $E_g/2$ ($E_g/2 < h\nu < E_g$), two-photon absorption is permitted. In our case, the stronger absorption of the plasma-treated sample is the result of a larger nonlinear absorption coefficient of this sample (Table 1).

As mentioned before, generally, the measurements of the normalized transmittance versus the sample position, for the cases of closed and open apertures, allow the determination of the nonlinear refractive index n_2 and two-photon absorption β .

The nonlinear absorption coefficient β can be calculated from the open aperture Z-scan data where S , the linear transmittance of aperture given in Equation 4, is equal to 0.5:

$$S = 1 - \exp\left(-\frac{2r_a^2}{\omega_a^2}\right). \quad (4)$$

In this equation, r_a is the radius of the aperture, and ω_a is the beam waist on the aperture:

$$\omega_a^2 = \omega_0^2(1 + (z_a/z_0)^2) \quad (5)$$

where ω_0 is the beam waist at the focus and z_a is the distance between the aperture and focal point; $z_0 = k\omega_0^2/2$ is the diffraction length of the beam with wave vector k .

The normalized transmittance for the open aperture condition is give as follows:

$$T(z, S = 1) = \sum_{m=0}^{\infty} \frac{[-q_0(z)]^m}{(m+1)^{3/2}}, \quad (6)$$

for $|q_0| < 1$, where:

$$q_0(z) = I_0 L_{\text{eff}} \beta / (1 + z^2/z_0^2) \quad (7)$$

where $L_{\text{eff}} = (1 - \exp(-\alpha L))/\alpha$ is known as the effective thickness of the sample; L is the thickness of the sample, α is the linear absorption coefficient, λ is the laser wavelength, and I_0 is the intensity of the laser beam at focus $z = 0$.

Here, since the closed aperture transmittance is affected by the nonlinear refraction and absorption, the determination of n_2 is less straightforward from the closed aperture scan. It is necessary to separate the effect of nonlinear refraction from that of the nonlinear absorption. A simple and approximate method to obtain purely effective n_2 is to divide the closed aperture transmittance by the corresponding open aperture scan. The data obtained in this way reflect purely the effect of nonlinear refraction [14].

The changes of the nonlinear refractive index for both pristine and plasma-treated samples can be due to different physical mechanisms, such as Kerr effect, thermal self focusing, defocusing, photo refraction etc. Here, it is attributed to the thermally induced variation in the refractive index of the medium, which resulted from the linear absorption of red BS dye-doped PMMA film. It may be noted that the linear absorption of this material occurs at 532 nm (wavelength of Nd-Yag laser), and the red BS dye-doped PMMA film investigated here is non-fluorescent at this wavelength, optimizing the conversion of the absorbed energy into heat [15].

For the calculation of n_2 , the Z-scan theory proposed by Sheik-Bahae [16] is used:

$$\Delta T_{p-v} = 0.406(1 - S)^{0.25} |\Delta\Phi_0|, \quad (8)$$

where ΔT_{p-v} is the difference between normalized peak and valley transmittance of the divided data and $|\Delta\Phi_0|$ is the induced phase distortion of radiation passed through the sample.

The nonlinear refractive index n_2 can be calculated by the following equation:

$$n_2 = \lambda |\Delta\Phi_0| / 2\pi I_0 L_{\text{eff}}. \quad (9)$$

The real and imaginary parts of the third-order nonlinear optical susceptibility χ^3 can be obtained from the nonlinear refractive index n_2 and nonlinear absorption coefficient β by the following equations:

$$\text{Re}\chi^3(\text{esu}) = (10^{-4} \epsilon_0 c^2 n_0^2 / \pi) n_2 (\text{cm}^2/\text{W}), \quad (10)$$

and

$$\text{Im}\chi^3(\text{esu}) = (10^{-2} \epsilon_0 c^2 n_0^2 \lambda / 4\pi^2) \beta (\text{cm}^2/\text{W}), \quad (11)$$

where ϵ_0 is the vacuum permittivity and c is the speed of light in the vacuum.

Table 1 Values of n_2 , β , and χ^3 of pristine and plasma-treated red BS dye-doped PMMA films

Sample	$n_2 \times 10^{-9}$ (cm^2/W)	β (cm/W)	Real (χ^3) $\times 10^{-8}$ (esu)	Image (χ^3) $\times 10^{-4}$ (esu)	ΔZ_{p-v} (mm)	FWHM (mm)
Pristine	-3.4288	0.5691	-9.8230	0.6903	3.0	2.75
Treated	-2.3937	1.4692	-7.0917	1.8428	2.7	2

Based on this theory, results of experiments are used to calculate n_2 , β , and χ^3 , which are presented in Table 1 for pristine and plasma-treated red BS dye-doped PMMA films.

The most important characteristics of the sample surface are determined by the functional groups present in the surface layer. Fourier transform Infrared (FT-IR) spectroscopy is used for this purpose. FT-IR spectrum of pristine red lake-doped PMMA and treated samples are presented in Figure 10, and the changes in the fundamental group of treated films due to plasma treatment are shown in Table 2. In this case, generated changes lead to decrease the dispersion part of surface energy. As can be realized, nitrogen ions in the plasma contribute in the formation of new bands. Noticeable growth in the C=O and C-N bands on the surface of thin film can be observed due to plasma treatment. We believe that these generated changes are responsible for the changes in nonlinear optical constants of the samples.

Conclusions

As is clear, plasma treatment affects the nonlinear parameters of polymers. In our case, by applying a 70-W dc nitrogen plasma for 8 min, which is a very common way to improve the surface characteristics of red BS dye-doped PMMA polymer film, the nonlinear refractive index and absorption coefficient of this material as well as its third-order nonlinear susceptibility are changed, while the linear parameters of this polymer are kept unchanged. These observed phenomena can be explained by the changes of structural and chemical bonds that occurred on the surface of the sample due to plasma treatment.

In conclusion, the negative lensing effect in pristine and nitrogen plasma-treated red BS dye-doped PMMA

Table 2 Changes of major peaks of FT-IR spectrum of samples before and after treatment

Wave number (cm ⁻¹)	Groups	Intensity (before treatment)	Intensity (after treatment)
1000-1350	C-O	92	68
1000-1400	C-N	38	0
1430	C=O	83	70
1479	C=O	86	51
1300-1700	C=O, N-H, N=O	82	63
1300-1700	C=C, C=O, N-H	84	80
1695	C=O	92	82
1778	C=O	92	39

film arising from thermal nonlinearity is investigated using different methods. The absolute value of nonlinear refraction index coefficient is decreased while the absolute value of nonlinear absorption coefficient is increased due to plasma treatment. In these experiments, it is confirmed that by plasma treatment, the nonlinear absorption coefficient of red BS dye-doped PMMA film has been enhanced. For third-order nonlinear susceptibility, also in our experiment, the absolute value of the imaginary part of the plasma-treated sample is three times larger than that of the pristine sample.

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Authors' information

Davoud Dorrnian was born in Tehran, Iran on October 15, 1962. He received the B.S. degree in Applied Physics from the Urmia University (Urmia, Iran), in 1991 and M.Sc. degree in Plasma Physics from I. Azad University of Tehran, Iran in 1995. From April 2000, he was doing his researches toward Ph.D. degree in Utsunomiya University (Utsunomiya, Tochigi, Japan). He presented his Ph.D. thesis "on the radiation phenomena in the interaction of high power short laser pulse with plasma" in September 2003 and received his Ph. D. degree. From November 2003 to November 2008 he has been working in Plasma Physics Research Center of I. Azad University in Tehran, Iran. He is now the research deputy and head of laser section there. He has published over 20 technical articles and letters in refereed scientific journals. His research has concentrated on nonlinear optics, intense laser interactions with plasmas and solid targets, and dusty plasma.

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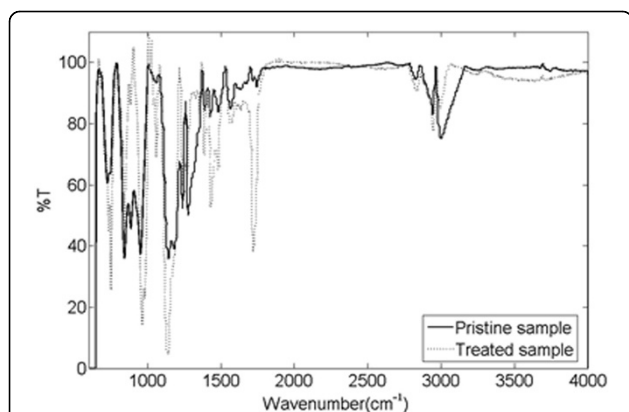


Figure 10 FT-IR spectrum for pristine and 8-min plasma-treated red BS dye-doped PMMA films.

and from 2005 till now he is dean of the Faculty of Science. He has published over 30 technical articles and letters in scientific journals. His research has concentrated on plasma physics, thin films and solid state physics.

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