

Study of Optical Limiting and Optical Phase Conjugation in DASP B Dye-doped Polymer Films

Shubhrajyotsna Aithal, Sreeramana Aithal and Gopala Krishna Bhat

Abstract— Nonlinear optical properties, optical limiting and Optical phase conjugation through degenerate four-wave mixing is observed in 4-[4-(Dimethylamino)styryl]-1-docosyl pyridinium bromide (DASP B) dye-doped in Polymethyl methacrylate – metacrylic acid (PMMA-MA) polymer films under low-power, continuous-wave laser irradiation. A maximum phase conjugate efficiency of 0.42% has been obtained for probe beam intensity at 2.5 W/cm². Phase conjugation is observed for both parallel- and orthogonally-polarized probe and pump beams. The maximum PC reflectivity is achieved when the angle between probe beam and forward pump beam is 8 degrees. The effects of dye concentration, inter beam angle between probe and forward pump beam on phase conjugation reflectivity are also studied. PC signal strength first increases and then decreases with time. PC reflectivity is also increased by increasing the intensity of the backward and forward pump beam. The polarization and intensity profile are verified to be preserved in the conjugate signal. The predominant phase conjugation signal is attributed due to the saturable absorption and two photon induced fluorescence property of the dye molecules.

Index Terms— Dye-doped polymer film, Four-wave-mixing, Optical limiting, Phase conjugation, Z-scan technique

I. INTRODUCTION

Materials with exceptional nonlinear optical properties are critical to the continuing development of photonic and electro-optical

devices, such as those used in optical communications, networking, optical computation for signal processing, and data storage equipments [1]. At present, it is with great enthusiasm to emphasis on exploring and synthesis of materials for all-optical switches based on the continual discovery of third order optical properties of new materials. There are other applications of third-order NLO materials, including optical limiting devices, Q-switch, passive mode locking, optical operation and light storage etc. These optical limiting devices are mainly based on the materials' third-order NLO properties, including self-focusing, self-defocusing, two-photon absorption, reverse saturable absorption and nonlinear scattering. Comparing to earlier laser protection devices, it has advantages of fast response, wide protected band, low optical limiting threshold, large damage threshold and high linear transmission, etc. The third-order NLO properties of materials can also be used in the compression (mode-locking) and shaping of laser pulses, optical bistability, etc. Third-order NLO materials also have many potential practical exciting applications, and motivated scientists to continually explore new materials with high third-order NLO properties. The demands of materials for all-optical information process and high-speed all-optical switches include large nonlinear refraction index, small linear and nonlinear absorption coefficient, fast response and low propagation loss [2]. Nonlinear optical phase conjugation by degenerate four-wave mixing (DFWM) is an important technique with applications in many fields of science and technology including image transmission, optical image processing, optical filtering, and laser resonators [3]. When two counter propagating and intense light beams interact with a nonlinear

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medium, together with a less intense third one, a fourth beam is generated from the medium, which will be the phase conjugation of the third beam. This technique is called four-wave mixing. The unique feature of a pair of phase-conjugate beams is that the aberration influence imposed on the forward (signal) beam passed through an inhomogeneous or disturbing medium can be automatically removed from the backward (phase-conjugated) beam passed through the same disturbing medium [4]. The main applications of degenerate DFWM techniques are nonlinear spectroscopy, real time holography, and phase conjugation. Phase conjugation by (DFWM) has been demonstrated in many organic or inorganic materials using pulsed or continuous-wave (cw) lasers. [5-6].

The organic molecules exhibit large polarizabilities because excited π -bond electrons are delocalized and hence easily polarizable. Nonlinear absorption like two photon absorption and saturable absorption plays a very important role when dyes are used for the production of phase conjugation light, because χ^3 is inversely proportional to the saturation intensity. These systems exhibit large third-order susceptibilities. OPC has been reported in Glasses and other solid matrices doped organic dyes emerged as promising materials for OPC because of their large third-order nonlinear susceptibilities $\chi^{(3)}$. In these materials, the phase-conjugate wave can be generated at low light intensities provided by the continuous-wave lasers. Moreover, these materials can be easily prepared in the laboratories. The important fundamental physical processes like nonlinear refraction, thermal grating, saturation and reverse saturable absorption, two photon induced fluorescence, photorefraction, and stimulated Brillouin scattering etc. may lead to the formation of a laser-induced grating in the medium are associated with the generation of phase conjugated wave.

Though the present best practice in Photonics technology is the usage of organic materials/dyes that exhibit exceptional nonlinear optical properties, these organic materials have few of the drawbacks inherent in the processing of comparable inorganic materials like of intense light induced degradation

or bleaching and aggregation at higher dye concentration. In order to overcome these drawbacks and for effective use of highly nonlinear dyes, the strategic idea for the next practice is doping the dye molecules in polymer matrix. This idea of dye-doped polymer material matrix may increase the concentration of absorptive or fluorescence centers as well as the opto-chemical and opto-physical stability [7 – 8].

In this paper, we have studied the third order optical properties of dye-doped polymer by considering an example of an organic dye 4-[4-(Dimethylamino)styryl]-1-docosyl pyridinium bromide, doped in a polymer matrix Polymethyl methacrylate methacrylic acid (PMMA-MA). We have studied the nonlinear properties like the two photon induced fluorescence and optical limiting capability of the dye DASPb both in solution form (in chloroform) as well as in PMMA-MA matrix. The linear absorption, single photon fluorescence, two photon induced fluorescence behavior are studied. The intensity dependent nonlinear absorption at various wavelengths and optical limiting behavior are studied using a pico second laser beam. We have also presented PC wave generation in DASPb doped in PMMA-MA polymer matrix using low-power continuous-wave laser excitation. PC signal strength at different time for different dye doped concentration, PC Reflectivity as function of angle between the probe beam and forward pump beam and transmittance as a function of time are studied.

II. DESIGN OF NONLINEAR MOLECULE

A. General Criteria

One of design strategy is proposed recently by Albota et.al, [9] dealing with molecules based on benzene ring as π -center which is attached symmetrically by either electron-donor (D) or electron-acceptor (A) through various lengths of conjugated connectors; D- π -D or A- π -A. They concluded that σ is increased by increasing the length of conjugation; change with the D/A strength and the extent of symmetric intramolecular charge-transfer (CT) from the D ends to the π -center or vice versa, meaning that symmetric charge

redistribution effectively occurs upon excitation of such symmetric molecules.

A similar approach was made in designing molecules by Reinhardt [10] and his coworkers. dealing with benzene ring as π -center which is symmetrically coupled with two electron acceptor (A- π -A) or asymmetrically with D and A (D- π -A), respectively. There is no clear effect of structural symmetry on σ values, although increasing conjugation length of π -center brings about a significant improvement of the value. In fact, an asymmetric structure, D- π -A. This seems to suggest that there must be more crucial molecular factors other than structural symmetry involved. In this study we have considered dye molecule as 4-[4-(Dimethylamino)styryl]-1-docosyl pyridinium bromide with π centre is used.

B. Sample Preparation

Commercially available DASPB (Aldrich Chemical Co.) is purified by recrystallization twice with spectrograde ethanol and by vacuum sublimation. The purity is determined spectroscopically. Purified chloroform is used as the solvent. To prepare the film, Polymethyl methacrylate – metacrylic acid was used as polymer matrix. The thin films of DASPB doped in PMMA-MA is prepared using hot press technique. Thin films of variable thickness are obtained between two glass slides.

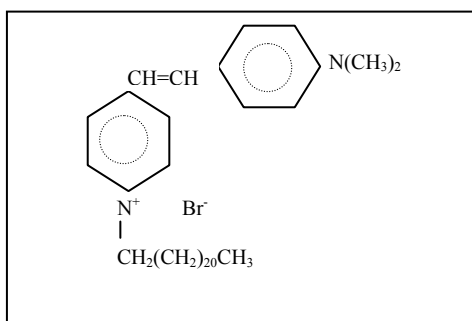


Fig. 1. Molecular structure of DASPB.

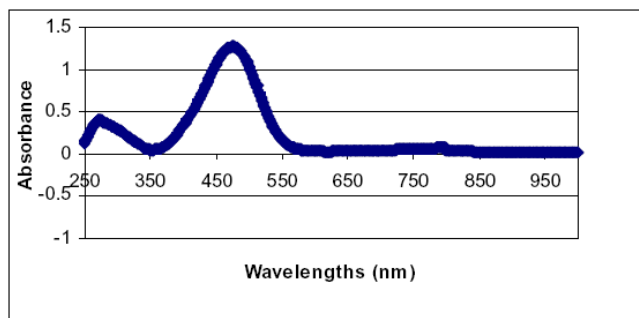


Fig. 2. Linear absorption spectrum of DASPB in PMMA-MA polymer matrix.

C. Linear Optical Properties of DASPB

The molecular structure of DASPB is shown in Figure 1. A charge-transfer in between the aromatic moiety (electron donor) and bromine unit (electron acceptor) can be proposed to explain large χ^3 value measured using Z-scan technique. The linear absorption spectrum of DASPB in chloroform is measured on a VARIAN Cary UV-vis-IR recording Spectrophotometer by using quartz cuvette with one cm path length as well as doping it in Polymethyl methacrylate methacrylic acid (PMMA-MA) film. The Figure 2 shows the linear absorption spectrum of a DASPB in chloroform with solute concentration of $d_0 = 0.0001$ mol/L, in which the solvent influence is not included. The spectral curve has shown that there is a strong absorption band with peak absorption located at 478 nm with a bandwidth of 100 nm, a medium absorption peaked at 270 nm with a bandwidth of 80 nm and no linear absorption is observed in entire spectral range of 580 to 2000 nm except IR absorption between 1200 nm to 1600 nm.

The single photon fluorescence spectrum of the sample is measured for a 1 cm –path DASPB in chloroform with the solute concentration of 0.0001 mol/L using a spectral fluorophotometer (Rf 50000U from Schmadza) with the spectral resolution of 1 nm. The peak wavelength of the single-photon induced fluorescence was 610 nm with a bandwidth of 60 nm (Figure 3). Figure 4 corresponds to single photon fluorescence when DASPB is excited at 532 nm using an Nd:YAG laser beam.

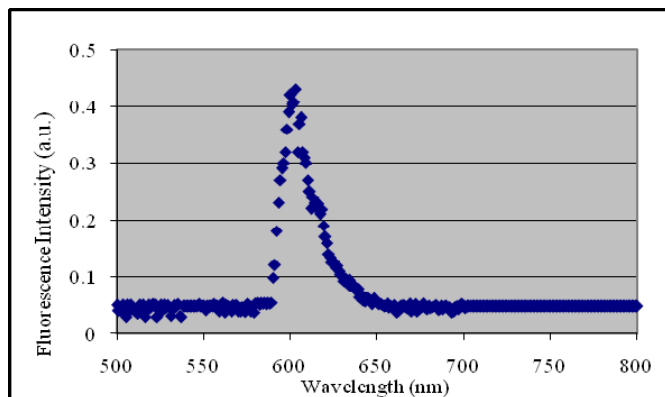


Fig. 3. One photon induced emission spectrum of DASP in chloroform.

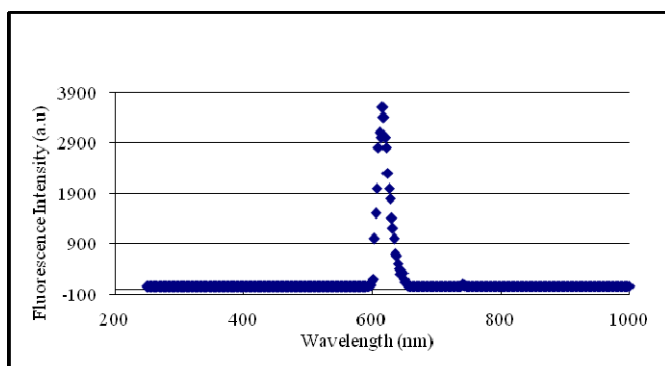


Fig. 4. One photon induced fluorescence at 532 nm irradiation.

D. TPA Cross-section Measurement

From absorption spectrum of DASP, we can see that there is no linear absorption in the entire spectral range from 580 nm to 1800 nm except the fact that IR radiation between 1,200 nm to 1,600 nm is strongly absorbed by DASP solution. It has been observed that this dye shows quite strong frequency unconverted fluorescence when exposed to near IR and IR laser beam above 700 nm. This suggests that a very strong TPA process may occur inside the sample.

E. Two-photon Excited Fluorescence Emission

The TPA induced emission spectrum of 0.0005 mol/L DASP in chloroform with 1 cm path length excited with 1064 nm laser beam is shown in Figure 5. In the measurement of the upconversion efficiencies, VIS cutting filters were used to cut transmitted pump energy. Comparing Figure 5 with Figure 4, we can see that the TPA induced emission spectrum of DASP with much higher

concentration has a red-shift as compared to that in much lower concentration single photon absorption study. This can be explained by re-absorption of Dye material [11, 12].

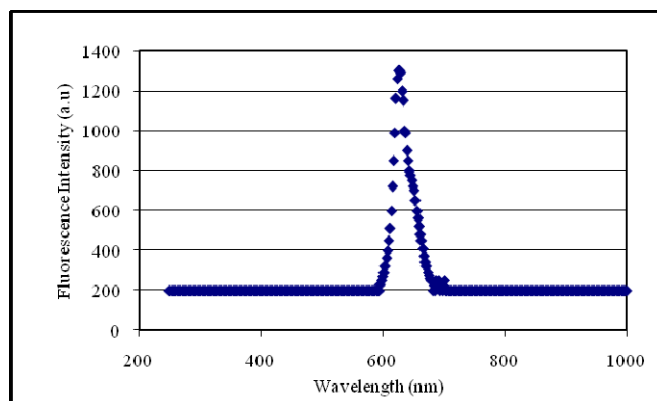


Fig. 5. Two-photon induced emission spectrum of DASP at 1.06 μm irradiation.

III. NONLINEAR OPTICAL PROPERTIES

A. Nonlinear Absorption Study

Z-scan technique is used to study the nonlinear optical properties of the sample [13 - 14]. In Z-scan approach, a single beam is tightly focused into a thin nonlinear medium. The transmittance through a small aperture in the far-field is measured and this allows the measurement of nonlinear refractive index n_2 . By removing the aperture, measurement of the transmittance allows the determination of the two photon absorption coefficient β . Figure 6 shows the experimental setup for the measurement of nonlinear absorption including saturation absorption in linear absorbing region and two-photon absorption in transmitting region of DASP. In open aperture ($S=1$) configuration, the system is insensitive to nonlinear refraction, and can be used to measure the nonlinear absorption cross section. Such Z-scan trace with no aperture is expected to be symmetric with respect to the focus ($Z = 0$), where the minimum transmittance (e.g., multi-photon absorption) or a maximum transmittance (e.g., saturation of absorption) occurs. The nonlinear coefficient can be easily calculated from Z-scan transmittance curve.

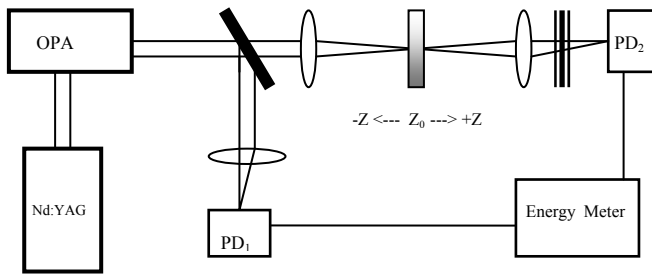


Fig. 6. Experimental set-up used for the measurement of the total transmittance and overall energy conversion efficiencies.

Figure 7 & 8 show the Z-scan results for DASPB at 532 nm using cw and pico second pulses at low energy and the material shows strong saturable absorption. Even if the linear transmittance of the sample is low, it is observed that due to nonlinear absorption, transmittance of the sample is increased initially with increase in intensity. But it is interesting to note that, the saturation absorption is over taken by reverse saturation absorption effect at higher input irradiance (figure 9). The transformation from SA to RSA is with increase in fluency level is first observed in organic dyes and this behavior can be used for optical limiting as well as optical switching.

Once an Open aperture Z-scan with $S = 1$ is performed, the two photon absorption coefficient β can be deduced. By knowing the concentration of dopant, the molecular two photon cross-section σ'_2 can be determined by using following relationship, with d_0 as molecular density of the dye.

$$\beta = \sigma_2 N_0 = h\nu \sigma'_2 N_A d_0 \times 10^{-3}$$

The nonlinear absorption coefficient β is estimated from the data in Fig. 8 by studying the best fit according to the relationship, $I(L) = [\ln(1 + I_0 L \beta)] / L \beta$ where $I(L)$ is the transmitted beam intensity, I_0 is the incident beam intensity, L is the thickness of the sample.

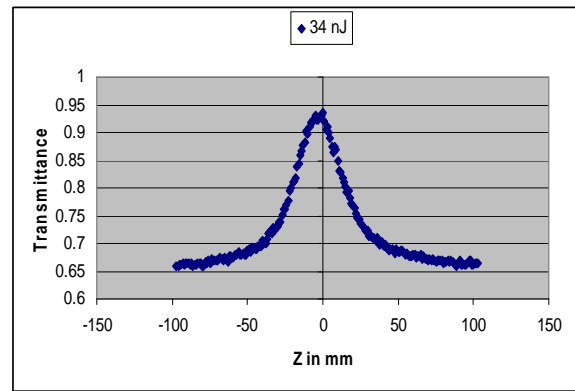


Fig. 7 (a). Picosecond 532nm open aperture Z-scan curves at energy 34 nJ.

To study the behavior of DASPB at its linear absorptive region, both pico (32 ps, 10 Hz.) and nano second laser (6 ns, 20 Hz.) beams (from second harmonic of a Q-switched, mode-locked Nd:YAG laser system and operating at 532 nm) are used. Two photon absorption cross section is measured for 0.0005 mol/L DASPB with sample thickness 1 mm in PMMA-MA polymer of thickness 0.1 mm by using a focused 32 ps mode-locked Nd:YAG laser at wavelength range 800 nm to 1000 nm [15]. The pump laser beam from the output of OPA was focused by a convex lens ($f = 15$ cm) on to the centre of the DASPB film. The pump energy and the output energy were recorded simultaneously by a two-channel energy meter. In the measurement of nonlinear absorption, IR cutting filters were used to cut the upconverted energy. All the experiments were done at room temperature. Table 1 gives the value of nonlinear coefficient β and corresponding TPA cross section σ'_2 at different wavelengths and corresponding peak irradiance.

TABLE I
EXPERIMENTAL VALUES OF TPA
COEFFICIENTS AND TPA CROSS SECTIONS
AT DIFFERENT WAVELENGTHS

Wavelength (nm)	β in cm/GW	σ'_2 in $\text{cm}^4 \cdot \text{s} / \text{photon}$
800	5.6	3.91×10^{-47}
900	6.4	4.23×10^{-47}
1000	5.9	3.98×10^{-47}

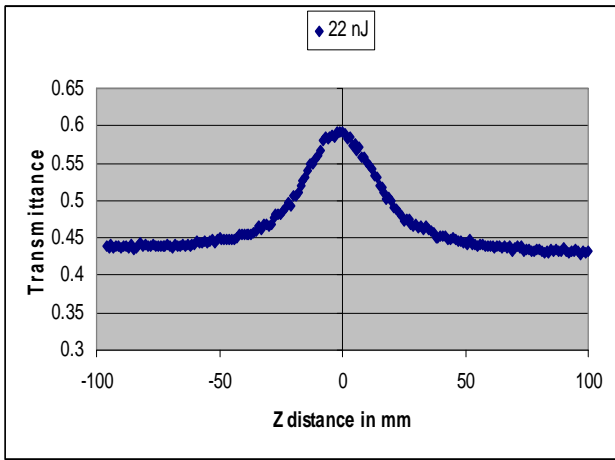


Fig. 7 (b). Picosecond 532nm open aperture Z-scan curves at energy 22 nJ.

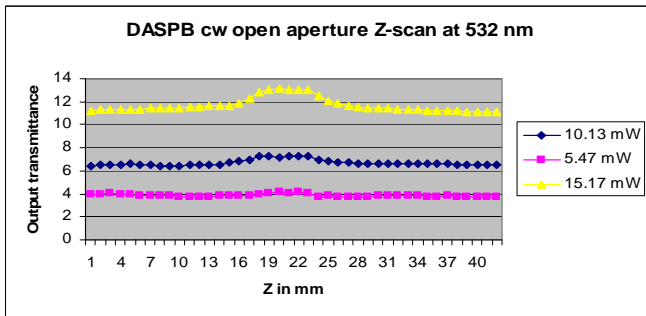


Fig. 8. CW 532 nm open aperture Z-scan at different input power.

B. Optical Limiting Study

The linear transmission of dye in the form of film was about 10 %. The optical limiting curves using 532 nm pico second and nano second pulses are shown in figure 11 and figure 12 respectively. Optical limiting study is performed in two photon absorbing region using pico-second 800 nm, 900 nm and 1,000 nm laser beam (figure 13). From the figures it can be noted that at very low input intensity, linear transmission is preserved, with increase in input intensity DASPB starts to contribute to saturation absorption which increases the transmission above linear level and at higher intensities the excited state absorption starts to contribute and it dominates with further increase in intensity. This kind of behavior has interesting applications in Optical switching [16].

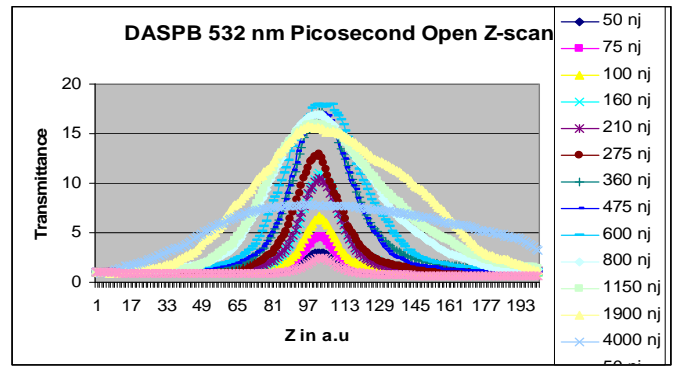


Fig. 9. Pico second 532 nm open aperture Z-scan of DASPB at different energies.

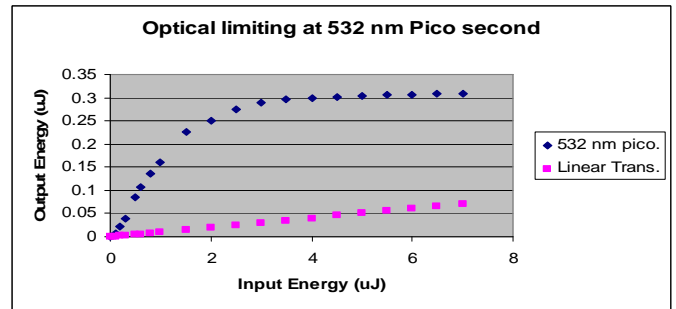


Figure 11. Optical limiting behavior at 532 nm pico second pulses.

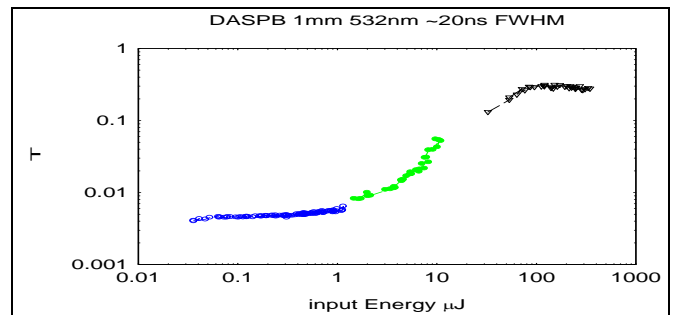


Figure 12. Optical limiting behavior of DASPB at 532 nm nano second pulses.

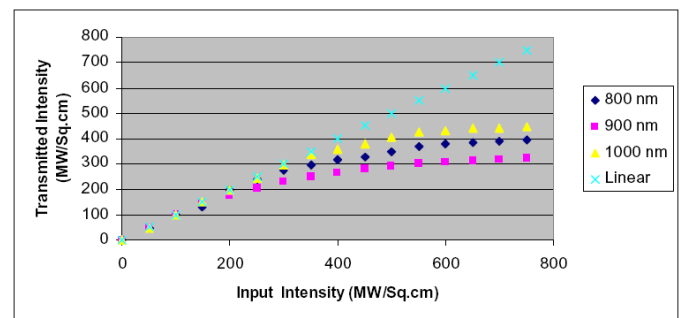


Figure 13. Optical limiting behavior of DASPB at 800 nm, 900nm and at 1000 nm.

IV. STUDY OF PHASE CONJUGATION

A. Experimental Configuration For DFWM

The schematic diagram of the phase conjugation experiment is shown in Figure. 14. A He-Ne laser (25 mW) beam at 633 nm was divided into three beams, two counter-propagating pump beams E_1 and E_2 namely forward-pump and backward-pump beams respectively and a probe beam E_3 to form the DFWM configuration. The spot size of each of these three unfocussed beams at the nonlinear medium was 1.0 mm in diameter. The constant power ratio of the probe beam (E_3), forward-pump beam (E_1) and backward-pump beam (E_2) used in this work was $\approx 1 : 10 : 10$. The angle between the probe beam and the forward-pump beam was 8° . The sample was exposed simultaneously to all these three beams. The optical path lengths of all the three beams were made equal, so that they were coherent at the sample. The phase-conjugate wave retraces the path in the opposite direction to that of the probe beam E_3 and was detected with the help of a photo detector and power meter. The experimental set-up was mounted on a vibration isolation table to avoid the destruction of the laser-induced gratings formed in the DASPB dye-doped polymer matrix due to mechanical disturbances.

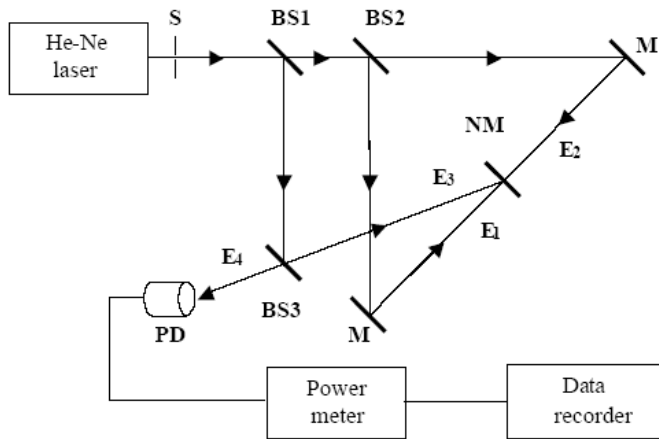


Fig. 14. Experimental set-up for observation of PC wave, S, Shutter; BS1–BS3, Beam splitters; M, Mirror; NM, Nonlinear medium; PD, Photo-detector.

B. Results of Degenerate Four Wave Mixing

The PC signal measurements are taken by varying the parameters which influence the PC

signal reflectivity during the DFWM process. Figure 15 shows the PC signal versus the time for different dye concentration of the doped polymer films. PC intensity rises linearly to a maximum and then starts decreasing. The phase grating formed is transient. To get maximum reflectivities, it is necessary that there be a perfect overlap of the probe and the pump beams in the nonlinear medium. Figure 16 shows the PC reflectivity as a function of recording angle between the forward pump and probe beam. It seems from the figure that, as the angle between the probe beam and the forward pump beam increases, the PC reflectivity first increases and then decreases. This may be because as the angle increases, the probe beam becomes elliptical and only a fraction of its area falls within the interaction region. Because of two-wave coupling, the maximum PC reflectivity is achieved when the angle is 8 degrees. A maximum reflectivity value of 0.42% is observed for probe beam intensity at 2.5 W/cm^2 , and further increase in probe beam intensity resulted to decrease in PC reflectivity.

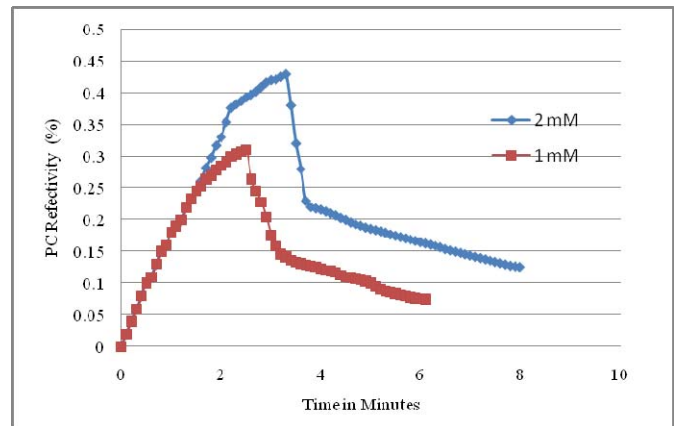


Fig. 15. PC signal versus recording time for 0.001M and 0.002 M concentrations.

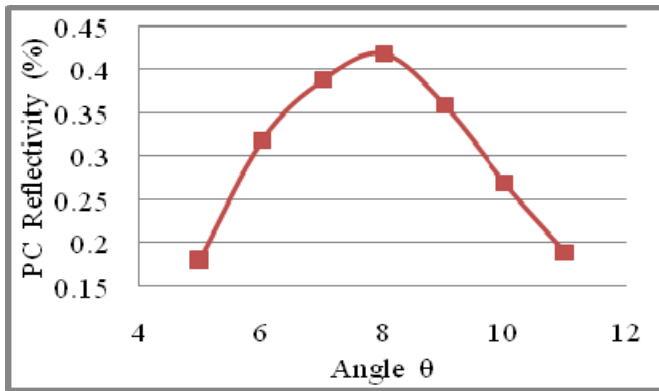


Fig. 16. PC Reflectivity as function of angle between the probe and forward pump beams.

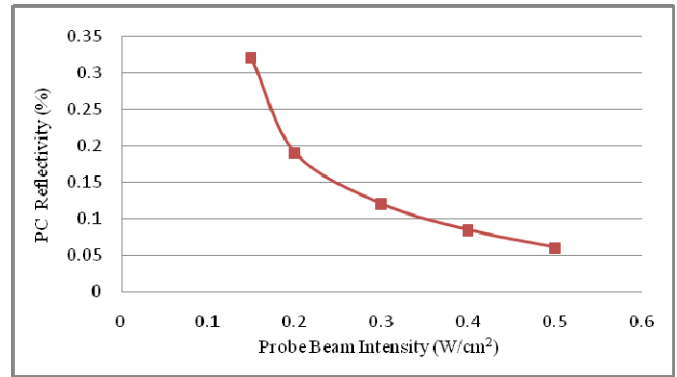


Fig. 18. Conjugate reflectivity as a function of probe beam intensity.

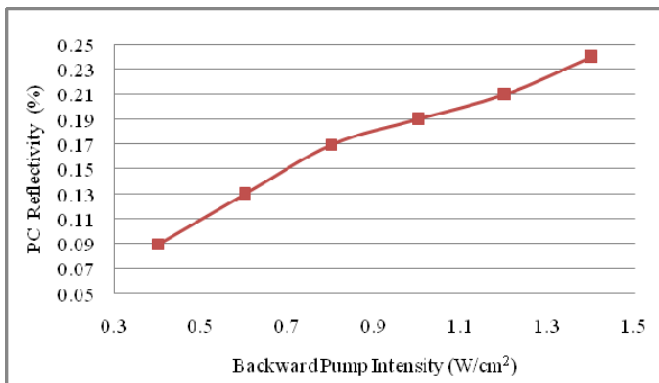


Fig. 17. Dependence of PC reflectivity on backward pump Intensity

The effect of the backward pump beam power on the PC reflectivity by keeping the power of the forward pump and probe beams constant and varying the backward pump beam is shown in Fig. 17. Figure 18 shows the influence of the input probe beam intensity on the conjugate beam reflectivity. A maximum reflectivity value of 0.42% is observed for probe beam intensity at 2.5 W/cm², and further increase in probe beam intensity resulted to decrease in PC reflectivity. Similar observations have been reported in other kinds of material doped with organic dyes [17].

Figure 19 shows the variation of reflectivity for different power of forward pump beam. The PC reflectivity increases linearly with the power of forward pump beam. There are two main processes which must be considered in the discussion of origin of OPC in dye doped PMMA-MA films: (1) the formation of thermal grating and (2) third order nonlinear optical processes.

The DASP dye doped film illuminated with 633 nm radiation of variable intensity and the transmittance of the sample is measured simultaneously by using photodetector. If the effect observed in our experiments is of purely thermal nature, bleaching of the sample film will be observed. The results obtained for the sample are shown in Fig. 20. It is clearly demonstrated that the transmission of sample increases with time. The experiment described above indicates that the third order nonlinear processes like two photon absorption is mainly responsible for OPC in the sample under study.

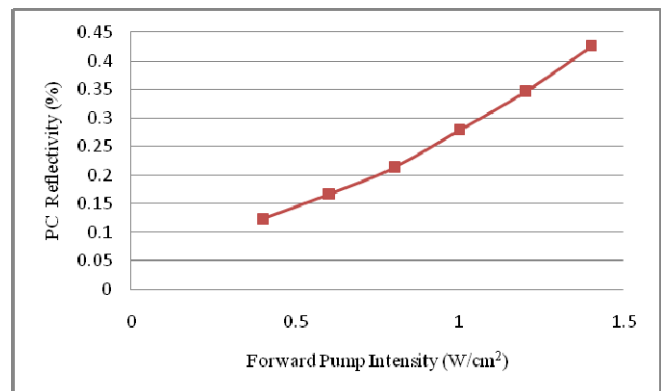


Fig. 19. Dependence of PC reflectivity on forward pump power.

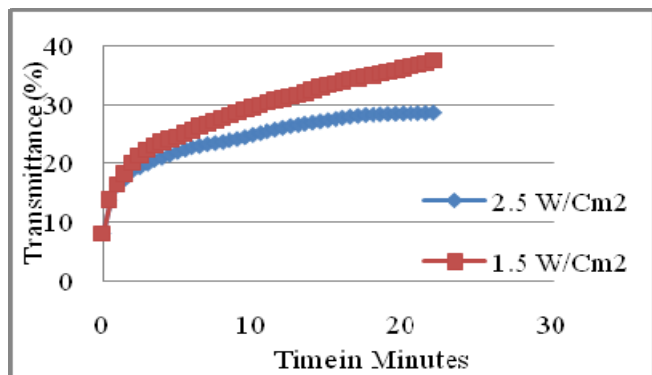


Fig. 20. Transmittance as a function of time.

V. CONCLUSION

We have characterized the nonlinear optical properties of DASPB in PMMA-MA polymer matrix at its linear absorbing region and linear transmitting region. We found that the type of nonlinear absorption depends on the intensity of input beam. In linear absorption region, at lower intensity, the dye has shown saturation absorption and with increase in input intensity, the excited state absorption became prominent. This mechanism contributed to optical limiting behavior in absorbing region of the dye. In non-absorbing region, the two photon induced fluorescence along with excited state absorption contributed for optical limiting. It is also found that DASPB has a much larger TPA cross-section of Rhodamine 6G listed in literature [18-19] as $10^{-48} - 10^{-50}$, at least one order of magnitude larger. We have also observed low-intensity optical phase-conjugation in DASPB dye in PMMA – MA polymer matrix using a degenerate four-wave mixing set-up, employing 633 nm light radiation from a He-Ne laser. The mechanism of phase-conjugate wave generation associated with this dye-doped system is discussed. The phase-conjugate signal is found to have contributions from the DFWM and the holographic processes. The maximum phase-conjugate beam reflectivity observed in these dye films is about 0.42%. The maximum PC reflectivity is achieved when the angle between probe and forward pump beam is 8 degrees. The effects of dye intensity backward, forward pump and interbeam angle between probe and forward pump beam on phase conjugation

reflectivity are also studied. PC signal first increases and then decreases. PC reflectivity is increased by increasing the intensity of the backward and forward pump beam. The polarization and intensity profile are verified to be preserved in the conjugate signal. The predominant phase conjugation signal is attributed to the fact that saturable absorption and two photon induced fluorescence property of the dye molecules. The decay time of the recorded grating at fixed pump intensity also decreases with decreasing temperature. Since the DASPB dye in PMMA – MA polymer film is used at 633 nm and this may be suitable for low-power semiconductor lasers in the red wavelength region, DASPB dye in PMMA – MA polymer film may be a promising material for real-time double-exposure phase-conjugate interferometry.

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