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RESEARCH

Extraction of Natural Pigment from *Ocimum tenuiflorum* Using Different Polar Solvents and Their Nonlinear Optical Characteristics

S. Jeyaram[1] · **D. Jeancy Rany**[2]

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Abstract

Herein, we report the extraction, characterization, linear and third-order nonlinear optical (TONLO) features of the natural pigment from *Ocimum tenuiflorum* leaves using different polar solvents. The polar solvents such as, ethanol, acetone, methanol and dimethyl sulfonate (DMSO) were used to extract the natural pigment from *Ocimum tenuiflorum* leaves. The effect of solvent on linear and TONLO properties of the natural pigment was studied and multi- parameter scale known as Kamlet-Abboud-Taft was used to analyse the solvent characteristics on solute molecule. The functional groups of the extracted natural pigments were examined via Fourier transform infrared (FT-IR) spectrometer. TONLO characteristics of the natural pigment was scrutinised by a continuous wave (CW) diode laser working at 650 nm wavelength. The nonlinear refractive index (n_2) and nonlinear coefficient of absorption (β) of the natural pigment was ascribed to the behavior of self-defocusing, saturable absorption (SA) and reverse saturable absorption (RSA). The TONLO susceptibility ($\chi^{(3)}$) of the natural pigment in polar solvents was found to be the order of 10^{-6} esu. The natural pigment extracted from *Ocimum tenuiflorum* leaves is a potential material for applications in nonlinear optical.

Keywords Nonlinear optical properties · *Ocimum tenuiflorum* · Polar solvents · Z–scan · Multi parameter scale

Introduction

With the widespread usage of lasers in a variety of applications, there is indeed a lot of interest in finding novel materials for nonlinear optical (NLO) applications. In the past three decades, organic materials are attracted by many research groups due to large NLO properties, fast response time, molecular flexibility and significant hyperpolarizability [1–3]. Among the organic materials, a large number of organic dyes with π-conjugated electronic structures have been investigated [4–7]. These materials have been widely used in all optical switching devices, optical data storage, optical limiting, optical computing, harmonic generation, etc., [8–11]. However, organic materials require an elaborated preparation process, chemically toxic and costly synthesis process. Thus, there is a growing interest in exploring with less restriction of some natural dyes extracted from plant leaves and vegetables with similar optical properties [12]. In recent times, natural dyes such as chlorophylls, carotenoids, red carmine, Chinese tea, betanin, anthocyanin, Saffron, Henna, have been extracted from plant leaves and vegetables [13–22]. A large TONLO susceptibility was observed in natural pigments is due to high transition dipole moment and strong narrow absorption characteristics [23]. Specific natural dyes were found to exhibit very attractive NLO features with nonlinear absorption coefficients 10–10,000 times greater than that of some standard materials such as CS_2, bismuth borate glasses and sulphur rich compounds [23]. *Ocimum tenuiflorum* is an aromatic perennial plant in the family of lamiaceae and mostly cultivated in the Indian subcontinent. It is widely used in herbal tea and commonly used in Ayurveda. Traditional uses of *Ocimum tenuiflorum* aqueous extracts are to treat different types of poisoning, stomach-ache, headache, malaria, inflammation and heart disease [24].

Intermolecular interactions, being one of the most important aspects, can have a considerable impact on the optical characteristics of materials. The physical, chemical and

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✉ S. Jeyaram
jeyaram.msc@gmail.com

[1] Department of Physics, Takshashila University, Ongur (PO), Tindivanam (TK), Villupuram 604305, Tamilnadu, India

[2] Department of Chemistry, School of Engineering and Technology, Surya Group of Institutions, Vikravandi, Villupuram 605652, Tamilnadu, India

biological behaviors of the molecules are influenced by their surrounding environment [25, 26]. Therefore, understanding the type of interaction between solute and solvent is most important and it is derived into specific (hydrogen bonding) and non-specific (dielectric enrichment) interactions. The solvent effects on the solute molecules can be investigated by means of solvent polarity scale and solvatochromism [27]. Individual solvent polarities parameters cannot be used to describe the solute–solvent interactions, because of large number of chemical and physical process are involved. Therefore, multi-parameter solvent polarity scales, known as Kamlet-Abboud-Taft solvent polarity parameters is used to investigate the solute–solvent interaction [28]. This paper reports the study of TONLO properties of natural pigment extracted from *Ocimum tenuiflorum* leaves using different polar solvents, namely, acetone, ethanol, methanol and DMSO. The CW diode laser operating at 650 nm wavelength with total power of 5 mW was used to execute the Z–scan study.

Experimental

Sample Preparation

The leaves of *Ocimum tenuiflorum* were collected from the university campus and dried in sunlight for a week. The dried leaves were pulverized into a fine powder. Highly analytical grade polar solvents such as acetone, ethanol, methanol and DMSO were purchased from Sigma Aldrich and used as such. The *Ocimum tenuiflorum* powder with 0.01 gm was dissolved into 10 ml of polar solvents and filtered using Whatman No. 1 filter paper. The obtained natural pigments were stored in a refrigerator for future study.

Method

Z–scan technique is a sensitive method to measure the nonlinear refraction (n_2) and nonlinear absorption (β) coefficient of materials [29]. Z–scan technique consists of a solid state diode laser working at 650 nm wavelength with total power of 5 mW. The beam was focused by a converging lens with the focal length of 5 cm. A cuvette containing the natural pigments with path length $L = 1$ mm was used, which is placed on the micrometer translational stage. The sample was moves along the propagation of beam direction between –Z and + Z positions. A photodetector connected to a digital power meter was placed at a far-field position, in order to measure the beam transmittance. The thin sample condition was verified, because the Rayleigh length is greater than sample length. Figure 1 is the schematic experimental setup of Z–scan technique.

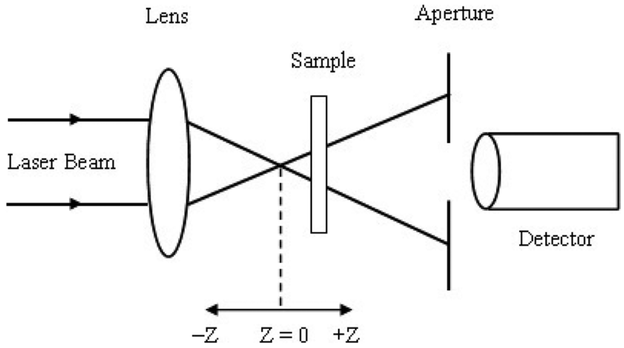

Fig. 1 Z–scan experimental setup

Results and Discussion

Effect of Solvent on Linear Absorption Behavior of Natural Pigment

The linear absorption coefficient of the natural pigment in different polar solvents was studied by UV–Visible spectrophotometer between 400 and 800 nm wavelength. The solvent polarity has a significant factor that affects the linear absorption coefficient of the natural pigment. The UV–visible absorption spectra of the sample dissolved in different polar solvents such as methanol, acetone, ethanol and DMSO is depicted in Fig. 2. The absorption peak of the pigment in all the solvents are at the resonance absorption range of used laser source. Further, the absorption peaks of the pigment vary with respect to the polarity of the used solvent. The UV–visible absorption spectrum covers the entire visible region and the absorption shift towards a red region by increasing the solvent polarity is

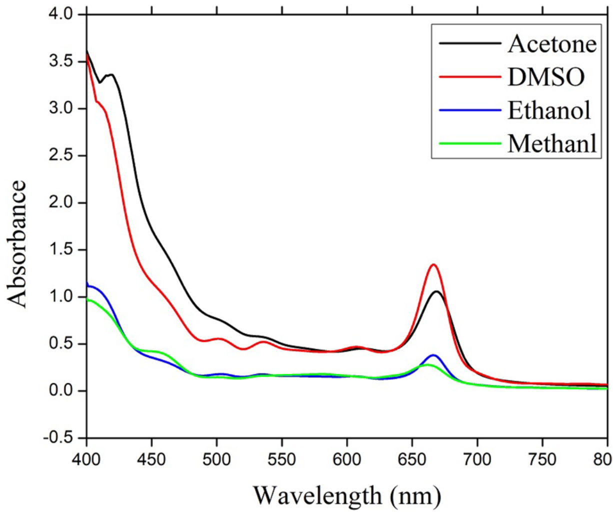

Fig. 2 UV–Visible absorption spectra of natural pigment in polar solvents

Table 1 Linear and spectral parameters of polar solvents

Solvent	Linear refractive index (n_0)	Dielectric constant (ε)	Hydrogen bond donor (α)	Hydrogen bond acceptor (β)	Polarizability (π*)	Linear absorption coefficient (α_0/cm)
Methanol	1.329	32.7	0.98	0.66	0.60	0.628
Acetone	1.358	20.7	0.08	0.48	0.62	2.438
Ethanol	1.361	24.50	0.86	0.75	0.52	0.878
DMSO	1.479	46.68	0.00	0.76	1.00	3.092

the result of positive solvatochromism or bathochromic shift [26]. The spectral features and linear absorption coefficient of the natural pigment is presented in Table 1.

FT-IR Study

The FT-IR spectrum of the natural pigment is recorded using Thermo Scientific Nicolet iS50 FT-IR Spectrometer. The FT-IR spectrum of the natural pigment extracted from *Ocimum tenuiflorum* using ethanol as a solvent is depicted in Fig. 3 and the corresponding vibrational frequencies are tabulated in Table 2. An intense band appeared at 3444 cm^{-1} is due to stretching of hydroxyl (O – H) group. The stretching frequencies at 2932 cm^{-1}, 2873 cm^{-1} and 2814 cm^{-1} are owing to aromatic C – H stretching group. The frequency band at 2814 cm^{-1} is owing to the presence of C – H stretching. A weak band at 2506 cm^{-1} is due to stretching of O – H group. The frequency at 1651 cm^{-1} is responsible for carboxamide carbonyl group. A band at 1496 cm^{-1} is owing to bending of O – H group. A sharp intense peak at 1390 cm^{-1} is associated with C – H bending frequencies. The C – O stretching vibrations was observed at 1255 cm^{-1} and 1099 cm^{-1}, respectively. The weak band at 1062 cm^{-1} confirms the presence of S = O stretching group. Furthermore, the medium band at 662 cm^{-1} is related to bending of C – OH group.

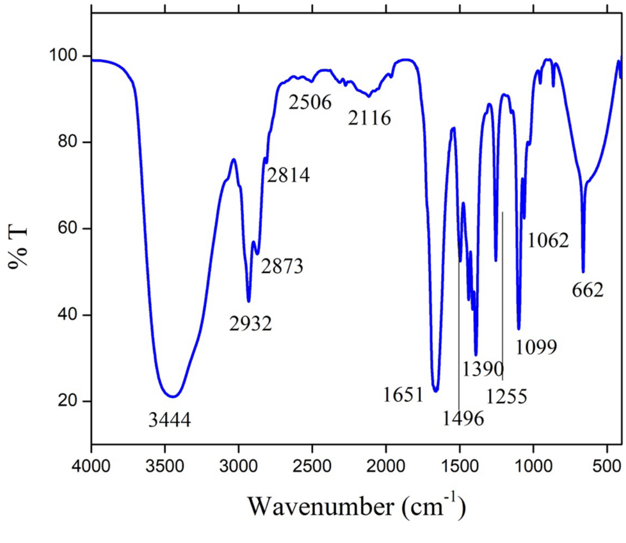

Fig. 3 FT-IR spectra of the natural pigment

Effect of Solvent on TONLO Features of the Natural Pigment

TONLO features of the natural pigment extracted from *Ocimum tenuiflorum* leaves were determined from open aperture (OA) and closed aperture (CA) methods, respectively. Figure 4a–d is the OA Z−scan result of the natural pigment dissolved in acetone, ethanol, methanol and DMSO. The positive and negative nonlinear absorption coefficient was obtained from OA transmittance curve due to SA and RSA phenomenon. The OA Z−scan curve of the natural pigment dissolved in acetone and ethanol shows SA feature, whereas the sample shows RSA property in methanol and DMSO. Both SA and RSA are the materials behaviors that arises from an increases or decreases of light transmittance at the focus (Z = 0). The beam intensity is maximum at the focus and forms a sharp peak is the outcome of SA. SA arises at high input intensities, which causes significant photon absorption before relaxing to the lowest level. In contrast, the natural pigment dissolved in methanol and DMSO exhibits the RSA behavior, due to strong interaction between light intensity and sample at the focus. Therefore, maximum absorption was observed and hence the light transmittance decreases at the focus.

Table 2 Vibrational assignments of natural pigments extracted from *Ocimum tenuiflorum*

Wavenumber (cm^{-1})	Group
662	C–OH bending
1062	S = O stretching
1099	C–O stretching
1255	C–O stretching
1390	C–H bending
1496	O–H bending
1651	C = O stretching
2506	O–H stretching
2814	C–H stretching
2873	C–H stretching
2932	C–H stretching
3444	O–H stretching

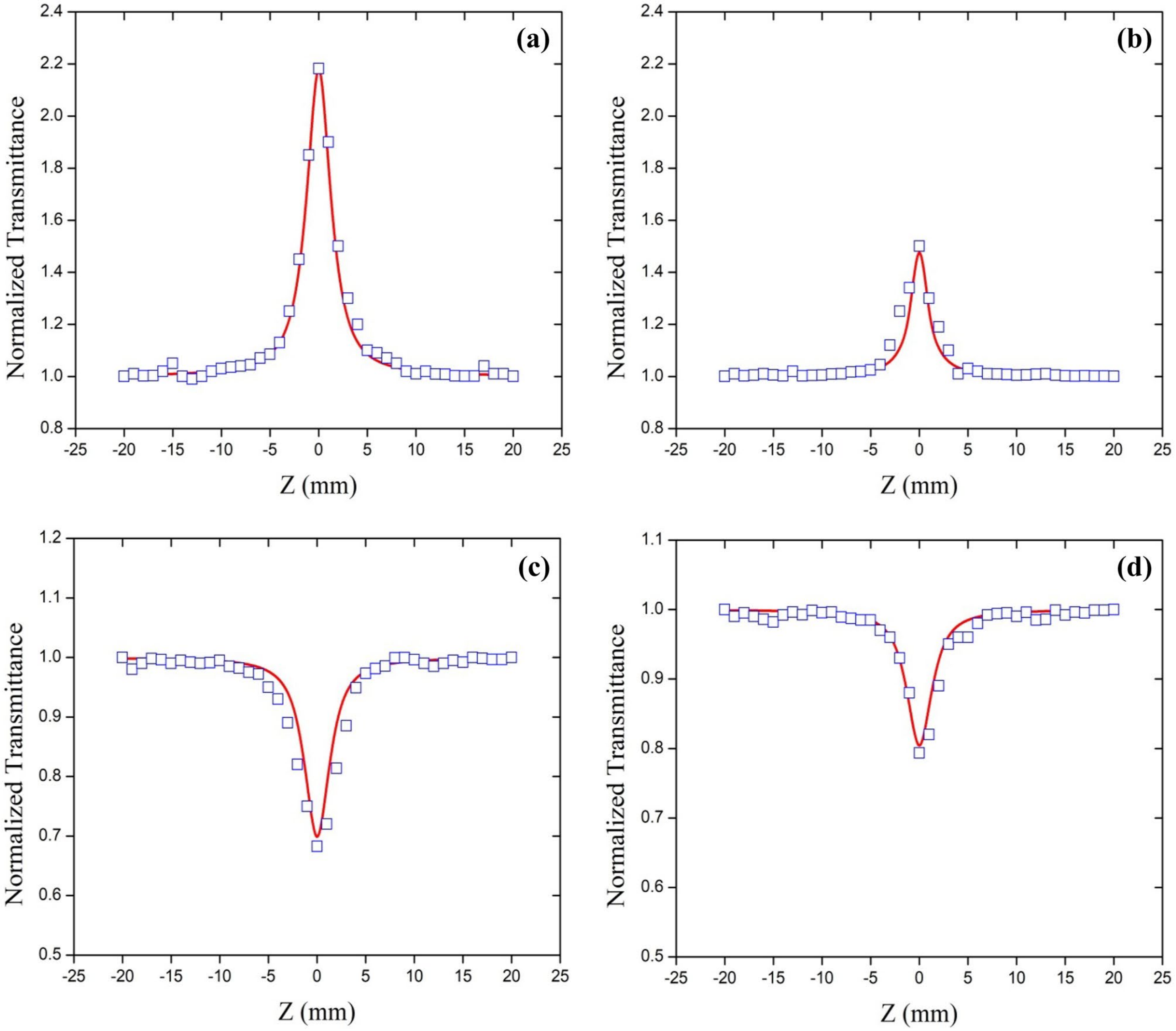

Fig. 4 Open aperture Z–scan curve of natural pigment in **a** acetone **b** ethanol **c** methanol **d** DMSO

The absorption cross-section of the excited state level is greater than ground state level is the outcome of RSA. Generally, the RSA property of the sample is explained by the five-level model, and the detailed explanation was previously reported [10]. The OA normalized transmittance is given by,

$$T(z, s = 1) = \sum_{m=0}^{\infty} \frac{\left[-q_o(z)\right]^m}{[m+1]^{\frac{3}{2}}},\ for\ \left|q_o(0)\right| < 1 \tag{1}$$

where

$$q_0 = \frac{\beta I_o L_{eff}}{\left(1 + {Z^2}/{Z_0^2}\right)} \tag{2}$$

where

$\mathrm{L_{eff}}$ = effective length of the sample

$\mathrm{Z_o}$ = diffraction length of the sample

The absorption coefficient (β) of the natural pigment extracted from *Ocimum tenuiflorum* in different polar solvents is given by,

$$\beta = \frac{2\sqrt{2}\Delta T}{I_0 L_{eff}}\left(\frac{cm}{W}\right) \tag{3}$$

The nonlinear index of refraction n_2 of the natural pigment extracted from *Ocimum tenuiflorum* using different

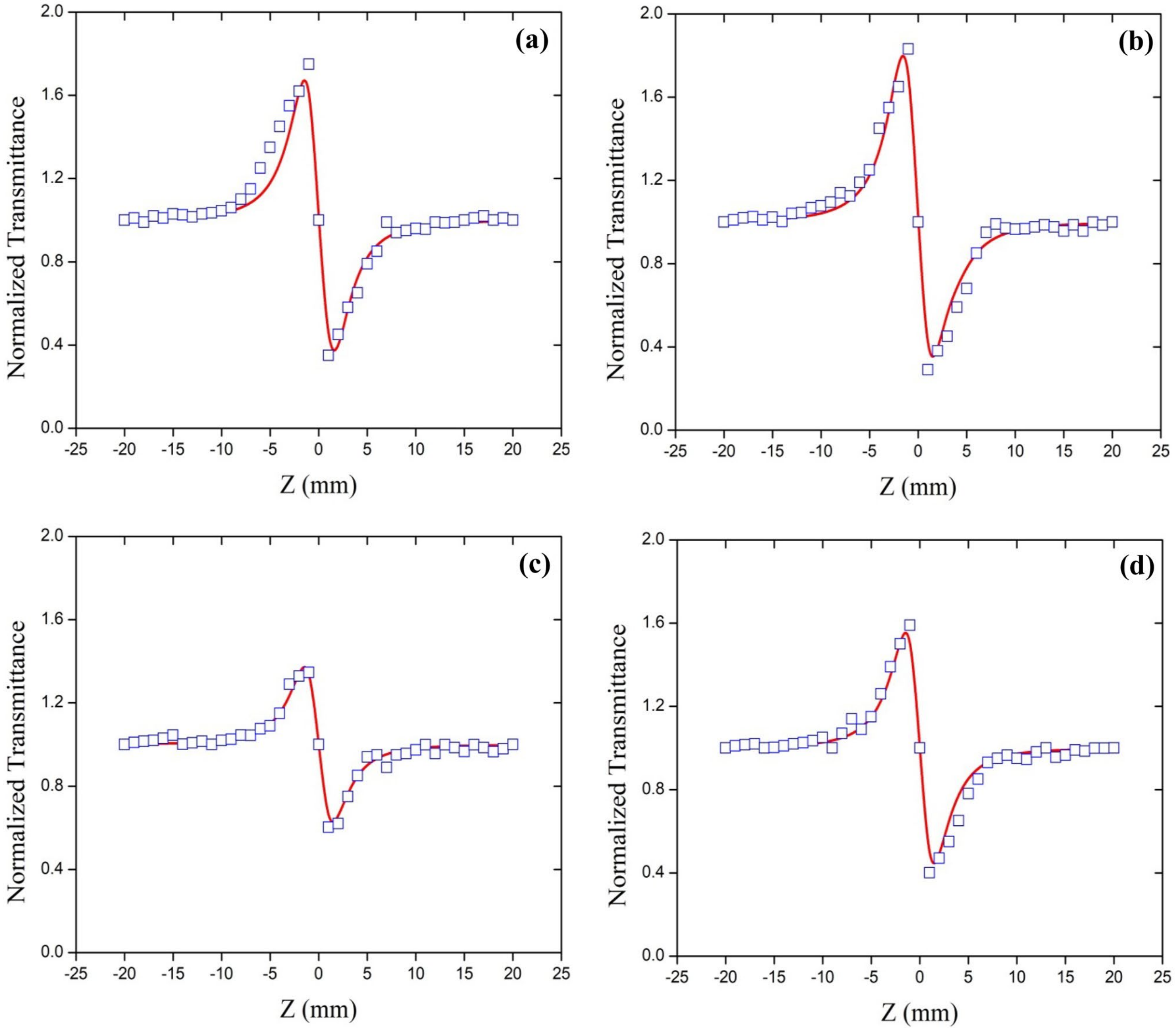

Fig. 5 Pure nonlinear refraction curve of natural pigment in **a** acetone **b** ethanol **c** methanol **d** DMSO

polar solvents is determined from CA Z–scan method. The measurement of nonlinear refractive index includes the contribution of nonlinear absorption (NLA). The pure nonlinear index of refraction is obtained by dividing CA data from the corresponding OA data. The pure nonlinear index of refraction curve of the natural pigment in different polar solvents is shown in Fig. 5a–d. The CA curve exhibits a peak-valley transmittance is the property of self-defocusing or negative nonlinear refractive index. The self-defocusing effect is the result of thermal nonlinearity which arises from continuous absorption of light source at 650 nm wavelength. The transmittance of the sample in different polar solvents is given by,

Table 3 Measured TONLO characteristics of natural pigment in polar solvents

Solvent	n_2 X 10^{-7} (cm^2/W)	β X 10^{-2} (cm/W)	Re (χ^3) X 10^{-6} (esu)	Im (χ^3) X 10^{-6} (esu)	$\chi^{(3)}$ X 10^{-6} (esu)
Methanol	–1.92	0.82	–0.62	0.13	0.63
Acetone	–3.92	–3.35	–1.34	–0.57	1.45
Ethanol	–4.21	–1.30	–1.42	–0.22	1.44
DMSO	–3.49	0.61	–1.39	0.12	1.40

Table 4 TNLO susceptibility of some recently reported natural pigments

S. No	Natural pigments	Technique	χ^3 (esu)	References
1	Aloe Vera	Z–scan	7.93 X 10^{-8}	[12]
2	Chloropyll-a	Z–scan	1.53 X 10^{-7}	[13]
3	β-Carotenoid	Z–scan	6.76 X 10^{-7}	[15]
4	Anthocyanin	Z–scan	5.28 X 10^{-7}	[19]
5	Natural laccaic acid dye	Z–scan	1.85 X 10^{-11}	[22]
6	Bixa Orellana dye	Z–scan	0.72 X 10^{-13}	[23]
7	Lycopene	Z–scan	2.65 X 10^{-7}	[30]

$$T(z) = 1 - \Delta\varnothing_o \frac{4X}{(X^2+1)(X^2+9)} \tag{4}$$

where $X = Z/Z_0$. The nonlinear index of refraction (n_2) is determined from the relation is given by,

$$n_2 = \frac{\Delta\varnothing_0 \lambda}{2\pi I_0 L_{eff}} \left(\frac{m^2}{W}\right) \tag{5}$$

where $\Delta\varnothing_0$, λ and I_0 are on-axis phase shift, wavelength and intensity of the light beam. The real and imaginary components of the TONLO susceptibility is given by,

$$Re\left[\chi^{(3)}\right](esu) = \frac{\varepsilon_0 c^2 n_0^2}{10^4 \pi} n_2 \left(\frac{m^2}{W}\right) \tag{6}$$

$$Im\left[\chi^{(3)}\right](esu) = \frac{\varepsilon_0 c^2 n_0^2 \lambda}{10^2 4\pi^2} \beta \left(\frac{m}{W}\right) \tag{7}$$

where ε_0 is the vacuum permittivity and c is the velocity of light in vacuum. The TONLO susceptibility is calculated by using the relation,

$$\chi^{(3)} = \sqrt{(Re(\chi^3)^2 + (Im(\chi^3)^2}(esu) \tag{8}$$

The measured values of the TONLO parameters of the natural pigment in polar solvents are tabulated in Table 3. The obtained results were compared with some reported natural pigments which is tabulated in Table 4 and value of TNLO susceptibility of the natural pigment extracted from *Ocimum tenuiflorum* is higher than that of reported materials [12, 13, 15, 19, 22, 23, 30].

The solvent environment plays a major role between solute and solvent interaction and it influences the TONLO characteristics of the materials [31]. Solvent parameters such as solvent hydrogen bond donor, solvent hydrogen bond acceptor and polarizability are the major spectral factors that affecting the NLO properties of the natural pigment. The value of nonlinear index of refraction and TONLO susceptibility of the natural pigment increases with respect to solvent hydrogen bond donor, which is shown in Fig. 6a, b. Figure 7a, b shows the solvent hydrogen bond acceptor as a function of nonlinear index of refraction and TONLO susceptibility. From Fig. 7a, b, the nonlinear index of refraction and TONLO

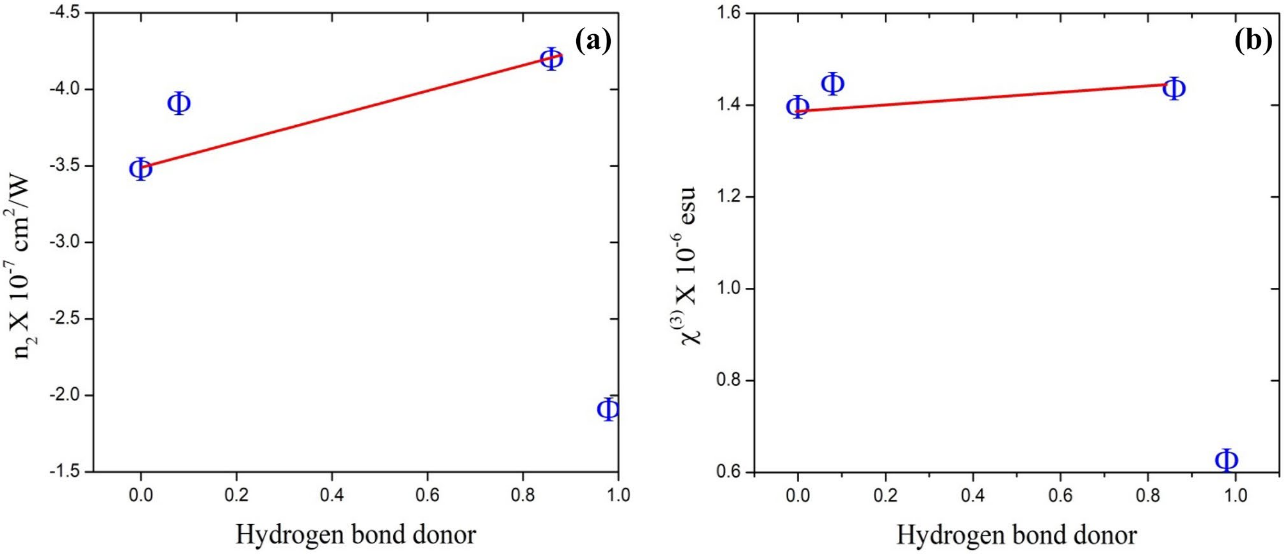

Fig. 6 Hydrogen bond donor as a function of **a** n_2 and **b** $\chi^{(3)}$

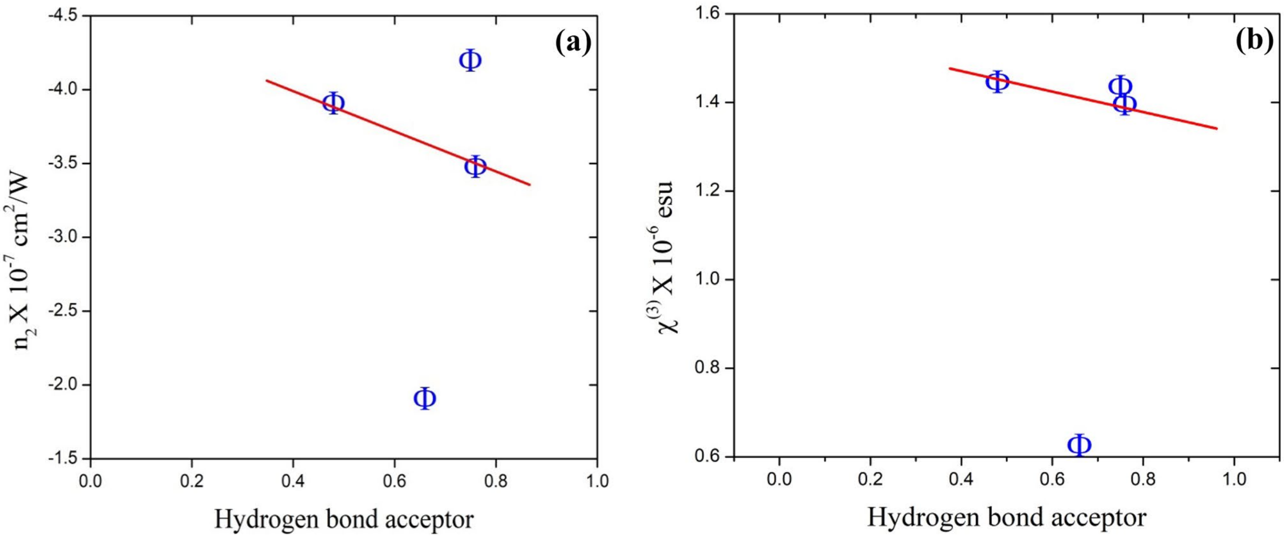

Fig. 7 Hydrogen bond acceptor as a function of **a** n_2 and **b** $\chi^{(3)}$

susceptibility decreases with respect to hydrogen bond acceptor ability. Further, the polarizability is one of the common investigated terms that affects the TONLO characteristics of the natural pigment. In Fig. 4a–d, the nonlinear absorption of the natural pigments switchover from SA to RSA is due to higher values of polarizability of the solvent.

Generally, it is more complicated to explaining the influence of solvent on TONLO characteristics of materials. Different kinds of solute–solvent interactions are involved and solvent polarity parameters alone cannot be used to describe the solvent effects. Therefore for a precise investigation, Kamlet-Abboud-Taft solvent polarity scale which comprises of specific and nonspecific interaction contributions is used. The solvent dependent NLO properties of the sample is derived by using the relation

$$A = A_o + a\alpha + b\beta + s\pi \tag{9}$$

where A is the solvent dependent parameter

A_o is the regression value of the solute molecules.

a,b,s are the regression coefficient.

The results obtained from the above equation are tabulated in Table 5. From Table 5, a good relationship was observed between multi-parameter scale and TONLO features of the natural pigment. Solvent hydrogen bond donor (α), solvent hydrogen bond acceptor (β) and solvent polarizability (π) with various contributions have played an important role in the TONLO properties of the natural pigment. For better comparison, the obtained data was transformed into contribution percentage and it is shown in Table 6. From Table 6, the solvent hydrogen bond acceptor has dominant contribution on TONLO parameters of the natural pigment extracted from the leaves of *Ocimum tenuiflorum.* The solvent hydrogen bond donor and solvent polarizability are also contributed and played a minor role and therefore both specific and nonspecific interactions are involved between solvent and solute molecule.

Table 5 Regression fit to solvent polarity scale

Multi-parameter scale	A_0	a	B	s
n_2	-5.12 X10^{-7}	2.31X10^{-7}	1.20 X10^{-6}	7.67 X10^{-7}
β	-1.60 X10^{-2}	5.10X10^{-2}	1.30 X10^{-1}	9.01 X10^{-2}

Table 6 Percentage contribution for n_2 and β

Multi-parameter scale	P_α (%)	P_β (%)	P_π (%)
n_2	11	54	34
β	19	48	33

Conclusion

The natural pigment was successfully extracted from *Ocimum tenuiflorum* leaves using different polar solvents. The functional group of the extracted pigment was acknowledged by FT-IR technique. The effect of polar solvent on linear and TONLO features of the natural pigment was studied. The nonlinear index of refraction and nonlinear absorption coefficient of the sample was measured to be the order of 10^{-7} cm^2/W and 10^{-2} cm/W. The real and imaginary factors of the TONLO susceptibility were found to be the order of 10^{-6} esu. The multi-parameter scale was used to analyse the solvent characteristics on solute molecules and it is found that the solvent hydrogen bond acceptor has played a dominant factor. The experimental results prove that the extracted natural pigment from *Ocimum tenuiflorum* leaves is a potential material for photonics and optoelectronics applications.

Author Contribution Conceptualization—SJ and DJ; Methodology—SJ and DJ; Validation—DJ; Writing-review and editing—SJ; Supervision—SJ.

Availability of Data and Materials All the data available with the authors.

Declarations

Ethics Approval The submitted work should be original and should not have been published elsewhere in any form or language.

Consent to Participate Yes.

Consent for Publication Yes granted.

Informed Consent Not applicable.

Competing Interests The authors have declared that no competing interests exist.

Research Involving Human Participants and/or Animals Research involving human participants.

Conflicts of Interest The authors declare that they have no conflict of interest.

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