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RESEARCH

Extraction of Natural Pigment *Curcumin* from *Curcuma Longa*: Spectral, DFT, Third-order Nonlinear Optical and Optical Limiting Study

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Abstract
Herein, we report the extraction of natural pigment *curcumin* from *curcuma longa* and their linear and third-order nonlinear optical (NLO) characteristics. The characterization techniques viz., UV–Visible absorption, FT-IR, Micro Raman and Gas Chromatography Mass Spectrum (GC–MS) are used to study the spectral characteristics of *curcumin*. Third-order NLO features of *curcumin* are studied using Z−scan technique with a semiconductor diode laser working at 405 nm wavelength. The natural pigment exhibits negative nonlinear index of refraction resulting from self-defocusing and positive coefficient of absorption is the consequence of reverse saturable absorption (RSA). The order of nonlinear index of refraction (n_2) and nonlinear coefficient of absorption (β) is measured to be 10^{-7} cm^2/W and 10^{-2} cm/W, respectively. Third-order NLO susceptibility ($\chi^{(3)}$) and second-order hyperpolarizability (γ) of *curcumin* is measured to be 2.73×10^{-7} esu and 1.67×10^{-31} esu, respectively. A low optical limiting (OL) threshold of 0.71 mW is observed in the extracted pigment. The experimental results are supplemented by quantum mechanical calculations of the NLO parameters. The overall result finding is that *curcumin* extracted from *curcuma longa* has the potential to be novel optical candidates for photonics and optoelectronics applications.

Keywords *Curcumin* · DFT · Nonlinear optical materials · Spectral characteristics · Z-scan · Optical limiting

Introduction

Nonlinear optical (NLO) materials are extensively used in photonics and optoelectronics applications including optical switching and limiting, optical data storage, optical communication and 3D imaging [1–4]. A lot of research work has been devoted towards finding suitable material for NLO applications. Therefore, materials with significant NLO properties have a key role in the aforementioned applications [5]. Hitherto, different materials are studied and proved to be optically nonlinear under low power regime [6–10]. In the past three decades, chemical dyes have received notable consideration in NLO study [2, 11–14]. The natural dyes made from plant leaves, roots, vegetables, minerals, etc. are the best alternative to synthetic dyes [15]. The natural pigments have received special place in nonlinear optics because of its large susceptibility, chemical stability and significant hyperpolarizability [4]. The important reasons for using natural pigments in NLO studies are: abundance in nature, low cost, non-toxic, synthetic ease, etc. A large number of natural pigments have been involved in NLO study such as chlorophylls, carotenoids, anthocyanin, lycopene, Hibiscus Sabdariffa dye, chinese tea, betanin, and henna [16–25]. The nonlinear absorption (NLA) coefficient of these natural pigments is very attractive and found to be 10–10000 times better than some conventional materials such as CS_2, bismuth borate glasses and sulphur rich compounds [26] etc. *Curcumin* is one among the available natural pigment extracted from turmeric (*curcuma longa*) which is a perennial herb plant belonging to the family of ginger [27]. The turmeric plant

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is widely cultivated in tropical parts of Southeast Asia. Curcuminoids are active compounds of *Curcuma longa*, which consists of *curcumin*, demethoxycurcumin and bis demethoxycurcumin [28]. *Curcumin* is an asymmetric compound named as (E,E)-1,7-bis (4-hydroxy-3-methoxyphenyl)-1–6-heptadiene-3,5-dione, with chemical formula $C_{21}H_{20}O_6$ and molecular weight 368.38 g/mol. *Curcumin* has three important functional groups: aromatic o-methoxy phenolic group, α, β-unsaturated β-diketo moiety, and seven carbon linkers. In recent time, *Curcuma Longa* is used as antioxidant, anti-inflammatory, antimicrobial and anticancer agent [29].

Various experimental techniques are used to quantify the third-order NLO features of the compounds such as degenerate four-wave and three-wave mixing, ellipse rotation, beam distortion, Z−scan technique, etc. [30–33]. Among the available experimental techniques, Z−scan is the most sensitive and simple tool to calculate the third-order NLO characteristics of the materials [16]. This technique has wide advantages including easy experimental procedure, sign and magnitude of the NLO index of refraction and NLO coefficient of absorption is simultaneously measured from closed and open aperture techniques, simple calculation, real and imaginary features of the sample is simultaneously measured from the experiments, etc.

This paper reports the spectral properties of *curcumin* extracted from *curcuma lango*. The density functional theory (DFT) study is used to find the molecular polarizability of *curcumin*. Third-order NLO features of the extracted natural pigments are determined by Z−scan technique with 405 nm wavelength and the OL study is carried out under same experimental condition.

Materials and Methods

Extraction of *Curcumin* from *Curcuma Longa*

The extraction technique applied in the current study is same as that of the reported work by Sahne et al. [34]. The pure turmeric is purchased from local market and dried in oven at 90 °C for 2 days. The dried turmeric is grinded using motor. The *curcumin* powder is highly soluble in ethanol which is purchased from Merck India. Soxhelt experiment [35] is used to extract the natural pigment *curcumin* from turmeric (*curcuma longa*). 20 g of turmeric powder is dissolved with 200 ml of diluted ethanol, which means that 190 ml ethanol mixed with 10 ml of water. The solution is placed in the Soxhelt apparatus and heated upto 80 °C for 8 h. Now, the ethanol is separated from the extract using rotary evaporator under vacuum at 40 °C. The residue is known as *curcumin* and further dried, stored in a refrigerator for future study.

Instrumentation

The FT-IR spectrum of *curcumin* is examined using a SHIMADZU, IRTRACER 100 FT-IR Spectrometer. The Micro Raman study of the sample is studied using HORIBA, LabRam HR evolution operating at 532 nm wavelength with resolution of 0.4 cm^{-1}. The UV–Visible absorption spectrum of *curcumin* is studied by SHIMADZU, UV 3600 plus spectrophotometer. The gas chromatography mass spectrometry (GC–MS) of *curcumin* is studied by SHIMADZU, QP 2010 plus.

Z–scan Technique

The Z–scan experimental method used in the present work is similar to that of our reported work [1]. A semiconductor diode laser with a CW output power of 5 mW operating at a wavelength of 405 nm is used for the studies. The beam intensity at the focus (I_0) and beam waist (ω_0) are measured to be 751 W/cm^2 and 20.58 μm, respectively. A convex lens with 50 mm focal length is placed before the cuvette. A 1 mm thick cuvette is filled with the natural pigment is placed on the micrometer stage and translate from -Z to + Z positions. The closed aperture (CA) and open aperture (OA) techniques are used to measure the n_2 and β of *curcumin*. To measure the beam transmittance, a power meter is positioned far from the source. The condition for thin sample is validated because the measured Rayleigh length is greater than sample length ($Z_R >> L$). In the present study, the measured Rayleigh length is 3.28 mm.

Results and Discussion

UV–Vis Absorption Study

Figure 1 depicts the UV–Visible absorption spectrum of *curcumin* in ethanol as a solvent between 300 and 600 nm. The spectrum covers the whole visible region and the maximum absorbance was observed at 427 nm due to low energy π-π* transition of the extracted natural pigment. The result obtained in UV–Vis spectra holds good agreement with the reported work by Kim et al. [36]. This transition is due to the shift in wavelength at longer wavelength region is called bathochromic shift or red shift. The linear absorption coefficient of the *curcumin* is calculated from the relation is given by,

$$\alpha = 2.303\frac{A}{d} \quad (1)$$

where A and d are the absorbance of the *curcumin* and thickness of the *curcumin* inside a cuvette, respectively.

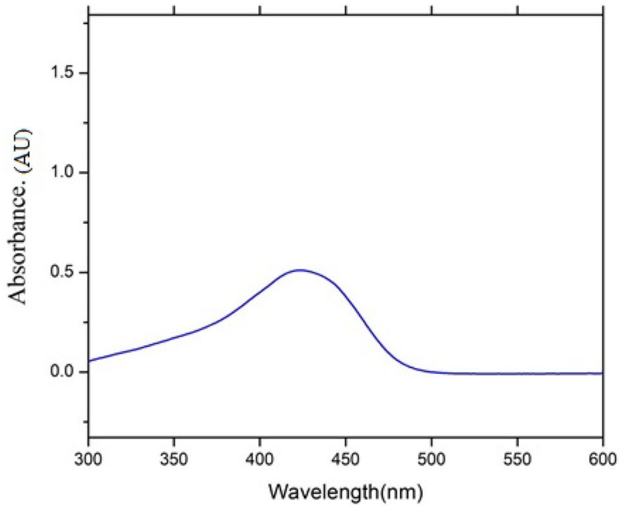


Fig. 1 UV–Visible absorption spectrum of *curcumin*

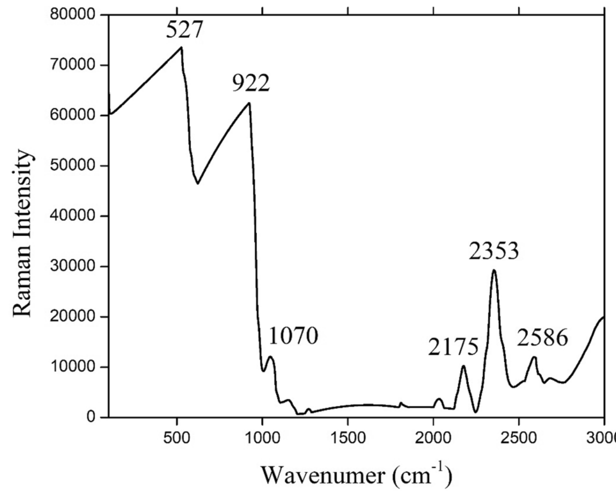


Fig. 3 Micro Raman spectrum of *curcumin*

FT-IR Study

The FT-IR analysis of *curcumin* is shown in Fig. 2. The frequency band at 3278 cm^{-1} is owing to alkyne C-H unit. The stretching band at 2972 cm^{-1} displays the existence of alkane C-H unit. Likewise, the stretching frequency respectively at 2345 and 2117 cm^{-1} seemed for aliphatic C-H stretching vibration. A weak band at 1552 cm^{-1} and 1529 cm^{-1} specifies the existence of C=C groups of aromatic moieties. The O–H bending vibration is observed at 1388 cm^{-1} and the C-O stretching vibration of the extracted pigment has observed at 1041 cm^{-1}.

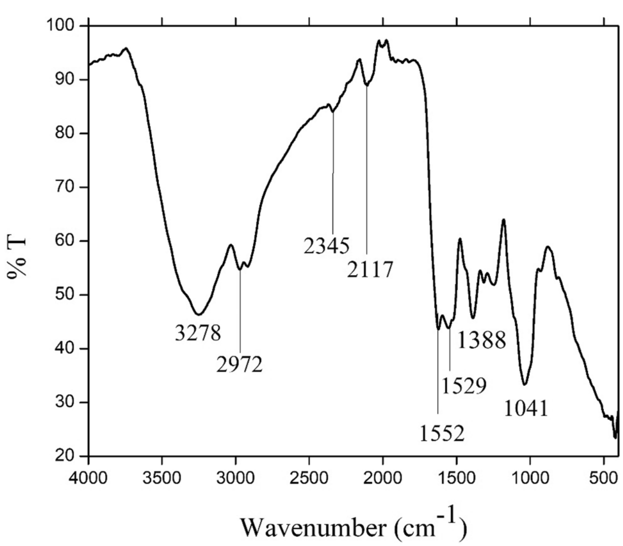


Fig. 2 FT-IR spectrum of *curcumin*

Micro Raman Study

The Micro Raman spectrum of *curcumin* extracted from *curcuma longa* is depicted in Fig. 3. The vibrational assignment at 2586 cm^{-1} is owing to hydroxyl (-OH) unit. The asymmetric stretching C-H unit is observed with corresponding frequency at 2353 cm^{-1}. The weak alkyne C-H unit is observed at 2175 cm^{-1}. The C=O stretching unit is observed at 1070 cm^{-1}. The aromatic rings observed in the sample with corresponding frequency at 922 cm^{-1}.

Gas Chromatography Mass Spectrum (GC–MS) Study

The gas chromatography mass spectrum (GC–MS) of *curcumin* extracted from *curcuma longa* is shown in Fig. 4. From Fig. 4, the major curcumin compounds such as 2-Hydroxy-2-Methyl-4-Pentanone (14.37%), Ar-tumerone (38.27%), Tetradecanoic acid (28.35%), n-Hexadecanoic acid (100%), 9-Octadecenal (21.18%), 4-Butylbenzoic acid, 2-dimethylaminoethyl ester (44.20%) are present in the extracted sample.

Third-Order NLO Study

Third-order NLO properties of *curcumin* obtained from *curcuma longa* is assessed from CA and OA Z−scan techniques. The NLO index of refraction is calculated from CA method, and NLO absorption coefficient is measured from OA method. The intensity dependent NLO absorption coefficient and refraction is given by [33],

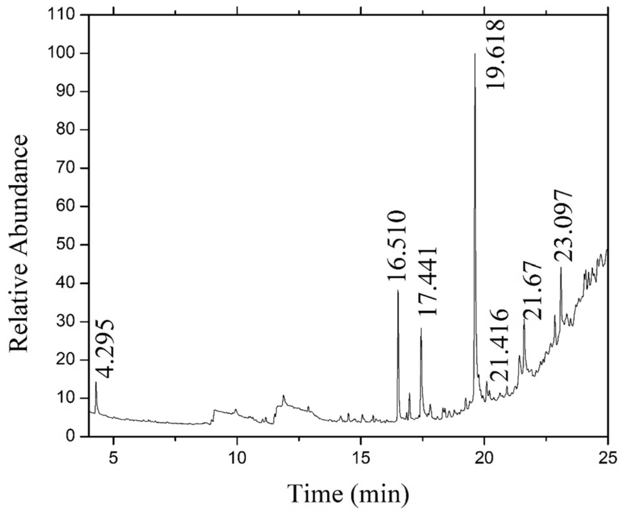


Fig. 4 Gas Chromatography Mass Spectrum of *curcumin*

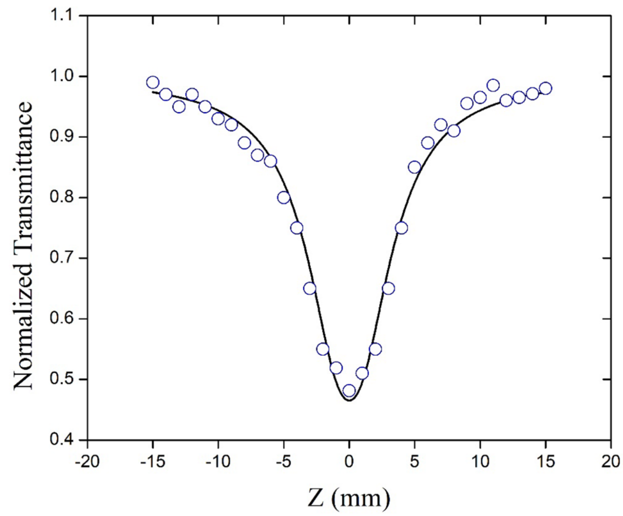


Fig. 5 Open aperture Z−scan curve of *curcumin*

$$\alpha(I) = \frac{\alpha_0}{1 + \frac{I}{I_s}} \tag{2}$$

$$n(I) = n_0 + n_2 I \tag{3}$$

where α_0 is the linear absorption coefficient, I_s is the saturation intensity which depends on the property of the sample and $n_{0,}$ n_2 are the linear and nonlinear refractive index of the material.

Figure 5 shows the outcome of OA Z−scan technique of *curcumin* extracted from *curcuma longa*. The absorption coefficient of *curcumin* shows negative nonlinearity due to RSA. RSA is the nonlinear absorption (NLA) phenomena which results from interaction of light source with *curcumin* at the focal point. A symmetric nature of the curve of Fig. 5 shows intensity reliant on absorption effect. The RSA is caused in *curcumin* may be due to the molecules in higher excited state has more absorption cross-section than the ground state. The five-level model consists of different energy level [5] is also used to explain the RSA property of the sample which is same as that of our reported work [5]. The normalized transmittance is given by [33],

$$T(z, s = 1) = \sum_{m=0}^{\infty} \frac{\left[-q_o(z)\right]^m}{[m+1]^{\frac{3}{2}}}, for \left|q_o(0)\right| < 1 \tag{4}$$

where

$$q_0 = \frac{\beta I_o L_{eff}}{\left(1 + Z^2/Z_0^2\right)} \tag{5}$$

where L_{eff} and Z_o are the effective and diffraction length of the sample. The β of *curcumin* is given by [33],

$$\beta = \frac{2\sqrt{2}\Delta T}{I_0 L_{eff}}\left(\frac{cm}{W}\right) \tag{6}$$

Table 1 Measured third-order NLO parameters of *curcumin*

Parameters	Value
Nonlinear refractive index (n2)	-2.88×10^{-8} cm^2/W
Nonlinear absorption coefficient (β)	2.36×10^{-2} cm/W
Real part of susceptibility [Re $(\chi)^3$]	-0.97×10^{-7} esu
Imaginary part of susceptibility [Re $(\chi)^3$]	2.56×10^{-7} esu
Third-order nonlinear susceptibility $(\chi)^3$	2.73×10^{-7} esu
Second-order hyperpolarizability (γ)	1.67×10^{-31} esu

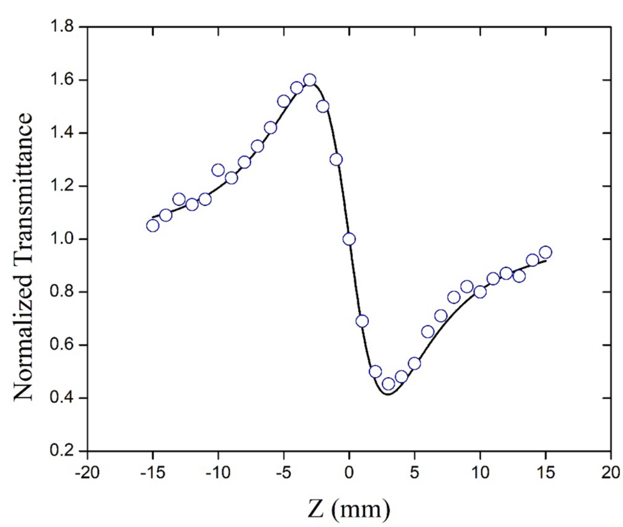


Fig. 6 Pure nonlinear refraction curve of *curcumin*

The sign and magnitude of nonlinear refractive index (NLR) is estimated from CA Z−scan method. In CA, the aperture is used with measurable size and the beam transmittance is determined at far-field using power meter. The NLR is attained from CA technique encompasses both NLR and NLA. Hence, the pure NLR part is determined by dividing CA data to the OA data. Figure 6 shows the pure NLR curve of *curcumin* extracted from *curcuma longa*. The transmittance curve unveils pre focal peak followed by post focal valley transmittance is the result of self-defocusing. Self-defocusing effect is owing to thermal nonlinearity, which arises from continuous absorption of used light source. The difference in temperature inside the natural pigment is owing to the continuous absorption of laser irradiation, which leads to thermal lensing effect. The normalized transmittance of *curcumin* extracted from *curcuma longa* is given by [33],

$$T(z) = 1 - \Delta\varnothing_o \frac{4X}{(X^2+1)(X^2+9)} \tag{7}$$

where $X = Z/Z_0$.

The nonlinear refractive index n_2 of *curcumin* is calculated by using the relation

$$n_2 = \frac{\Delta\varnothing_0 \lambda}{2\pi I_0 L_{eff}} \left(\frac{cm^2}{W}\right) \tag{8}$$

where $\Delta\varnothing_0$ is the on-axis phase shift, λ is the wavelength of the light source and I_0 be the intensity of the light beam at the focus. The measured n_2 value of *curcumin* is tabulated in Table 1. The real and imaginary features of the third-order NLO susceptibility is given by [33],

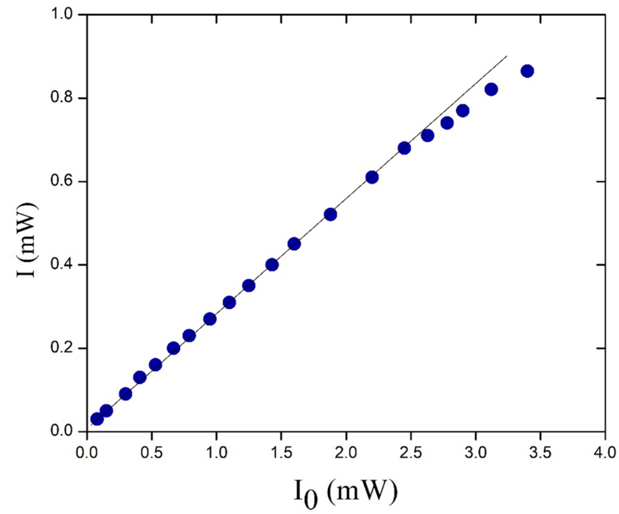
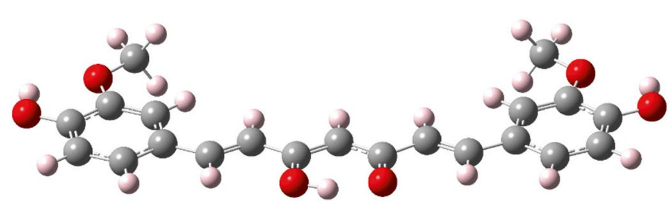


Fig. 7 Optical limiting curve of *curcumin*

$$Re\left[\chi^{(3)}\right](esu) = \frac{\varepsilon_0 c^2 n_0^2}{10^4 \pi} n_2 \left(\frac{cm^2}{W}\right) \tag{9}$$

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

Table 2 Optimised geometry of *curcumin* along with the NLO parameters computed at cam-B3LYP/6–311++G** level of theory; dipole moment μ, mean polarizability α_0, anisotropy of the polarizability $\Delta\alpha_{aniso}$, first order hyperpolarizability $\beta_{\|}$ and second order hyperpolarizability $\gamma_{\|}$



Nature of calculation		μ (Debye)	α_0 ($x\ 10^{-24}$ *esu*)	$\Delta\alpha_{aniso}$ ($x\ 10^{-24}$ *esu*)	$\beta_{\|}$ ($x\ 10^{-30}$ *esu*)	$\gamma_{\|}$ ($x\ 10^{-31}$ *esu*)
Gas Phase	Static; ω = 0	7.5	52.09	63.45	23.3	0.003
	ω = 405 nm		116.72	245.50	392.37	0.133
In ethanol solution	Static; ω = 0	10.05	69.52	82.44	61.53	0.010
	ω = 405 nm		229.96	570.61	3015.21	1.441

where c is the velocity of light in vacuum and ε_0 is the vacuum permittivity. The third-order NLO susceptibility is given by [33],

$$\chi^{(3)} = \sqrt{(Re(\chi^3)^2 + (Im(\chi^3)^2}(esu) \quad (11)$$

The calculated value of $\chi^{(3)}$ of *curcumin* extracted from *curcuma longa* is tabulated in Table 1. It is observed from the Table 1 that, the natural pigment *curcumin* shows large third-order NLO susceptibility over some recently reported natural materials [37, 38]. The second-order hyperpolarizability (γ) of *curcumin* is related to the nonlinear susceptibility by the following relation [39],

$$\gamma = \chi^3/(L^4N) \quad (6)$$

where N is the number density of the molecules in cm^{-3} and L is the local field correction factor given by $[(n^2+2)/3]$, where n is the linear refractive index. The measured second order hyperpolarizability of *curcumin* is tabulated in Table 1.

Optical Limiting Study

Optical limiting materials are widely used for eye and sensor protection from the harmful high intense lasers [40–43]. The low power lasers even power less than 1 mW can also damage the retina of human eye when it is directly exposed [44]. In order to reduce the intensity of the lasers OL are used to safeguard the devices. OL are the materials with low optical limiting threshold can act as perfect optical limiters. In the present work, *curcumin* obtained from *curcuma longa* in ethanol as a solvent is placed at focal point of the lens. The intensity of the beam is varied by a neutral density filter and the beam transmittance at far-filed is measured by photo detector. Figure 7 depicts the optical limiting property of *curcumin*. A low optical limiting threshold of 0.71 mW is observed and concluded that the natural pigment *curcumin* extracted from *curcuma longa* is suitable candidates for applications in optical limiting devices. The observed optical limiting threshold results compared with some reported natural pigments obtained from different plants and vegetables and noticed that the *curcumin* has better optical limiting threshold than reported materials [45–48].

Quantum Mechanical Calculations

The enol-form of *curcumin* which is found to be more stable than its keto form due to the extended conjugation [49] is taken for the Density Functional Theory (DFT) calculations using Gaussian16 [50]. Among the three isomers possible [51] with respect to the relative arrangement of the methoxy group and the enol group, the one with methoxy groups lying trans to the enol group has the highest dipole moment (Table 2) and is therefore employed for computing the NLO properties. The molecule was optimised at CAM-B3LYP/6–311 + + G** level of theory [52] and vibrational analysis is done at the same level of theory to characterize the nature of the stationary point. It is found to be a minimum on the potential energy surface (PES) with the lowest frequency of 9.97 cm^{-1}. Computations under ethanol as a solvent is done using the self-consistent reaction field (SCRF) incorporated into the Polarizable Continuum Model (PCM) [53]. The optimised molecule with symmetry C_s is shown in Table 2 along with NLO associated parameters calculated at the same level of theory.

Solvation energy of *curcumin* in ethanol is found to be -11.96 $kcalmol^{-1}$ and is a minimum on the PES with the lowest frequency of 12.25 cm^{-1}. The optical properties of *curcumin* also see a significant enhancement in ethanol solution with dipole moment increasing from 7.5 Debye to 10.05. Accordingly, the associated polarizability and hyperpolarizabilities are also amplified. The gas-phase and the solvation-modelled calculations are done under both static limit as well as at the single-photon absorption wavelength of 405 nm which is employed in the experimental set-up. Nonlinearity of the optical properties of *curcumin* at 405 nm is prominent as evident from the computed hyperpolarizabilities. Calculated γ-values at $\omega = 405$ nm in the order of 10^{-31} is 0.133 *esu* which increases to 1.441 *esu* in ethanol as a solvent and is very close to the observed experimental value (1.67 *esu*). So far, hyperpolarizabilities were found to be highly sensitive towards the choice of the basis-set [54, 55].

Conclusions

The natural pigment *curcumin* was extracted from *curcuma longa* using Soxhelt experiment and their spectral, third-order NLO features and optical limiting were studied. The functional group present in the extracted pigment was studied by FT-IR and Micro Raman spectrometer. The mass spectrum image confirmed the compounds present in *curcumin*. Third-order NLO characteristics of *curcumin* were examined via Z−scan technique using CW diode laser working at 405 nm wavelength with total power of 5 mW. The natural pigments displayed self-defocusing and RSA based optical nonlinearities. The order of real and imaginary part of $\chi^{(3)}$ was measured to be 10^{-7} esu. The order of third-order NLO susceptibility and second-order hyperpolarizability of *curcumin* extracted from *curcuma longa* was found to be 10^{-7} esu and 10^{-31} esu, respectively. DFT calculations correlate well with the experimental observations and support the choice of ethanol as the solvent to enhance the NLO properties of the compound. A low optical limiting threshold was observed in *curcumin* dye dissolved in ethanol solvent and suggests that the natural pigment is an opted candidate for optical limiting applications.

Author Contribution Conceptualization-S. Deepa, S. Madhu, S. Devasenan, Methodology- S. Jeyaram, M. Maaza, G. Murali Validation- M. Maaza, K. Kaviyarasu, Pattath. D. Pancharatna Writing-review and editing- Pattath. D. Pancharatna, S. Deepa, S. Jeyaram Supervision−S. Jeyaram.

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.

Informed Consent Not applicable.

Consent to Participate Yes.

Consent for Publication Yes granted.

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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