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# LINEAR AND NONLINEAR OPTICAL ANALYSIS OF CADMIUM THIOSEMICARBAZIDE CHLORIDE CRYSTALS FOR OPTICAL LIMITING APPLICATIONS

**J. Martin Sam Gnanaraj[1] and J. Thomas Joseph Prakash*[1]**

[1]*Post Graduate and Research Department of Physics, Government Arts College, (Affiliated to Bharathidasan University), Trichy, Tamilnadu, India.

## ABSTRACT

Semi organic Cadmium Thiosemicarbazide crystals were grown by conventional slow evaporation solution growth method. The linear and nonlinear optical parameters were examined by UV-Visible-NIR spectral analysis and SHG efficiency test respectively. The SHG results suggest that the title material is phase matchable. The third order nonlinear optical studies were evaluated by z-scan test and it was found that the material is self-defocusing nature. The results suggest that the material can be used as an optical limiter for safeguarding human eyes from harmful radiations.

## KEYWORDS

UV, Third order nonlinear optical and Z-scan.

**Author for Correspondence:**

Thomas Joseph Prakash J,
PG and Research Department of Physics,
Government Arts College,
(Affiliated to Bharathidasan University),
Trichy, Tamilnadu, India.

**Email:** armyjpr1@yahoo.co.in

## INTRODUCTION

Optical limiters are the devices that strongly attenuate intensity of optical beams when input laser beam exceeds the threshold, which can protect sensors or human eyes from optical damage[1]. With a wide spread use of the detectors with high sensitivity and fast response, the need has rapidly arisen for devices called optical limiters that protect fine components from intense optical radiation[2]. The extensive use of CW lasers for various applications with power levels ranging from mW to kW has induced a need to protect the human eyes and sensors. In order to find the suitability of a material for nonlinear applications one needs to study its photo physical as well as its optical characteristics such as type of nonlinearity, its magnitude, response time etc[3]. Among the various techniques developed to measure the nonlinear optical refractive index, the single beam Z-scan technique, developed by Sheik Bahae *et al*, is a simple and effective tool[4]. It is a method which can rapidly measure both the nonlinear refraction and the nonlinear absorption in solid and liquid samples. Since the development of laser technology, the research carried out on materials exhibiting strong nonlinear optical properties has been studied intensively because of their potential applications in photonics devices such as optical limiter. A wide range of materials with various nonlinear optical mechanisms contributing for the optical limiting and nonlinear absorption has been investigated. Many publications on optical limiting materials were focused on nonlinear optical responses of organo-metallic compounds, semiconducting materials and most recently, nanoparticles of metals and semiconductors because of their large nonlinearity and ultrafast response time. The optical limiting behavior can be achieved by one or more of the nonlinear optical mechanisms such as excited state absorption reverse saturable absorption, free-carrier absorption, multi-photon absorption, thermal defocusing/scattering, photo refraction, nonlinear refraction and induced scattering[5].

The theory of double-radical model (organic conjugated molecular groups are included in the distorted polyhedron of coordination complex) was brought up in 1987 metal-organic coordination compounds as NLO materials have attracted much more attention for their considerable high NLO coefficients (contrast to inorganic materials), stable physico-chemical properties and better mechanical intension (contrast to organic materials). With the guidance of this theory, many metal–organic coordination materials with good NLO effect have been designed and synthesized[6-13]. The metal-organic coordination complexes can also provide the following advantages: (i) an enhancement of the physico-chemical stability, (ii) the breaking up of the centro-symmetry of the ligand in the crystal, and (iii) an increase in NLO intensity, via metal-ligand bridging interactions. The central metal ion (together with its hybrid electronic orbital) not only offers a certain anisotropic field to keep the NLO active chronopher ligands in a favorable acentric arrangement but also involved in the NLO processes. In this respect thiosemicarbazide and its derivatives are interesting materials for SHG generation. Synthesis of Thiosemicarbazide cadmium chloride monohydrate crystals was already reported by R. Sankar *et al.,*[14] and P. Maadeswaran *et al.,*[15]. J. Thomas Joseph Prakash *et al.,*[16], reported the crystal growth and the structural, optical, thermal and NLO properties of Cadmium Thiosemicarbazide Crystal. In this manuscript, Cadmium Thiosemicarbazide chloride crystals were grown by conventional slow evaporation solution growth method. Cell parameters and Linear optical constants were estimated from singe crystal x-ray diffraction and UV-Visible NIR spectral analysis respectively. Second and Third order NLO properties were analyzed which showed promising results that are discussed in detail.

## Synthesis of CTSC salt

Analytical Reagent grade Thiosemicarbazide (SRL, 99%) and Cadmium Chloride were used as Precursors. The precursors were separately dissolved in deionized water. After 30mins of stirring the contents were mixed together and the temperature of the solution was maintained at 80°C and the solution was agitated vigorously for 8 hours. Multiple phases were formed and precipitation of the compounds were avoided by maintaining the reaction temperature constantly (80°C). The synthesized salt was collected at the bottom of the beaker after 8 hours of vigorous agitation. The obtained CTSC salt was dried at room temperature for one day.

## Crystal Growth

Cadmium Thiosemicarbazide Chloride salt was obtained from the reaction scheme mentioned in 3.0. The synthesized salt is then taken in a clean beaker and saturated solution was prepared using deionized water as a solvent. The temperature was maintained at 40°C throughout the entire reaction.

The solution was stirred well for 5 hours and filtered in a clean beaker. Perforated foil sheet was used to cover the beaker so as to facilitate controlled evaporation of the solvent. Transparent crystals were obtained after a time span of 20 days. A photograph of the as grown crystal is shown in Figure No.1.

## Characterization Analysis

The grown crystals were subjected to various characterization analysis in order to estimate its structure, examine its linear and nonlinear optical properties. ENRAF NONIOUS CAD4 X-Ray diffractometer was used to estimate the cell parameters viz., Length of the unit vectors, Interfacial angles, volume, symmetry and space group of the unit cell. PERKIN ELMER LAMBDA 35 UV Visible spectrophotometer was used to examine the optical properties of the material. Optical constants like band gap, extinction coefficient, reflectance, refractive index, optical conductivity were estimated from the linear optical analysis. The variation of extinction coefficient with as function of photon energy, reflectance with wavelength were studied from the linear optical analysis. Kurtz and Perry experimental setup was used to test the second harmonic generation efficiency of the synthesized material. Single crystals were ground and sieved using Haver EML digital test sieve shaker into different particle sizes using which the phase matching property of the title material was estimated. The nonlinear optical parameters were studied by Z scan technique using He –Ne laser (λ = 632.8 nm) and focused by a lens of 22.5 cm focal length.

## Single Crystal X-Ray diffraction

The structure of CTSC crystals were. From the single crystal XRD results it was concluded that CTSC crystallizes in monoclinic system with the space group Cc. The estimated lattice parameter values are a= 10.108 Å, b= 13.917 Å, c= 6.88 Å, α= β= 90.58°, γ= 125.86° and V= 967.830 $Å^3$. The obtained lattice parameters agree very well with the reported values[17].

## Linear optical analysis

The optical nature of CTSC crystals and the optical parameters such as percentage of transmission, optical band gap are obtained from the UV visible NIR spectral analysis. Transmission spectral analysis is important for any NLO material because a NLO material can be of practical use only if it has wide transparency window. The optical characterization of CTSC crystal was carried with the help of a LAMBDA 35 UV Visible spectrophotometer. The transmission spectrum was traced within the range of 190 to 1100 nm. From Figure No.2 it is evident that the title material is transparent in the entire visible region and it has a wide transparency window. Cadmium thiosemicarbazide chloride crystal possesses the transparency of 45%. From the linear optical analysis it further confirmed that the title material possesses nonlinear optical effect and the optical constants viz., optical band gap, extinction coefficient, refractive index and optical conductivity were estimated from the linear optical analysis.

## Estimation of Optical Constants

Absorption coefficient (α) was calculated from the transmittance value (T) from the following relation[18]:

$$\alpha = \frac{2.3026 \log\left(\frac{1}{T}\right)}{t}$$

Where T is the transmittance and t is the thickness of the crystal. The optical band gap (Eg) was calculated from the relation

$$h\alpha\nu = A(h\nu - Eg)^{1/2}$$

Where A is the constant, Eg is the optical band gap, h is the Planck’s constant and ν is the frequency of incident photons. The optical band gap was estimated by plotting $(\alpha h\nu)^2$ vs hν (Tauc’s plot, Figure No.3). From Figure No.3 the optical band gap of the material was calculated to be 3.8 eV. This confirms that CTSC crystal exhibits good transmittance in the visible region. Since the band gap is 3.8 eV with the lower cut off around 290 nm, CTSC crystal is an eligible candidate in optoelectronic devices fabrications like LED and LASER.

Extinction coefficient (K) can be obtained from the relation

$$K= \frac{\alpha\lambda}{4\pi}$$

The plot of extinction coefficient as a function of photon energy is shown in Figure No.4 and that of wavelength vs extinction coefficient is shown in Figure No.5. An inverse dependence with E is observed in low energy value and a linear variation is observed with energy with addition.

The reflectance (R) and refractive index (n) can be derived from the relations:

$$R = \frac{1 \pm \sqrt{(1-\exp(-\alpha t)+\exp(\alpha t))}}{(1+\exp(-\alpha t))}$$

$$n= \frac{-(R+1) \pm \sqrt{(-3R^2+10R-3))}}{2(R-1)}$$

The reflectance was also plotted as a function of wavelength and shown in Figure No.6. The refractive index was calculated from the plot of wavelength vs refractive index (Figure No.7) and it was observed to be 1.4.

The optical conductivity is one of the powerful tools for studying the electronic states in materials. The frequency dependence of dielectric reflects the fact that a material's polarization does not respond instantaneously to an applied field. Optical conductivity is given by the relation

$\sigma = \alpha nc/4\pi$

Where $\alpha$ is the absorption coefficient, n is the refractive index, c is the velocity of light. The variation of optical conductivity with photon energy is depicted in Figure No.8. From the plot it is confirmed that the optical conductance increases with the increase of photon energy.

## Nonlinear Optical analysis

The Second Harmonic Generation efficiency of the CTSC crystals were tested by Kurtz and Perry experimental setup[19]. The grown single crystal of CTSC was powdered with a uniform particle size and then packed in a micro capillary of uniform bore and was illuminated using Spectra Physics Quanta Ray DHS2. Nd: YAG laser using the first harmonics output of 1064 nm with pulse width of 8 ns and repetition rate 10 Hz. The second harmonics signal, generated in the crystal was confirmed from the emission of green radiation by the crystal. A finely powdered sample of potassium dihydrogen orthophosphate was used as a reference material in the present measurement. The SHG radiation of 532nm green light was collected by a photomultiplier tube (PMT-Philips Photonics-Triax-550) to collect only the 532nm radiation. The optical signal incident on the PMT was converted into voltage output at the CRO (Tektronix-TDS 3052B). The input laser energy incident on the powdered sample was chosen to be 0.68 J. Powder SHG efficiency obtained for CTSC monohydrate is about 1.9 times that of potassium dihydrogen orthophosphate crystal.

## Phase Matching Studies

SHG efficiency measurement is an efficient technique to identify the non-centro symmetric behavior of crystal structures. In addition to this, it is also used as a screening technique to identify the phase matching capability of materials. The linear increase of SHG efficiency with respect to the increase in particle size confirms the phase matching behavior of the material. Only those materials exhibiting phase matching behavior are grown into single crystals of large size for NLO applications.

The particle size dependency of SHG intensity was studied in order to confirm the existence of phase matching property. The SHG efficiency is strongly dependent on the particle size. CTSC single crystal was ground and sieved using Haver EML digital test sieve shaker into different particle sizes. To make pertinent comparisons, with the known SHG materials, KDP crystal was ground and sieved into ranges of same particles size. The particle size dependency of SHG intensity in CTSC is shown in Figure No.9. The SHG intensity increases linearly with the increase in particle size till 355µm and above this range, the intensity gets deviated from the linearity and starts attaining saturation. This property of particle size dependency of SHG intensity is observed in phase matchable crystals[20]. Hence from the obtained results, CTSC crystal is an efficient candidate in frequency doubling applications and as a parametric oscillator.

## Z -scan measurement

The Z-scan technique is a simple, highly sensitive and a very accurate method for the determination of both nonlinear refractive index ($n_2$) and nonlinear absorption coefficient (β). The maximum advantage of this method is that we can measure both the magnitude and sign of the nonlinear refractive index and the nonlinear absorption coefficient of the samples simultaneously. The nonlinear refractive index is proportional to the real part of the third-order susceptibility [Re $\chi^{(3)}$] and the nonlinear absorption coefficient is proportional to imaginary part [Im $\chi^{(3)}$]. The sample is moved across the focal region (-z to +z) along the axial direction, which is the direction of propagation of the laser beam transmitted though the sample was collected by a photo detector through an aperture and intensity was measured by a digital power meter attached to the detector.

The measurements are performed in two different modes i.e. (i) open aperture and (ii) closed aperture. The measurement in open aperture mode gives us information about the nonlinear absorption coefficient and the closed aperture mode helps us to calculate the third order nonlinear refractive index.

In the closed aperture Z scan, the transmittance of the sample through the aperture is monitored in the far field as a function of the Z –position. He –Ne laser ($\lambda$ = 632.8 nm) was used as the light source and focused by a lens of 22.5 cm focal length. A polished crystal of1mm thickness was used for the measurement. The experiment was done by placing the sample in the beam at different positions with respect to the focus and measuring the corresponding light transmission. A spatial distribution of the temperature in the crystal surface is produced due to the localized absorption of a tightly focused beam propagating through the absorbing sample. Hence a spatial variation of the refractive index is produced which acts as a thermal lens resulting in the phase distortion of the propagating beam. Figure No.10 (a) and 10 (b) shows the closed and open aperture Z scan curve for CTSC crystals respectively. A peak followed by the valley pattern observed reveals the negative sign of the nonlinear refractive index of the complex. The estimation of nonlinear refractive index ($n_2$) of the crystal calculated using the standard relations.

$n_2 = \Delta\varphi_0 / KI_0L_{eff}$

Where K is the wave number ($K=2\pi / \lambda$), $I_0$ is the intensity of laser beam at focus Z= 0.

$L_{eff} = [1-\exp(\alpha L)]/\alpha$

Where, $\alpha$ is the linear absorption coefficient measured from UV-Vis Spectrophotometer

L is the thickness of the sample.

$\Delta T$ is the one-peak value at the open aperture Z-scan curve and is written in terms of theon axis phase shift at the focus[21] as

$\Delta T= 0.406 (1-S)^{0.25} |\Delta\varphi_0|$

Where S is the aperture linear transmittance and is calculated using the relation

$S= (1-\exp(-2r_a^2 \omega_a^2))$

Where $r_a$ is the aperture radius

$\omega_a$is the spot size diameter in front of the aperture

The nonlinear absorption coefficient (β) was calculated using the relation

$\beta = 2\sqrt{2}\, \Delta T/ I_0L_{eff}$

Where, $\Delta T$ is the one-peak value at the open aperture Z-scan curve.

From the results, the negative refractive index shows the self-defocusing effect of the grown crystals. This may be an added advantage for the application in production of optical sensors such as night vision devices. The negative β value indicates that the complex shows the saturable absorption[22].

The real and imaginary part of the third order non-linear optical susceptibility $\chi^{(3)}$ are defined as

$\mathrm{Re}(\chi^3) = 10^{-4}(\varepsilon_0 C^2 n_0^2 n_2)/ \pi$

$\mathrm{Im}(\chi^3)= 10^{-2} (\varepsilon_0 C^2 n_0^2\lambda\beta)/4\pi^2$

Where $\varepsilon_0$ is the permittivity in vacuum

Cis the velocity of light

$n_0$ is the linear refractive index

Thus the third order nonlinear susceptibility is given by,

$(\chi^3)= \sqrt{}\,(\mathrm{Re}(\chi^3)^2+ \mathrm{Im}(\chi^3))$

The nonlinear parameters such as nonlinear refractive index ($n_2$) and nonlinear absorption coefficient (β) and nonlinear optical susceptibility $\chi^{(3)}$ have been evaluated and tabulated in Table No.1

## Optical Limiting Applications

The ability to protect the optical sensors and devices by controlling the intensity of light can be achieved by optical limiters. The optical limiting behavior indicates the output intensity to vary linearly with small increase in input and for high input intensity the output intensity deviates from the linearity which could be explained by inverse square law.

The optical limiting experiment of the samples was carried out by placing the sample beyond the focal point and the obtained characteristic curves are shown in Figure No.11. The transmitted output intensity was found to vary linearly with the incident input intensities at very low input intensities but starts to deviate at high incident intensities. With further increment of the input power, the transmitted intensity reaches a plateau and is saturated at a point defined as the limiting amplitude: i.e. the maximum output intensity, showing obvious limiting property.

Thus at low incident powers, the output varies according to Beers law and beyond 18 mW, it becomes nonlinear. Numerically the response time of optical limiting action for the samples can be estimated using the relation[23],

$\tau = R^2/\alpha_d$

Where R (=15μm) is the beam radius of the tightly collimated laser beam and $\alpha_d$ is the thermal diffusivity of the sample. The response time is approximately in the order of ms in the case of the metal complexes. This shows that the heat load equilibrates in a characteristic time of milliseconds and does not change much matrix composition. Hence it is inferred that CTSC crystals can be used as an optical limiter in thermo optic sensors.

**Table No.1: Third order NLO parameters of CTSC crystals**

| S.No | Parameters | Values |
|---|---|---|
| 1 | Nonlinear refractive index ($n_2$) x $10^{-8}$ $cm^2$/ W | -3.22 |
| 2 | Nonlinear absorption coefficient (β) x $10^{-3}$ cm / W | -4.68 |
| 3 | Real part of the third order non-linear optical susceptibility [Re $\chi^{(3)}$] x $10^{-6}$esu | 2.10 |
| 4 | Imaginary part of the third order non-linear optical susceptibility [Im $\chi^{(3)}$] x $10^{-6}$esu | 1.68 |
| 5 | Third-order nonlinear optical susceptibility [$\chi^{(3)}$] x $10^{-6}$esu | 3.5 |

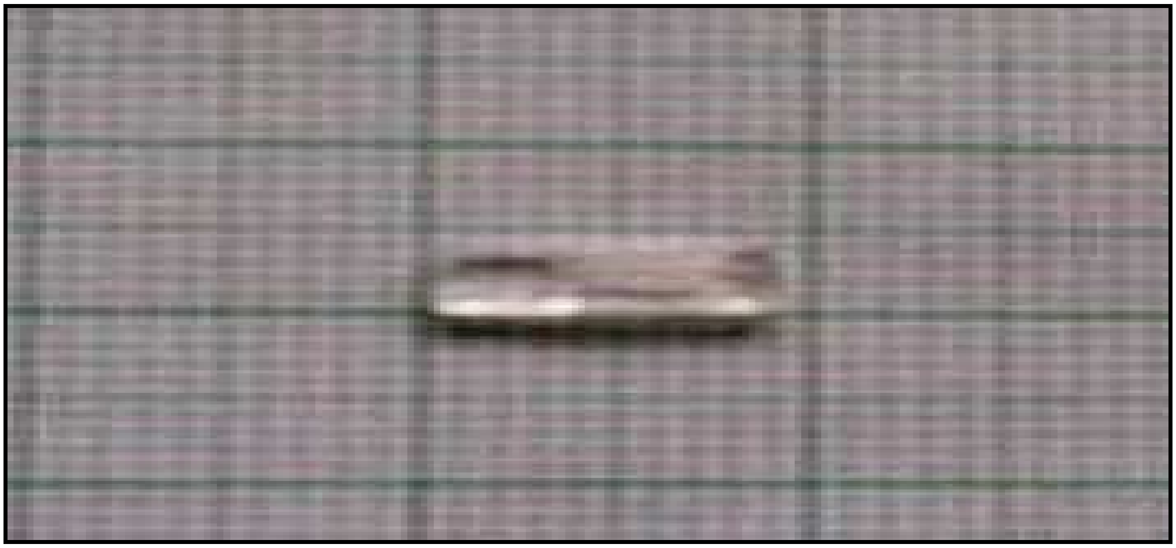

Figure No.1: As Grown CTSC crystal

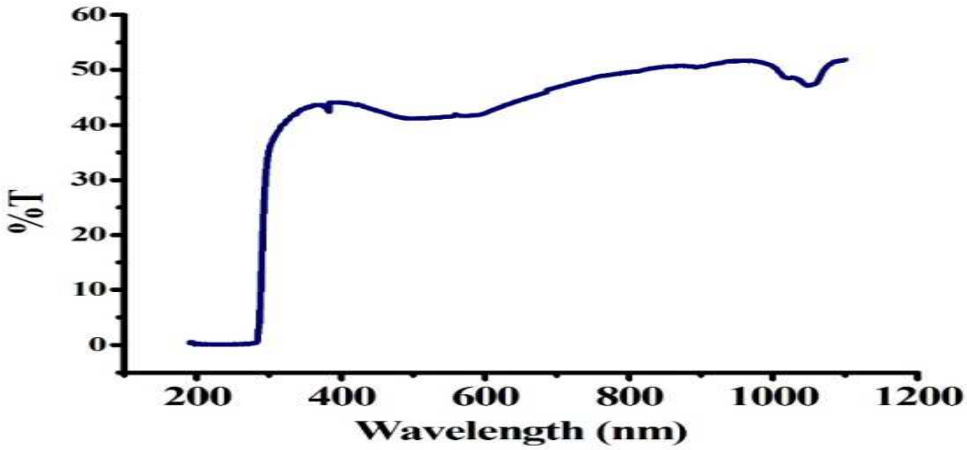

**Figure No.2: UV Transmission Curve of CTSC crystals**

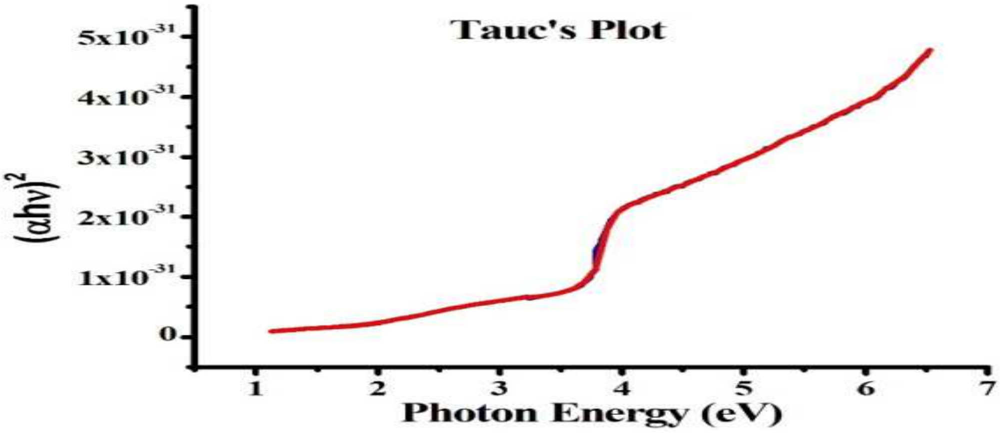

**Figure No.3: Tauc's plot of CTSC crystals**

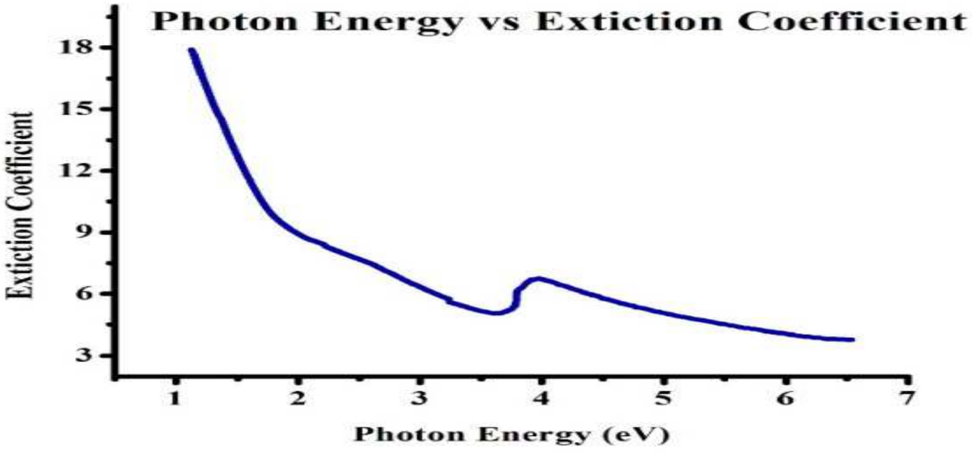

**Figure No.4: Photon energy vs Extinction Coefficient of CTSC crystals**

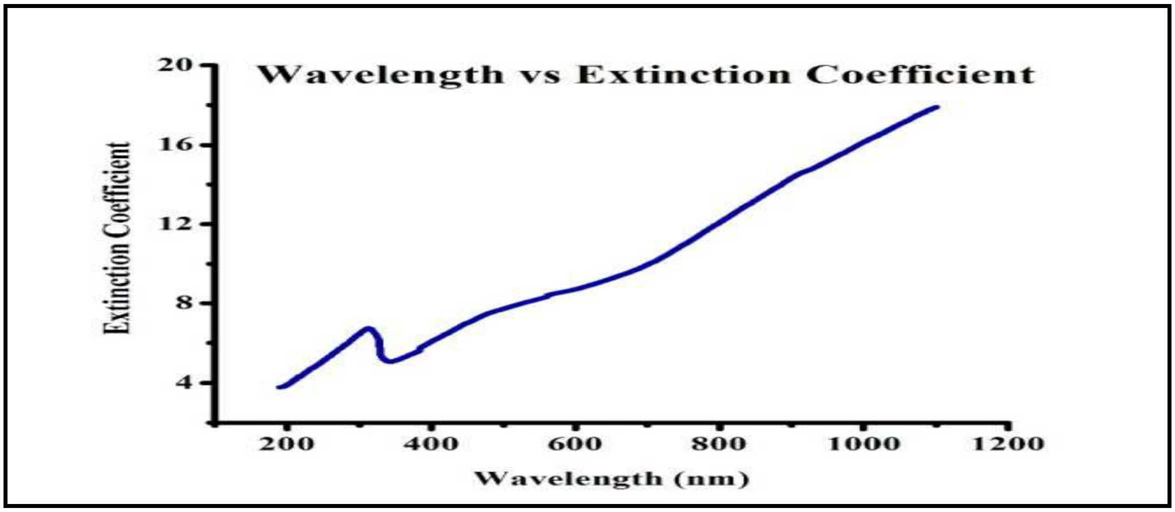

**Figure No.5: Wavelength vs Extinction Coefficient of CTSC crystals**

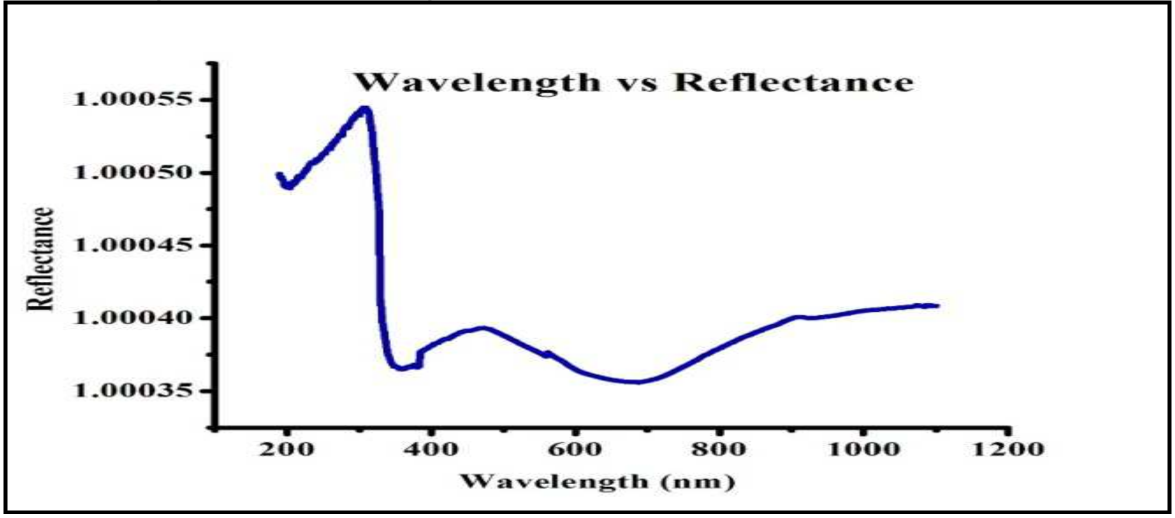

**Figure No.6: Wavelength vs Reflectance of CTSC crystals**

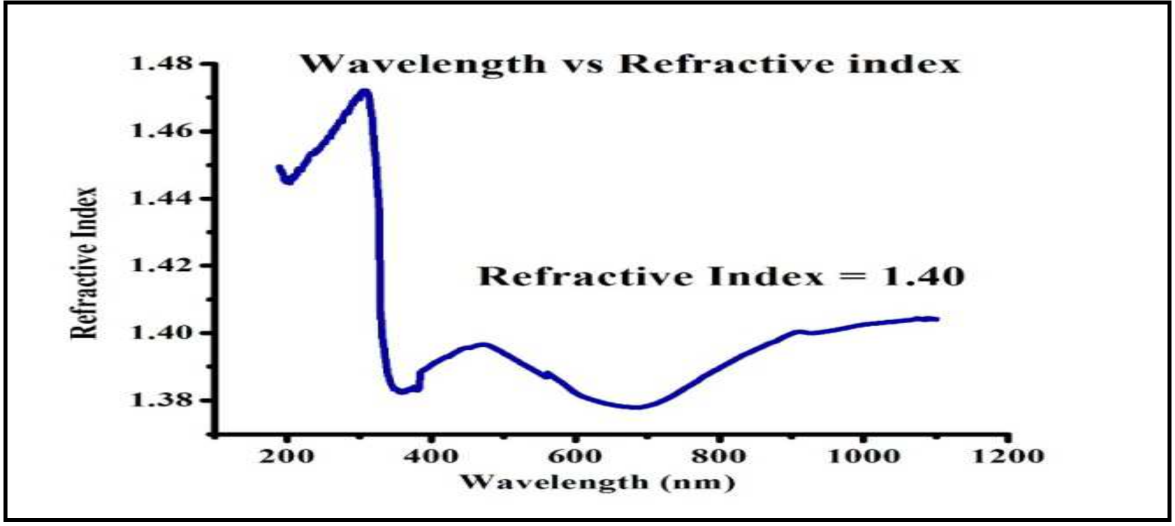

**Figure No.7: Refractive index of CTSC crystals**

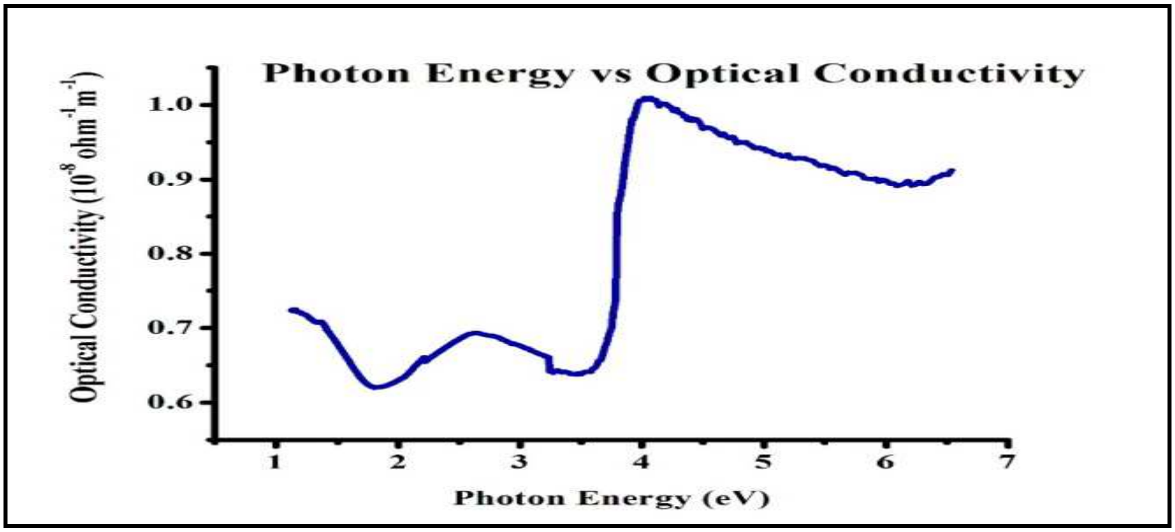

**Figure No.8: Photon energy vs Optical Conductivity of CTSC crystals**

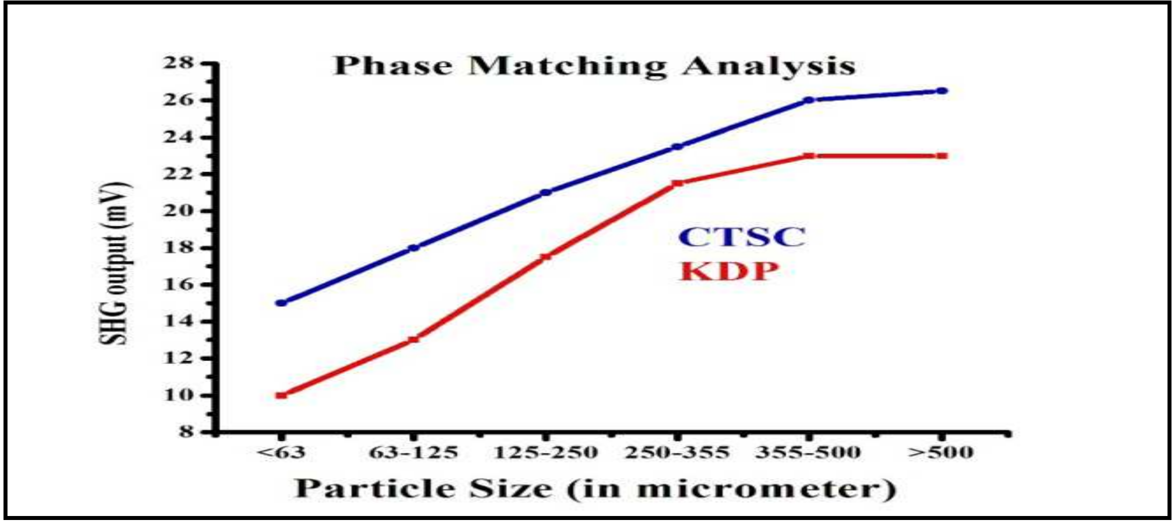

**Figure No.9: Particle size dependency of CTSC crystals**

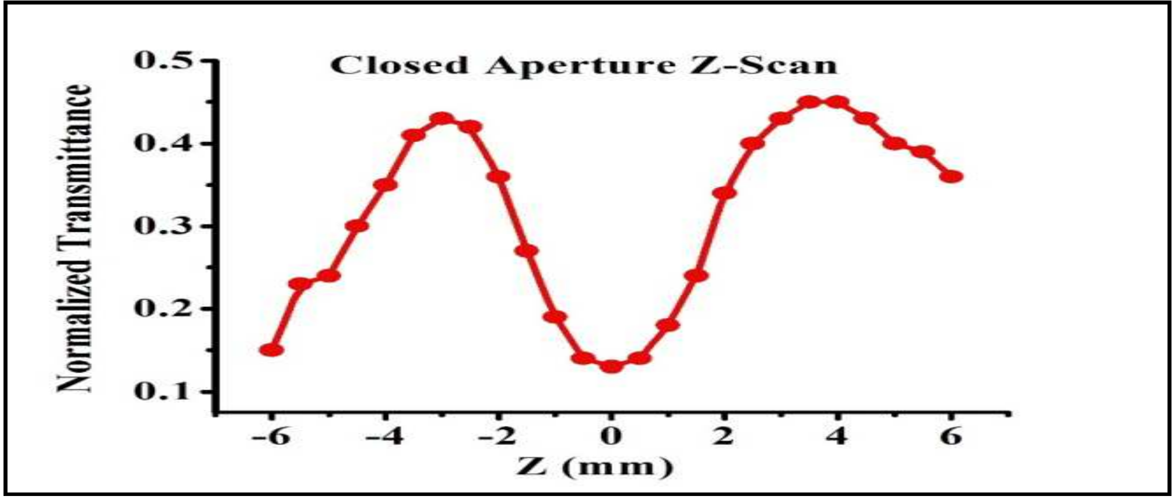

**Figure No.10: (a) Closed Aperture Z-scan of CTSC crystals**

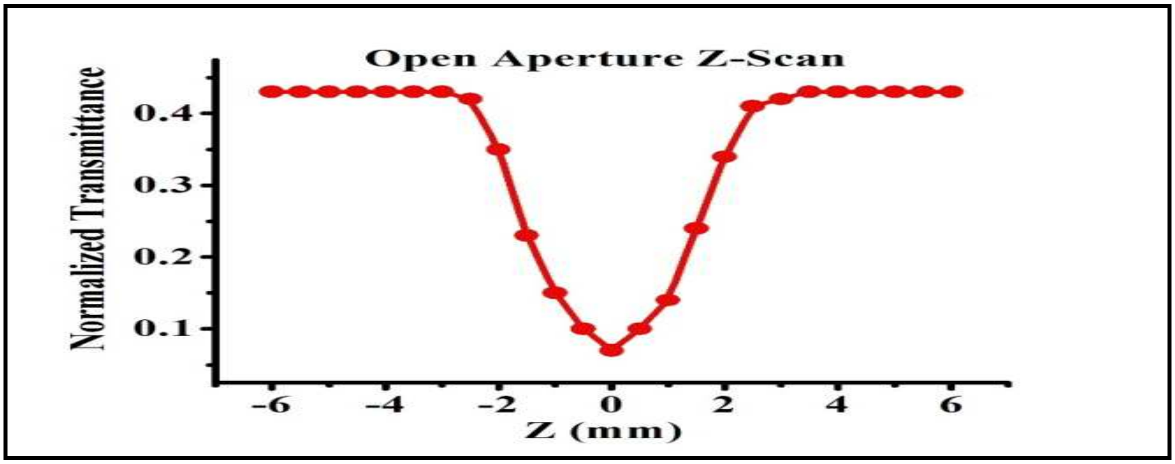

**Figure No.10: (b) Open Aperture Z-scan of CTSC crystals**

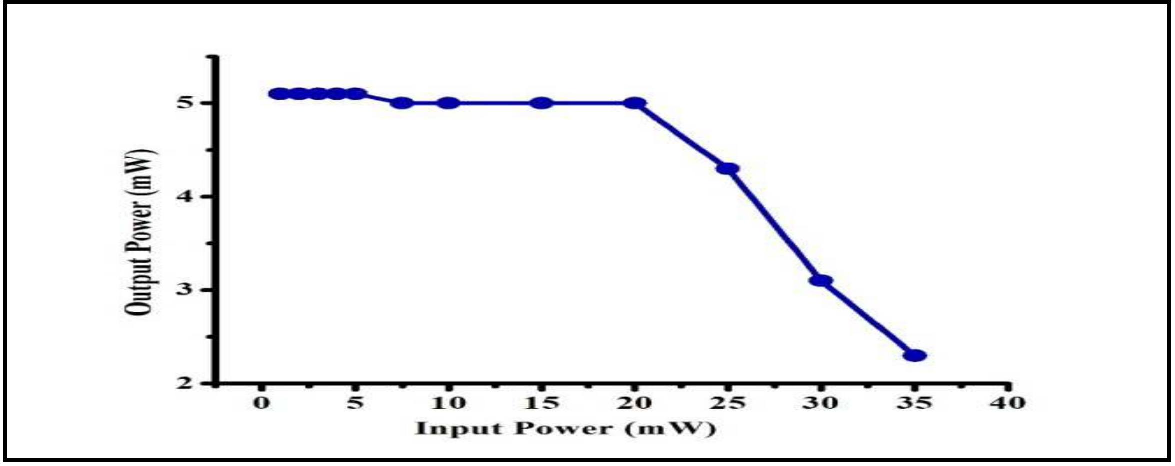

**Figure No.11: Optical Limiting behavior of CTSC crystals**

## CONCLUSION

CTSC crystals were grown by cost effective slow evaporation solution growth technique. Cell parameters were estimated by single crystal x-ray diffraction analysis. It was observed that the material crystallizes in monoclinic system with space group Cc. Linear optical analysis was carried out on the grown crystal and the optical constants were estimated. The refractive index of the title material was estimated and it was found to be 1.40. Particle size dependency of CTSB crystals was analyzed and the results show that CTSC crystals are phase matchable. Third order NLO susceptibility and optical limiting behavior was studied by Z-Scan technique. The results show that CTSB crystals are potential candidates in optical limiting applications.

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## CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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