# INVESTIGATION ON THE GROWTH, SPECTRAL, OPTICAL, AND LASER DAMAGE THRESHOLD STUDIES OF L -TARTARIC ACID SINGLE CRYSTAL

**S.R. Meeraa [1], S. Hemavathy [2], N. Mahalakshmi [3], M. Parthasarathy [1*]**

[1,2,3.] *Department of Physics, School of Basic Sciences, Vels Institute of Science, Technology and Advanced Studies (VISTAS), Pallavaram, Chennai-600 117, Tamil Nādu, India.*

***Corresponding author: Dr M. Parthasarathy**

mps2k7@gmail.com

## ABSTRACT

Good quality L-tartaric acid (LTA) single crystal of size 10mm × 3mm × 2 mm has been grown by slow solvent evaporation technique. Single crystal X-ray diffraction analysis reveals that the crystal belongs to the triclinic crystal system with space group $P\bar{1}$. Using FT-IR, spectral analyses were identified. UV–Visible optical analysis has been performed to, determine the optical transparency of LTA crystal in the range of 200–1200 nm, and the optical band gap was calculated using Tauc's plot, and the value was found to be 5.1eV. Laser damage threshold studies were carried out to the grown crystals. A powdered sample of LTA was tested by second harmonic generation.

**keywords**: *Single crystal X-ray diffraction, Slow Evaporation, Optical studies, FT-IR, Laser damage threshold .*

## 1. INTRODUCTION

In contemporary times the interest in research about organic nonlinear single crystals is increasing because of its wonderful performance in storage, high-density and speed data processing and telecommunications [1,2]. Organic materials have attracted much attention because the NLO (non-linear optical) responses in this broad class of materials is microscopic in origin, offering an opportunity to use theoretical modelling coupled with extremely synthetic flexibility to design and breed new materials [3,4]. Researchers have preferred to focus on semi-organic compounds due to their large nonlinearity, high resistance to laser damage threshold, low angular sensitivity, and good mechanical hardness [5]. L-Tartaric acid (2,3-dihydroxybutanedioic acid) is a naturally occurring dicarboxylic acid containing two stereo enters. It exists as a pair of enantiomers and an achiral meso compound. It is present in many fruits (fruit acid), and its monopotassium salt is found as a deposit during the fermentation of grape juice. Pure laevorotatory (S,S)-d-(−)-tartaric acid is rare. The highly functionalized and C2-symmetric tartaric acid molecule is perfectly tailored for applications as a resolving agent and chiral ligand. In the current investigation the novelty has been achieved by focusing on the growth of LTA crystal by employing it to single X-ray diffraction, FT-IR analysis, UV–visible, SHG efficiency and Laser damage threshold characterization techniques to explore LTA as potential crystal for NLO facilitated photonic devices.

## 2.MATERIALS AND METHODS

The commercially available L-Tartaric acid (SRL extra pure 99%) was used for growth process. The single crystals of L-Tartaric acid were successfully grown from slow evaporation solution growth technique using water as solvent at room temperature. The solution was well stirred for about 6hrs to attain homogeneity. The solution was carefully filtered using Whatman filter paper and kept in an undisturbed position for nucleation. Optically good quality crystals of L-Tartaric acid with dimensions 10mm×3mm×2mm were harvested from the mother solution after a time span of 25 days. The as grown single crystal of L-Tartaric acid is shown in Fig. 1.

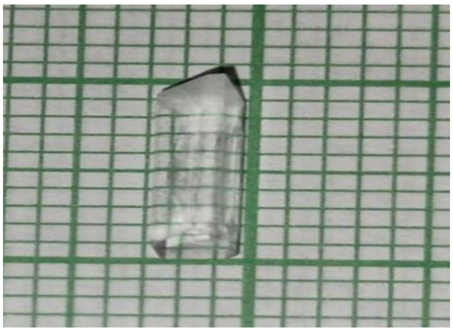

**Fig. 1 As-grown L-Tartaric acid single crystal**

## 3.RESULTS AND DISCUSSIONS

### 3.1 Single Crystal X Ray Diffraction

X-ray diffraction is based on constructive interference of monochromatic x-rays and a crystalline sample. These X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample. The interaction of incident rays with the sample produces constructive interference when conditions satisfy Bragg’s law. The diffracted patterns were recorded using a BRUKER X-ray diffractometer with Cu Kα radiation (λ=1.5418 Å). From the single crystal X-ray diffraction studies, it is confirmed that L-Tartaric acid belongs to the triclinic system with a space group $P\bar{1}$. The obtained lattice parameters are tabulated in Table 1 and it is well matched with the reported literature [6].

**Table 1. Lattice parameters of L-tartaric acid single crystal**

| Lattice parameters | Reported literature Okaya. et al (1966) | Present work |
|---|---|---|
| a | 6.580Å | 6.56Å |
| b | 9.186Å | 9.20Å |
| c | 4.896Å | 4.90Å |
| Crystal system with space group | Triclinic, $P\bar{1}$ | Triclinic, $P\bar{1}$ |

**3.2 FTIR Spectral analysis**

Fourier-Transform Infrared spectroscopy gives information about thefunctional groups present in the title material. The spectrum range of infrared assignments was noted from 500 to 4000 cm$^{-1}$ using Bruker AXS FT-IR with the KBr pellet technique. Each peak in the spectrum gives absorption frequencies which are listed in Table 2. The recorded FT-IR spectrum is presented in Fig. 2. One can clearly notice that the figure that a wide peak appeared at 3445 cm$^{-1}$ indicating the occurrence of O-H bond stretching of carboxyl group. The intense peak at 1706 cm$^{-1}$ is obtained due to carbonyl C=O group stretching. A peak at 1450 cm$^{-1}$ can be attributed to the mixture of O-H deformation and of C-O bond stretching. C-C Stretching of L-Tartaric acid was observed at 1056 cm$^{-1}$. Table 2 shows the absorption frequencies and their tentative vibrational assignment of L-Tartaric acid.

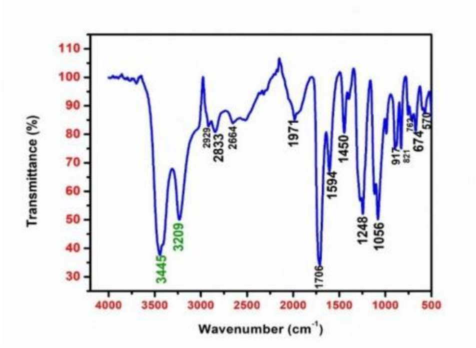


**Figure 2 FTIR spectrum of LTA single crystal**

**Table 2 FTIR spectrum Assignments of LTA single crystal**

| **Wavenumber ($cm^{-1}$)** | **Vibration** |
|---|---|
| 3445 | O-H Stretching |
| 1706 | C=O Stretching |
| 1450 | COO- Symmetric stretching |
| 1248 | C-C Stretching |
| 1056 | C-C Stretching |
| 917 | C-C Stretching |
| 762 | C-C Stretching |
| 674 | COO-Scissoring |
| 570 | COO-Wagging |

## 3.3 Optical Studies

The determination of exact optical nature of crystal helps to identify its extended credibility for optoelectronics [7] and NLO device applications [8]. The optical transmission window of LTA single crystals was investigated using UV–Vis spectroscopy employing CARY 5000 UV spectrophotometer with a wavelength ranging from 200–1200 nm. For the analysis, we used a thin and visibly transparent LTA single crystal of thickness 1mm.

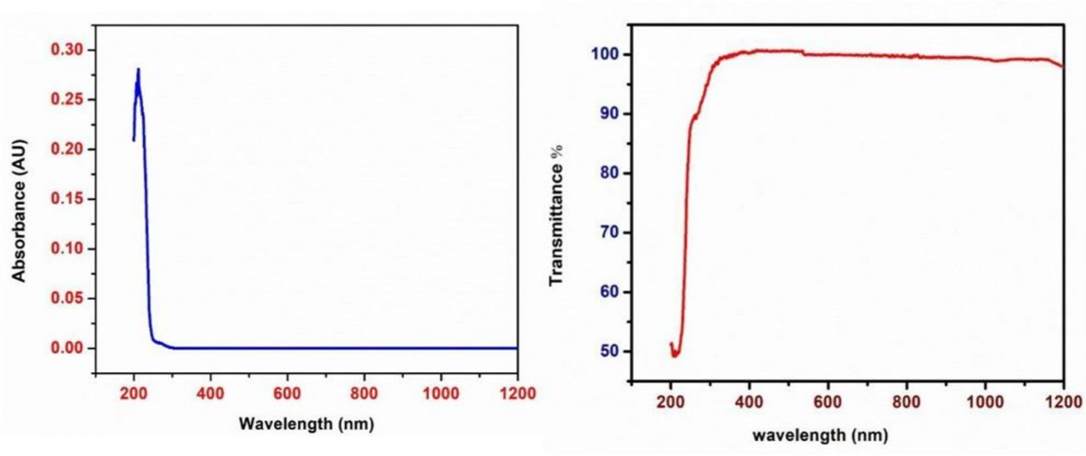


**Fig. 3(a) UV-Visible spectrum of the LTA single crystal**

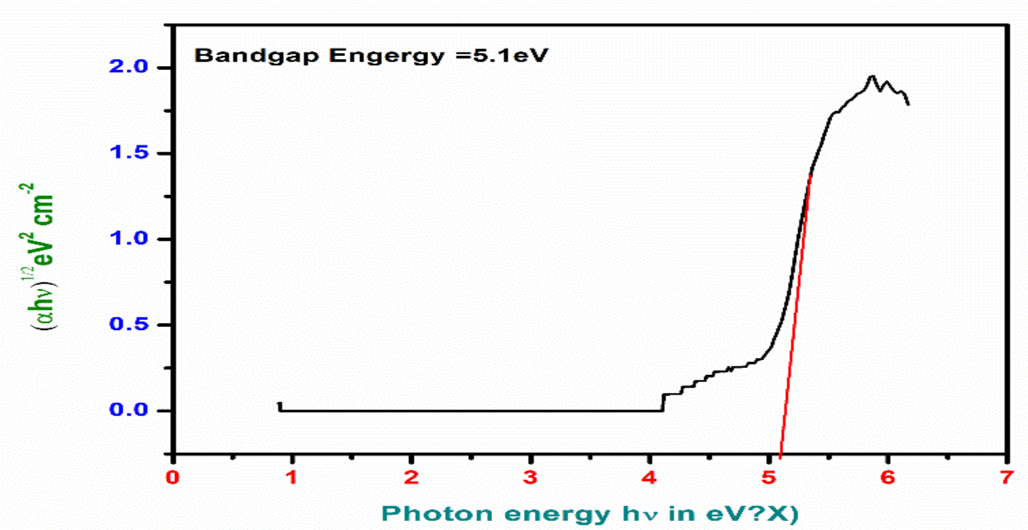


**Fig. 3(b) Tauc's plot**

The spectrum reveals the lower cut of wavelength to be about 231 nm with ~95% transmittance in the optical window, which makes this material useful for non-linear optical applications. The relationship between optical absorption coefficient and incident photon energy provides information regarding the band structure and nature of optical transitions of electrons in materials [9]. The optical absorption coefficient (α) for materials is related to the transmittance as given by equation: where 'α' is the absorption coefficient, 't' is thickness of the sample, 'T' is transmittance (%). The optical bandgap of the material was calculated by using Tauc's plot as shown n Fig. 3(b), using the Eq.1

$$(\alpha h\nu)^n = T(h\nu - E_g) \quad (1)$$

Fig. 3(a) depicts the UV–Visible spectrum for LTA crystal, where 'α' is the optical absorption coefficient, 'h' is planks constant, 'ν' is the frequency of the incident photons, '$E_g$' is the optical bandgap, and 'n' is 1/2 for allowed direct transitions. By extrapolating the Tauc's plot between $(\alpha h\nu)^{1/2}$ verses hν the optical band gap was found to be 5.16 eV. Thus, the obtained band gap reveals that the material may have high light conversion efficiency. This shows that LTA single crystals can be a good choice for the fabrication of optical devices such as light-emitting diodes (LEDs) and laser diodes.

### 3.4 Second Harmonic Generation (SHG)

The SHG efficiency of the title compound was determined using Kurtz– Perry Powder technique. [10]. A Q-switched Nd: YAG laser beam operating at 1064 nm, with an input energy of 0.70 J and pulse width of 6 ns with an input repetition rate of 10 Hz was used for this study. In order to confirm the NLO property the grown specimen was powdered with an average particle size range and filled in a micro-capillary tube of uniform bore and exposed to laser radiation and compared with standard KDP. There is no significant green emission from the specimen. It was concluded that SHG efficiency varies with particle size [11].

### 3.5 Laser Damage Threshold Studies

The limit of crystal to withstand high energy radiations defines its credibility for applications in laser-assisted devices. Hence the LDT of LTA crystal has been determined using the Nd: YAG laser operating at 1064 nm. The experimental setup was aligned and the crystal sample was placed at the focus point of the converging lens of focal length 10 cm. The polarized Gaussian beam of Nd: YAG laser was made incident on the sample for 6s/shot and the surface was observed. The energy was tuned using the attenuator and the same process was repeated at different energies. The LTA crystal showed major damage at 78 mJ. The ns pulse scale lasers induce the thermal gradient at the focused area of the crystal which results in photoionization of the material leading to decomposition/cracking/melting/fusion of the material [12-14]. Thus, it can be implicated that the LTA crystal could be used in laser-assisted NLO applications operative below the energy range of 78 mJ.

## 4.Conclusion

L-Tartaric acid single crystal was grown successfully by SEST method. Single crystal X-ray diffraction study shows the belongingness of crystal structure and the lattice parameters, which match the reported literature. Using the FTIR spectrum, all the functional groups were identified. The UV–Vis studies expose the transparency of crystal covering the entire visible spectrum region along with a cut-off wavelength of 231 nm with a bandgap of 5.1eV. Laser damage Threshold studies reveal that the LTA crystal could be used in NLO applications below the operation energy range of 78 mJ. Second-harmonic Generation of L-Tartaric acid was tested using Kurtz-Perry powder technique.

**Acknowledgements**

The authors gratefully acknowledge Vels Institute of Science, Technology and Advanced Studies (VISTAS), Pallavaram, Chennai - 600 117 for providing financial support to Ms S R Meeraa to carry out this research work under Vels Research Fellowship (VRF) scheme. The authors extend their acknowledgments to SAIF, IIT-Madras, Chennai - 600 036 for the data collection of single crystal X-ray diffraction.

## References

[1] C. Bosshard, K. Sutter, P. Pretre, J. Hulliger, M. Florsheimer, P. Kaatz, P. Gunter, *Adv. Non-linear Opt. 1 (1995).*

[2] D.S. Chemla, J. Zyss, Nonlinear Optical Properties of Organic Molecules and Crystals, 1,2, Academic Press, Orlando, FL, 1987

[3] *P.N. Prasad, D.J. Williams, Introduction to Nonlinear Optical Effects in Organic Molecules and Polymers Wiley, New York,1991*

[4] D.S. Chemla, J. Zyss, Nonlinear Optical Properties of Organic Molecules and Crystals Academic Press, New York, (1987)

[5] Redrothu and Kalainathan, optical properties of potassium nitrate doped l -threonine single crystal JAAST:Material Science, 1 (2014) 224-227

[6] Okaya, Y., Stemple, N. R. and Kay, M. I. Acta Cryst. 21 (1966) 237-243.

[7] V.G. Mohd Shkir, S AlFaify, I S Yahia, Mohd Anis, Structural, vibrational, optical, photoluminescence, thermal, dielectric, and mechanical studies on zinc (tris) thiourea sulfate single crystal: A noticeable effect of organic dye, Chin. Phys. B, 27 (2018) 54217-054217.

[8] M.Saravanan, Growth and characterization of dexterous nonlinear optical material: Dimethyl amino pyridinium 4-nitrophenolate 4-nitrophenol (DMAPNP), Optical Materials, 58 (2016) 327-341

[9] T. Jayapalan, S.J.D. Sathiyadhas, J. Michael, B. Settu, M.B.D. Sathiyadhas, Amala pushpam, thermophysical and optical properties of L-tartaric acid crystal. Cryst. Res. Technol. 53, (2018) 1–5

[10] S. Kurtz, T. Perry, A powder technique for the evaluation of nonlinear optical materials, Journal of Applied Physics, 39, (1968) 3798-3813

[11] M. Parthasarathy, R.Gopalakrishnan, Spectrochim. Acta A, 97, (2012) 1152-1158.

[12] S. Vanishri, J.B. Reddy, H. Bhat, S. Ghosh, Laser damage studies in nonlinear optical crystal sodium pnitrophenolate dihydrate, Applied Physics B, 88, (2007) 457-461.

[13] M.I. Baig, M. Anis, S. Kalainathan, B. Babu, G.G. Muley, Laser induced optical and microscopic studies of salicylic acid influenced KH2PO4 crystal for photonic device applications, Materials Technology, 32, (2017) 560-568.

[14] J.B. Reddy, S. Vanishri, G. Kamath, S. Elizabeth, H. Bhat, D. Isakov, M. Belsley, E. de Matos Gomes, T. Aroso, Growth and physical properties of a new chiral open-framework crystal for NLO applications: Cesium hydrogen l-malate monohydrate, Journal of crystal growth, 311, (2009) 4044-4049