

SYNTHESIS, GROWTH & CHARACTERIZATION OF TUSN CRYSTALS FOR THEIR OPTICAL AND CONDUCTIVITY ANALYSIS

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Abstract - Slow evaporation was used to generate the Thiourea strontium nitrate single crystals from saturated solution. Pure thiourea strontium nitrate single crystals' structure and crystallinity were verified by powder XRD, and FTIR spectroscopy revealed the frequency assignments of the several internal vibrational modes for the functional groups. UV-Vis spectroscopy was used for optical and transparency investigations. The material's value for optoelectronic applications is attested by its reduced cutoff wavelength of 241.67 nm and its high transmittance throughout the visible spectrum. Dielectric, and research was done to analyze its electrical characteristics. The reduced dielectric loss values imply that there are fewer flaws in the produced crystals.

Key Words: NLO semi organic crystals, PXRD, FTIR, UV-VIS Studies, Electrical Studies.

1. INTRODUCTION

The developing field of photonics heavily relies on nonlinear optics. The use of photons for information and image processing is known as photonics. [1] Applications for nonlinear optical processes can be found in essential operations like optical switching and frequency conversion. Compared to inorganic crystals, organic crystals can have very large nonlinear susceptibilities; nonetheless, they have poor processibility and a low damage threshold. Conversely, since they lack broad electron delocalization, pure inorganic NLO materials frequently have relatively minor optical nonlinearities despite having outstanding mechanical and thermal properties. Due to impurities and flaws brought on by the incredibly non-equilibrium development circumstances, inorganic crystals produced from high temperature melts may generally have lower laser damage thresholds and greater optical homogeneities across the bulk. A semi-organic crystal is one that combines the advantageous mechanical and thermal characteristics of an inorganic counter ion with the generally high optical nonlinearity of a completely organic ion. [2,3,4]

Compared to organic and inorganic materials, semi-organic materials have a significant nonlinearity, a strong resistance to laser-induced damage, a low angular sensitivity, and good mechanical hardness. Growing new semi-organic nonlinear optical materials has therefore

received a lot of attention due to its potential uses in second harmonic production, optical information storage devices, and telecommunications. The thiourea molecule's enormous dipole moment and capacity to build a vast network of hydrogen bonds make it an intriguing inorganic matrix modification.[5]

2. EXPERIMENTAL

Slow evaporation was used to generate the thiourea strontium nitrate single crystals from saturated solution. 18.75 grams of thiourea were finely powdered and combined with 100 milliliters of distilled water in a beaker to create the saturated solution^[6,7]. The polluted and scraped material of Strontium nitrate was added to this in little amounts until it was fully dissolved. Photographs of pure TUSN single crystals are shown in Fig-1.

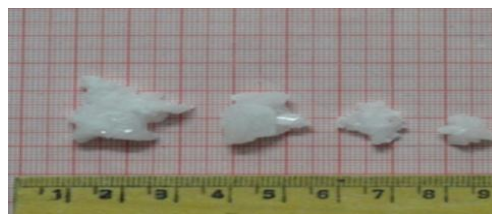


Fig 1 : Pure single crystal of TUSN

Using a magnetic stirrer, the solution was thoroughly mixed to increase its solubility, and the operation was repeated until the last bit of the material had been dissolved. Filter paper was then used to filter the mixture. Polythene paper was used to cover the beaker's top and create a few tiny holes. This beaker was not disturbed and was maintained at room temperature. Ten to fifteen days were needed to gather the individual crystals.

2.1 Characterization of Crystals

Powder X-ray diffraction experiments were performed on pure thiourea crystals. Using an XRD-ULTIMA 111 diffractometer, the generated crystals' X-ray powder diffraction pattern was acquired, and the diffraction peaks

are indexed. A FT-IR Perkin Elmer RX-1 spectrophotometer is used to perform FT-IR studies on the produced crystals, identifying functional groups using the pellet approach over a 400–4000 cm⁻¹ KBr range. The spectra showed the suggested assignments of the several internal vibrational modes.

A UV-Perkin Elmer Lampda 35 spectrophotometer has been used to evaluate optical transmission and absorption spectral analysis over a wavelength range of 200–1200 nm. Light in the visible portion of the electromagnetic spectrum has a uniformly high transmission rate (99%), which may be advantageous for device applications. A Dielectric-LCRZ meter TH2816A with a standard two terminal sample holder was used to test the dielectric of a polished part of the samples with known dimensions.

3. RESULTS AND DISCUSSION

3.1 XRD ANALYSIS:

Powder X-ray diffraction experiments were performed on pure TUSN single crystal. The XRD-ULTIMA 111 diffractometer was used to get the generated crystals' X-ray powder diffraction pattern. By using the reflection mode for scanning, the scanning rate was kept constant over a 2θ range of 10° to 80°. This investigation verified the crystallinity of pure TUSN single crystal, and the diffraction peaks are indexed. Figure 2 displays these, and Table 1 displays the intensity along with the accompanying d values. The distinct, sharp peaks in the XRD patterns indicate that the crystals of pure thiourea are single-phase and have good crystalline characteristics.^[8]

Table -1: XRD data for TUSN Crystal

2θ (degree)	d-value	Intensity (cps)	I/I ₀ (%)
19.380	4.5764	137	4
20.040	4.4271	2	54
20.900	4.2468	4	100
23.360	3.8049	1	30
25.620	3.4741	9	22
28.520	3.1271	9	24
29.040	3.0723	7	18
30.440	2.9341	3	10
31.460	2.8413	4	10
35.580	2.5211	3	9
36.420	2.4649	3	8
38.940	2.3110	2	6
41.900	2.1543	1	4
51.080	1.7866	3	8

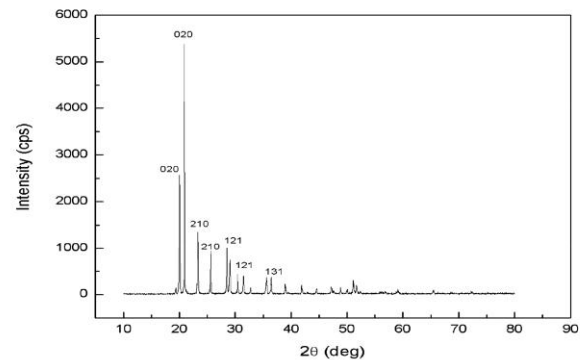


Fig -2: Powder X-ray diffraction patterns of grown TUSN single crystal

3.2 FT - IR ANALYSIS:

A FT-IR Perkin Elmer RX-1 spectrophotometer was used to perform FT-IR analyses on the produced crystals utilizing the pellet method throughout a 400–4000 cm⁻¹ KBr range. The FTIR spectra of pure TUSN single crystal are displayed in Fig. 3. The spectra showed the suggested assignments of the several internal vibrational modes.^[9] The infrared spectra of the crystals have been used to explain TUSN single crystal, which may be able to create coordinate bonds through both nitrogen and sulfur. The asymmetric stretching modes of NH₂ are responsible for the absorption band at frequency 3406 and 3271 cm⁻¹. Table 2 illustrate the vibrational modes observed in the TUSN single crystal.

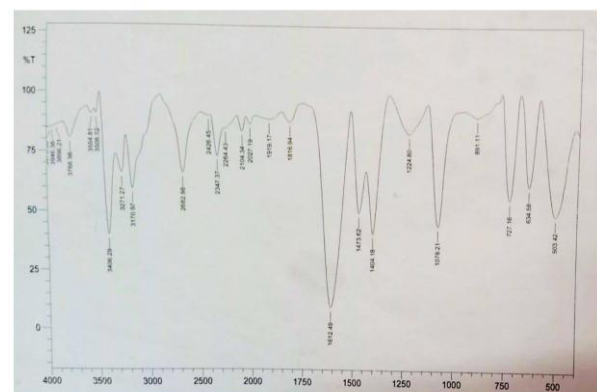


Fig-3: FTIR spectra of grown TUSN single crystals

Table -2: Vibration modes observed in the Thiourea crystals

Pure TUSN Wave number (cm ⁻¹)	Band Assignments
3406.29	NH ₂ asymmetric stretching
3271.27	NH ₂ asymmetric stretching
3170.97	NH ₂ symmetric stretching
1612.49	NH ₂ bending
1473.62	C-N asymmetric stretching
1404.18	C=S asymmetric stretching
1078.21	C-N symmetric stretching
727.16	C=S symmetric stretching
634.58	N-C-S asymmetric bending
503.42	N-C-N asymmetric bending

3.3 UV - VISIBLE ANALYSIS

A UV-Perkin Elmer Lampda 35 spectrophotometer has been used to measure the UV-Vis spectral analysis over a wavelength range of 200–1200 nm. Since the molecule's absorption of UV and visible light promotes electrons in the σ and π orbitals from the ground state to a higher energy state, the optical transmission spectrum typically provides useful information about the molecule's structure. Since the formed crystal can only be employed in the extremely transparent region, the transmission spectrum is significant from the device's point of view. Figure 4 displayed the optical transmission spectrum that was recorded. In this case, the crystal exhibits good transmittance throughout the visible spectrum. Together with the aforementioned, the lower cutoff wavelength of 241.67 nm confirms the material's suitability for optoelectronic applications. The wavelength range in which the absorption spectrum was obtained was 200–1200 nm. Figure 5 displays the TUSN single crystal UV-visible absorbance spectrum. According to the absorption spectrum, there is a significant absorption in the 190–300 nm range in the TUSN single crystal.^[10,11,12]

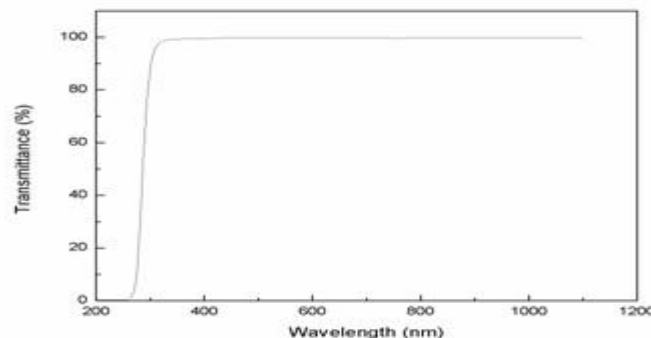


Fig-4: Transmission spectra of grown TUSN single crystal

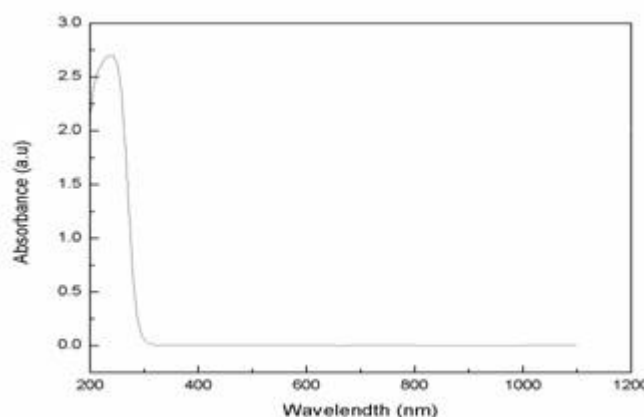


Fig - 5 : Absorption spectra of grown TUSN single crystal

3.4 ELECTRICAL STUDIES - DIELECTRIC AND CONDUCTIVITY ANALYSIS

A Dielectric-LCRZ meter TH2816A with a standard two terminal sample holder was used to test the dielectric of a polished part of the samples with known dimensions. By creating a parallel plate capacitor, the pure TUSN single crystal was positioned in between the two copper electrodes. The frequency was then changed from 50 Hz to 200 KHz in order to determine the capacitance on the sample. Figure 6. demonstrates how the dielectric constant of pure TUSN single crystal changes with frequency. In this case, it was discovered that the dielectric constant of pure TUSN single crystal first had high values at low frequencies and then decreased as the frequency increased. The reduced dielectric loss values imply that there are fewer flaws in the produced crystals. Dielectric loss and dielectric constant fluctuation as a function of frequency are nearly identical in nature. The resistivity of pure TUSN single crystal as a function of frequency is displayed in Fig. 8. At low frequency, this too has high values.

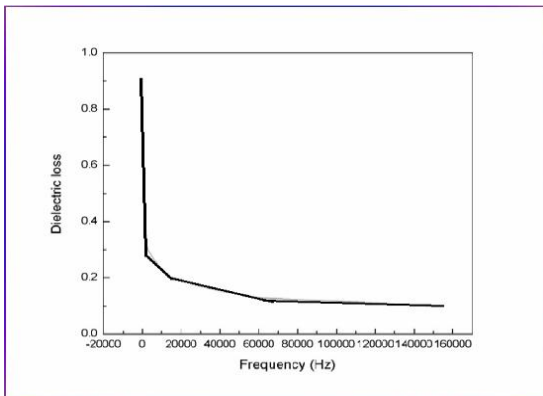


Fig – 6 : Dielectric constant analysis of TUBN single crystal

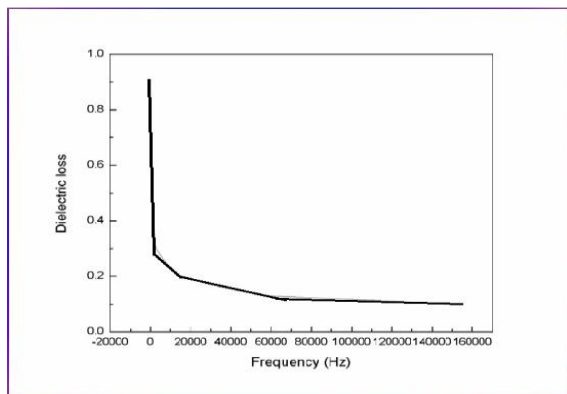


Fig – 7 : Dielectric loss analysis of TUBN

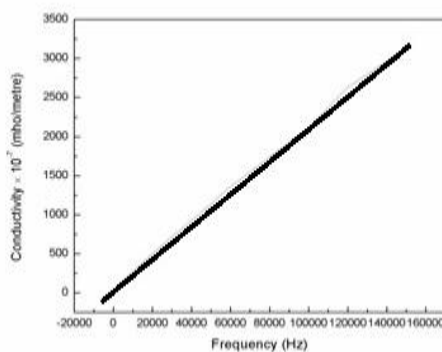


Fig – 8 : Resistivity variations of TUBN with frequency

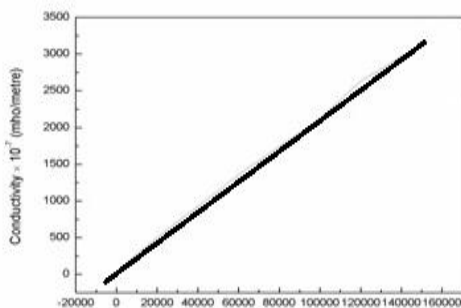


Fig – 9 : Conductivity variations of TUBN with frequency

3.4 SCANNING ELECTRON MICROSCOPY (SEM) WITH ENERGY DISPERSIVE X – RAY SPECTROSCOPY (EDS)

The JEOL JSM 5610 LV Scanning Electron Microscope, which has a maximum magnification of 2,00,000 times with a resolution of 5 nm and an acceleration voltage of 0.3, was used to investigate the surface morphology. The chemical microanalysis method known as EDS is used in conjunction with SEM. The quantity and energy of X-ray pictures that were released were measured using the EDS X-ray detector. The surface nature and suitability for device production are described in [SEM study JEOL JSM 5610 v], which is also used to check for defects. It has been found that different contaminants have varying degrees of efficiency in altering the surface morphology. Scanning electron micrographs of TUSN single crystal are seen in Figure 10. The micrograph illustrates the doped specimen's surface characteristics. It exhibits a nice, uniform surface that resembles a diamond and is hexagonal in shape, with a few bubble spaces that may be caused by solvent evaporating off the crystal surface. [13]

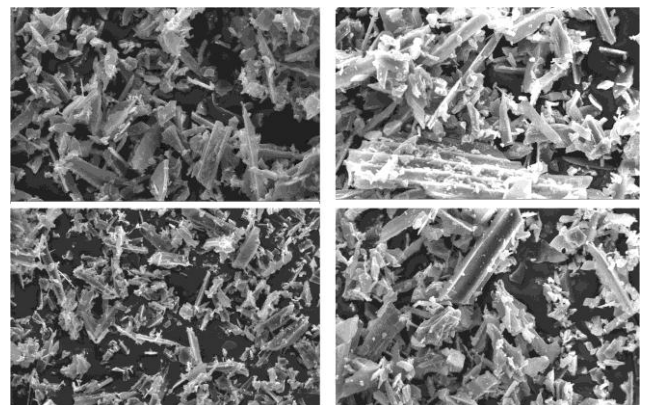


Fig 10 : SEM images of TUSN single crystal

EDS figure 11 provides strong confirmation of strontium doping, which results in its incorporation into the crystalline matrix. The graph clearly shows the increased strontium concentration in the TUSN crystalline matrix. Additionally, Sr accommodating capacity on the crystal's surface is uniform. [14,15]

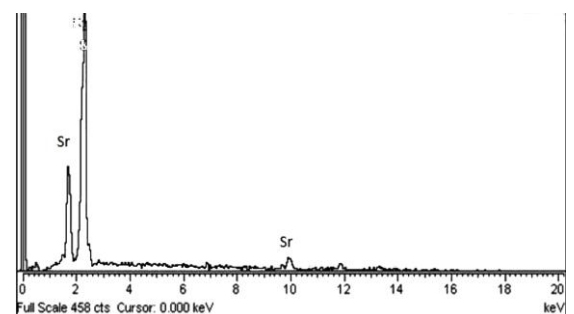


Fig 11 : EDS graph of Sr doped TU crystal

4. CONCLUSIONS

Slow evaporation was used to generate TUSN single crystal of very high purity from saturated solution. The existence of functional groups was verified by the FTIR spectra, and vibration modes were identified. The TUSN single crystal exhibits good transparency throughout the visible spectrum and substantial absorption in the 190–300 nm range, according to the UV transmission and absorption spectrum. The material's formation and crystalline nature were verified by XRD analysis. A low dielectric loss was found when the fluctuations in dielectric constant, dielectric loss, conductivity, and resistivity were examined at various frequencies. The crystal's surface morphology was examined using SEM, which revealed a hexagonal and diamond-like appearance with some bubble voids that may have resulted from solvent evaporating off the crystal surface.

EDS also confirmed the presence of strontium in the doped specimen and a slight variation in the doped specimen's intensity when compared to the pure TU specimen because of the doping effect. The FT-IR spectrum

shows more vibration peaks when strontium is heavily doped on thiourea.^[16]



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REFERENCES

- [1] P. N. Prasad and D. J. Williams, Introduction to Nonlinear Optical Effects in Molecules and Polymers, John-Wiley and Sons Inc, New York, (1991).
- [2] D. Ledoux, A. Lepers, J. Perigaud, J. Badan, and J. Zyss, Opt. Commun., **80**, 149 (1990).

- [3] S.X.Dou, D.Josse, and J.Zyss, *J. Opt. Soc. Am. B*, **10**, 1708 (1993).
- [4] G.Knopfle, R. Schlessler, R. Ducret, and Gunter, *Nonlinear Opt.*, **9**, 143 (1995).
- [5] C.Serbutoviez, C.Bosshard, G. Knopfle, P.Wyss, P.Pretre, P.Gunter, K.Schenk, E.Solari, and G.Chapuis, *Chem. Mater.*, **7**, 1198 (1995).
- [6] N.R.Dhumane, S.S.Hussaini et al., *Appl. Phy. A: Mat. Sci. and Proc*, **95**, 3 (2009).
- [7] G.Madhurambal, M.Mariappan, *Ind.J.Pure and Appl. Phy.*, **48**, .264 (2010).
- [8] C.Krishnan, P.Selvarajan, , *J. Expt. Sci.*, **1**,3 (2010).
- [9] T.Thaila, S.Kumara Raman, *Archives Appl. Sci. Res*,**3**, 1 (2011).
- [10] J.Thomas Joseph Prakash, M.Lawrence, *Inter. J. Comp. Appli.*, **8**, 3 (2010).
- [11] K.Nagarajan, S.Kumara Raman, R.Sundaram, *Cry. Res. Tech.*, **44**, 4 (2009).
- [12] Z.Kotler, R. Hierle, D.Josse, J. Zyss, and R.Masse, *J. Opt. Soc. Am. B*, **9**, 534 (1992).
- [13] S.Velsko, "Laser Program Annual Report", Lawrence Livermore National Laboratory, Livermore, CA, (1990).
- [14] N.B.Singh, T.Henningsen, R.H. Hopkins, R.D. Mazelsky, E.P.Hamacher, F. Supertzi, D.E.Hopkins, O. Zelmon, P.Singh, *J. Cryst. Growth*, **128**, 976 (1993).
- [15] R.Mohan Kumar, D.Rajan Babu, D. Jayaraman, R.Jayavel, and K.Kitamura, *J. Cryst. Growth*, **275**, e1935 (2005).
- [16] J.Badan, R. Hierle, A. Perigand, J. Zyss, In and D.J. Williams,(Ed.). "Nonlinear Optical Properties of Organic Molecules and Polymeric Materials", 233 D.5, American Chemical Society, Washington, DC, (1993).