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Synthesis, Growth and Characterization of L-Proline Bisthiourea Single Crystal

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ABSTRACT: *Single crystals of L-proline bisthiourea were grown by the slow evaporation method at room temperature. The grown crystal characterized by the unit cell parameters obtained from single crystal X- ray diffraction analysis, optical properties of the grown crystals were investigated by UV-Vis spectroscopy, FTIR, EDAX, Thermogravimetric analysis and Differential thermal analysis.*
Keywords: *Crystal growth; Single X-ray diffraction; FTIR; EDAX; UV; TGA and DTA.*

I. INTRODUCTION

In this recent researchers, the nonlinear optical crystal produced in industrial process are needed to be of high quality because the performance of the (laser, high speed computer, electro-optic and acoustic devices, optical information storage device) using crystals depends height on the crystal quality. Non linear optical materials are also providing the major function of optical logic, frequency shifting, frequency doubling etc. Organic amino acids are fascinating stuffs formed by weak Vandervaal's , hydrogen bonds and as they contain a proton donor carboxyl acid (- COO) group and proton acceptor amino group in them and amino have substantial properties make them supreme candidates for NLO applications[1,2]. In the present work, single crystals of L-Proline bisthiourea has been grown from the aqueous solution by the slow evaporation method at room temperature. The grown crystal were subjected to various characterization studies such as Single crystal X-ray diffraction , UV-Visible studies, FTIR studies, Thermal studies and EDAX test.

II. EXPERIMENTAL PROCEDURE

Fig.1. Photography of grown crystal

The title of the material was synthesized by taking L-Proline and thiourea in the 1:2 molar ratio and dissolved in double distilled water and stirrer well using magnetic stirrer for 3 hours to form a transparent into a fresh 150 ml beaker and covered with aluminum foil before leaving it for slow evaporation method at room temperature. The good quality single crystal (fig.1.) was obtained after 46 days.

III. RESULTS AND DISCUSSION

3.1. Single crystal X-ray diffraction analysis

The grown crystal was subjected to single crystal X-ray diffraction analysis was carried out using a ENRAF NONIUS CAD4 X-ray diffractometer with MO Kα (λ = 0.71073 Å) radiation to identify the cell parameters . The single crystal X-ray diffraction analysis confirms the orthorhombic crystal system with space group $P2_12_12_1$. The estimated unit cell parameter values of grown crystal are, a = 5.487 Å, b = 7.641 Å, c =8.857 Å, with cell volume V = 357.9Å^3.

3.2. UV – Visible spectral analysis

The optical absorption spectrum carried out using by Varian carries 5E UV-Vis Spectrophotometer in the range 200 nm 1400 nm. The recorded absorption spectrum is shown in fig.2.in which lower cutoff wavelength is 301 nm. The optical absorption spectrum of grown crystal shows a good absorbance in the entire visible region;

there is no transmittance is the range from 301nm to 1400 nm which is useful for optoelectronic applications [3]. The optical band gap energy of grown crystal is determined as 3.96 eV

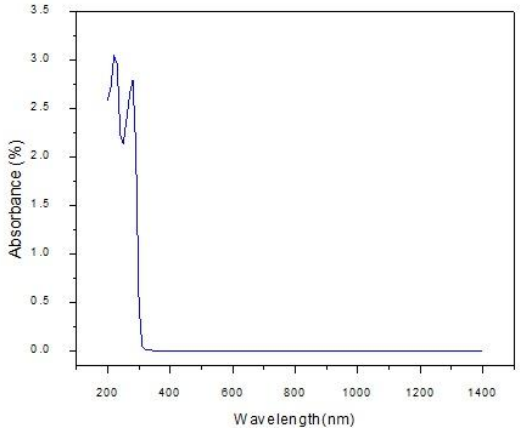

Fig.2. UV-Visible spectrum of LPBT crystal.

3.3. FT-IR spectral analysis

The Fourier transform infrared spectra were analyzed for powder crystals using BRUKER-Fourier transform infrared spectrometer by KBr pellet technique in the range 4000 cm^{-1} to 400 cm^{-1}. The observed FT-IR spectrum of grown crystal is shown in fig.3.

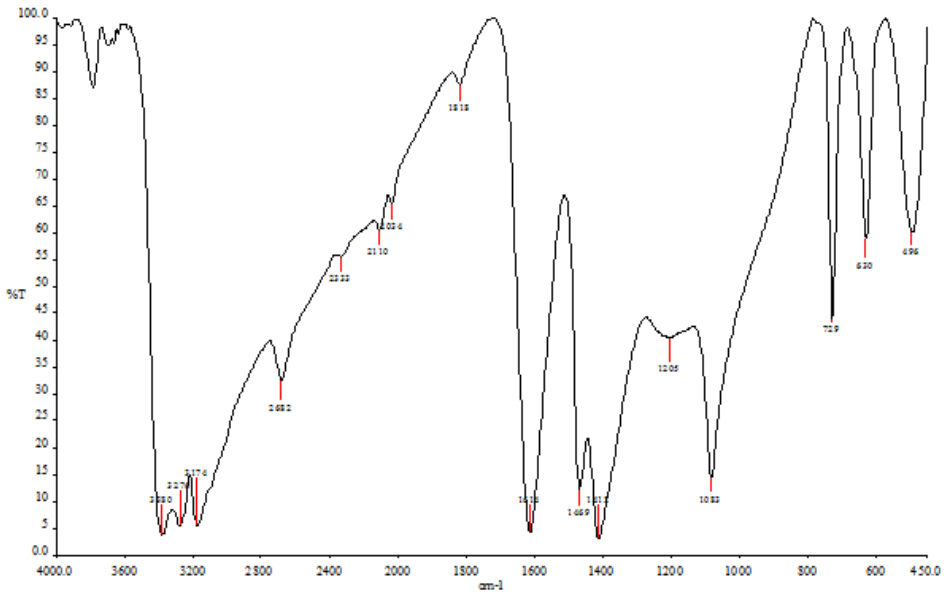

Fig.3.FT-IR spectrum of LPBT crystal.

From the spectrum is observed that the very broad peak 3174cm^{-1} indicates the presence of C-H stretching. The peaks at 1818 cm^{-1} may be attributed to overtone of C-C stretching, the peak at 1616 cm^{-1} is assigned to NH_2^+ bending vibration of L-Proline. The symmetric stretching mode of CS group is present at the peak 729 cm^{-1}. The zwitterionic COO^- wagging is predicted by the peak at 630 cm^{-1}. The peak at 496 cm^{-1} is assigned to the S-C-N bending vibration.

3.4. Thermal Studies

The thermal stability of grown crystal LPBT was studied using Thermogravimetric (TG) and Differential thermal analysis (DTA). The thermogravimetric analysis of LPBT was carried out in nitrogen atmosphere between the temperature ranges from 0°C to 450°C using CNST analyzer. The resulting thermogram and its differential derivative are shown in fig.4. From LPBT graph, it is clear that the grown crystal of LPBT is stable upto 177°C. After that the crystal starts to melt and the decomposition continues upto 400°C. In DTA, very strong endothermic peak is observed at 238°C corresponds to the decomposition of the material.

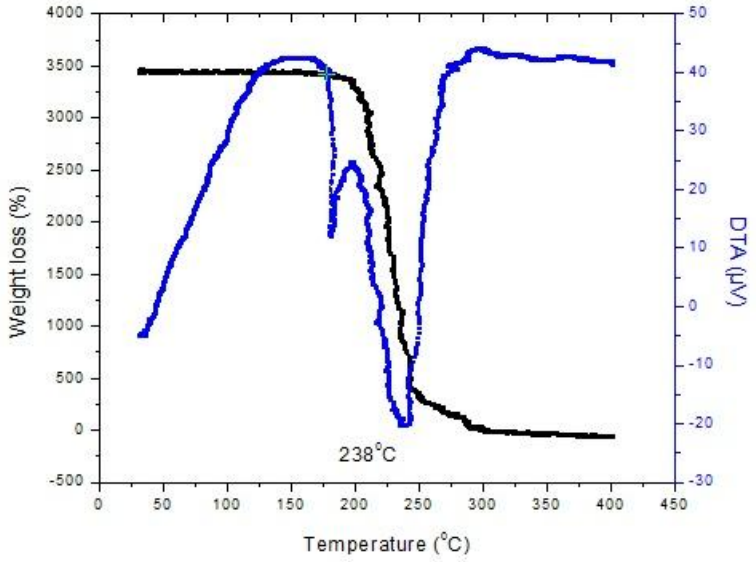

Fig .4. TGA/DTA curve of grown crystal

3.5. EDAX Analysis

The energy dispersive analysis carried out on LPBT crystal was used to detect the real concentration percent in the in the sample. The EDAX spectrum of l-proline bisthiourea crystal has the peaks attributed to the C, S, O at different energies is depicted in fig.5. Table 1 shows the weight percentage of the elements present in the sample.

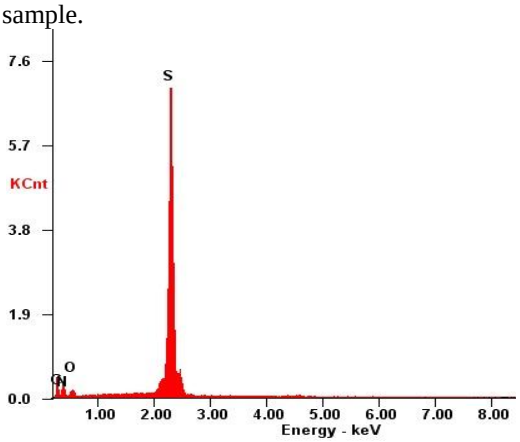

Fig.5. EDAX spectrum.

Element	*Wt%*	*At%*
CK	32.80	45.99
NK	23.45	28.19
OK	05.38	05.67
SK	38.37	20.15
Matrix	Correction	ZAF

TABLE.1

IV. CONCLUSION

Optically good quality of LPBT single crystals were successfully grown by slow evaporation technique at room temperature. The grown crystal belongs to orthorhombic system with space group $P2_12_12_1$.Fourier transform infrared spectroscopic analysis confirmed the functional groups and modes of the vibration of the grown crystal. The thermal studies indicate that the grown crystal is stable upto 177◦C. The elemental composition was determined by EDAX studies, which show that the grown crystal is of good quality.

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