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Crystal growth, spectral, optical, laser damage, photoconductivity and dielectric properties of semiorganic L-cystine hydrochloride single crystal

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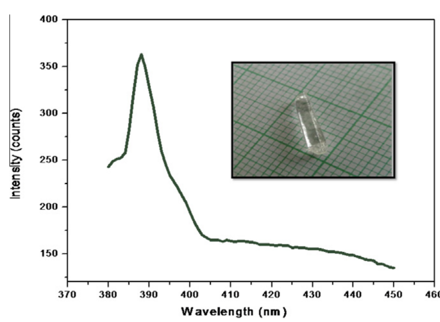
HIGHLIGHTS

- L-Cystine hydrochloride single crystal is grown by slow evaporation technique at 40 °C.
- Optical band gap of the crystal is found to be 3.8 eV.
- Thermally it is stable up to 201 °C.
- It exhibits violet fluorescence emission peak at 388 nm.

GRAPHICAL ABSTRACT



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ABSTRACT

The semiorganic single crystals of L-cystine hydrochloride have been grown by slow evaporation solution growth technique at 40 °C. The grown crystals were subjected to single crystal XRD, FTIR, optical absorbance, laser damage threshold, photoluminescence, photoconductivity and dielectric studies. Single crystal XRD studies reveal that the crystal belongs to monoclinic system with space group C2 and the lattice parameters are $a = 18.63$ (Å), $b = 5.28$ (Å), $c = 7.26$ (Å), $\alpha = 90°$, $\beta = 103.70°$, $\gamma = 90°$ and $V = 696$ (Å^3). FTIR spectroscopy confirms that a band at 1731 cm^{-1} represents characteristic of α-amino acid hydrochlorides. The UV–Vis–NIR absorption spectrum was analyzed and the optical band gap energy was found to be 3.8 eV. The crystal exhibits sharp emission peak at 388 nm. The thermal characteristics of crystals were studied by TG-DTA, which indicate that there is no weight loss up to 201 °C. Surface laser damage threshold value of title compound was estimated using high power Q-switched Nd:YAG laser operating at 1064 nm. Dielectric and photoconductivity studies were also carried out for the grown crystals.

1. Introduction

In last two decades, extensive studies have been made for obtaining new nonlinear optical (NLO) materials because of their potential applications in the field of optical modulators, telecommunications, color displays, optical switching and optical signal processing [1–3]. Inorganic materials have high melting point, high mechanical strength, high degree of chemical inertness and low optical nonlinearity. Organic materials have excellent properties compared to the inorganic solids which show lower dielectric constants and enhanced NLO responses. Organic non-linear optical crystals are usually formed by weak Vander-Waals and hydrogen bonds. So they possess poor mechanical and thermal properties [2–4]. In order to overcome the limitations of those materials, in recent years the organic materials were mixed with inorganic

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material (semi-organic) to improve their chemical stability, physico-chemical properties, mechanical strength, laser damage threshold, optical non-linearity and thermal stability [5–7]. In this point of view, some complexes of amino acid have been combined with inorganic compounds such as dichloro (4-hydroxy-l-proline) cadmium(II) [2], L-proline cadmium chloride monohydrate [3], L-alanine sodium nitrate [5], glycine sodium nitrite [6], L-arginine hydrochlorobromide [7], L-cystine dihydrochloride [8] and L-cystine dihydrobromide [9]. In this series, L-cystine hydrochloride is also a good and promising candidate for SHG and various other applications in the semi-organic family. Amino acids are bifunctional organic molecules that contain both a proton donor carboxylic (COO^-) and proton acceptor amino group (NH_2). This dipolar nature of amino acids shows peculiar physical and chemical properties. L-Cystine is a sulfur-containing amino acid. In the L-cystine molecule, the functional groups, such as NH_2 and COOH have a strong tendency to coordinate with inorganic cations and metals [8–10]. The molecular structure of L-cystine hydrochloride was reported by Srinivasan [10] and the NLO and mechanical stability were studied by Selvaraju et al. [11]. Single crystals play vital role in laser technology, optoelectronics, microelectronics industry and so on. From application point of view, its needs wide optical transparency, high mechanical strength, large laser damage threshold, high thermal behavior and low dielectric properties [5,6,12–15]. Based on the above aspects, the present paper describes and discusses the optical, laser damage threshold, photoconductivity, photoluminescence, TG/DTA and dielectric properties of semi-organic L-cystine hydrochloride crystal.

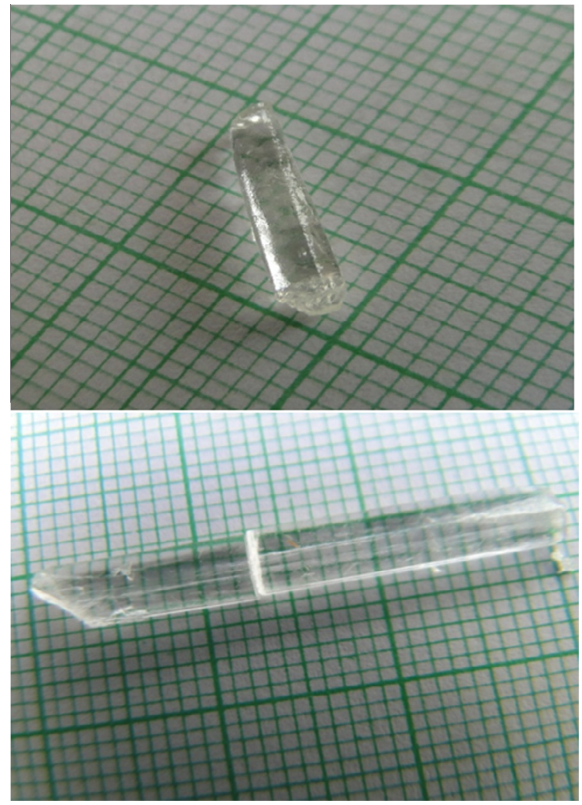

Fig. 1. L-Cystine hydrochloride single crystals.

2. Experimental technique

2.1. Material synthesis and crystal growth

L-Cystine hydrochloride crystal is synthesized using L-cystine and hydrochloric acid (Merck GR) which are mixed in the stoichiometric ratio of 1:1 in deionised water which has resistivity of 18.2 MΩ cm. The calculated amount of L-cystine was first dissolved in the deionised water. The solution was thoroughly stirred for 4 h using magnetic stirrer at 40 °C. The mixture of solution was found to be cloudy and HCl was added and stirred well for 24 h till a clear solution was obtained. Then the saturated solution was filtered using Whatman filter paper in clean vessels and the vessels containing the solution were closed with polythene covers and domiciliated in the constant temperature bath at 40 °C. The synthesized salt was sanctified by consecutive recrystallization activity. Optically transparent single crystals were obtained after 35 days and the harvested crystals are shown in Fig. 1.

2.2. Characterization techniques

Single crystal X-ray diffraction analysis was carried out using a Bruker AXS Kappa APEX II CCD Diffractometer, equipped with graphite-monochromated MoKα radiation (λ = 0.71073 Å) to identify the crystal structure and to estimate the lattice parameter values. FTIR spectrum of the title compound was recorded using Bruker AXS FTIR spectrometer in the range of 500–4000 cm^{-1} with single reflection ATR accessory. The UV–Vis–NIR spectrum for the L-cystine hydrochloride crystal was recorded using Perkin Elmer Lambda 35 spectrometer at room temperature in the range 200–1100 nm. The LDT study for the grown crystal was carried out using the high-power Q-switched Nd:YAG laser with 10 Hz pulse repetition rate. The pulse width of the laser is 7 ns for 1064 nm. A highly polished clear surface and defect free crystal with the thickness of 1.5 mm is used for the LDT measurement. The dielectric behavior of L-cystine hydrochloride crystal was carried out using Agilent Model 4284A LCR meter in the frequency range 1 kHz–1 MHz at various temperatures (40–100 °C). Silver paste was coated on both sides of the L-cystine hydrochloride crystal and then placed between the two copper electrodes to form the parallel plate capacitor. The photoconductivity studies were carried out on a cut and polished sample of the grown single crystal by using KEITHLEY 6487 picoammeter in the presence of DC electric field at room temperature (303 K). Silver paint was coated on the surface of the crystal in order to make contact between the electrode and the crystal. The sample is kept in vacuum. The light from a halogen lamp of 100 W was used to measure the photocurrent (I_p). The weight loss (TG) and energy change analyses (DTA) of L-cystine hydrochloride samples were carried out in the temperature range between 35 °C and 300 °C at a heating rate of 10 °C/min under nitrogen atmosphere using Perkin Elmer Diamond TG-DTA instrument. A small piece of crystal weighing 4.432 mg was taken for the measurement. The photoluminescence measurements have been carried out using Shimadzu Spectrofluorophotometer R.F-5031 PC series with the slit width of 3 nm at room temperature.

3. Results and discussion

3.1. Single crystal X-ray diffraction

From single crystal X-ray diffraction analysis, it was confirmed that the crystal belongs to monoclinic system with space group C2. Lattice parameters are a = 18.63 (Å), b = 5.28 (Å), c = 7.26 (Å), α = 90°, β = 103.70°, γ = 90° and the volume of the unit cells is found to be V = 696 (Å^3), which is in close agreement with the reported values [10]. The lattice parameters are given in Table 1.