

# GROWTH, STRUCTURAL AND OPTICAL STUDIES ON PURE AND L-HISTIDINE DOPED SINGLE CRYSTALS OF COPPER SULPHATE

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## Abstract

Single crystals of pure and L-Histidine doped copper sulphate penta hydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) were grown by slow evaporation method at room temperature. The grown crystals were carried out by Powder X-ray diffraction analysis to find out the crystalline nature. The different modes of vibration present in the pure and L-Histidine doped  $\text{CuSO}_4$  crystals were identified by FTIR spectra. The wide optical transparency in the entire visible region was shown by UV-Vis-spectral analysis.

**Keywords:** Crystal Growth, XRD, FTIR, UV-Visible

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## 1. INTRODUCTION

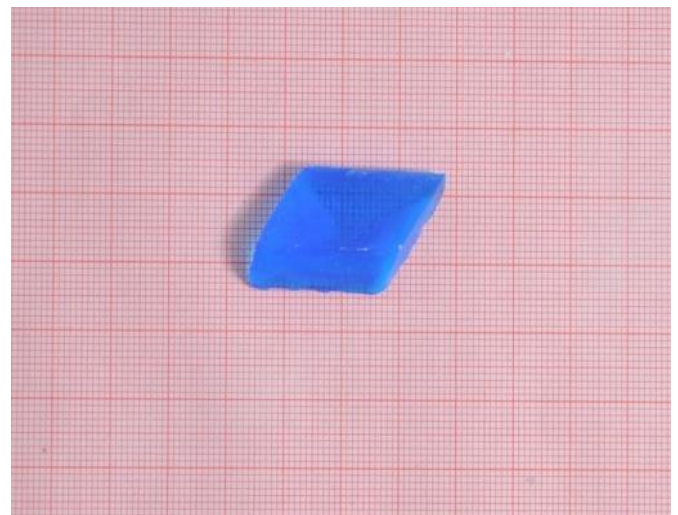
The anhydrous form of copper sulphate is a pale green or gray-white powder. However, its pentahydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) form is bright blue in colour. Copper sulphate exothermically dissolves in water to give the aquo complex  $[\text{Cu}(\text{H}_2\text{O})_6]^{2+}$ , which has octahedral molecular geometry and is paramagnetic. Other names for copper sulphates are copper (II) sulphate, "blue vitriol" and "bluestone". Crystals exhibit non-linear optical (NLO) property has been of great importance in recent years because of their wide spread applications such as high-speed information processing, optical communications and optical data storage, photonics and electro optics modulation [1,2]. These crystals are useful for nonlinear optical devices based on their optical second harmonic generation. The NLO property of glycine added copper sulphate single crystal was reported by S.N. Jeyanthi et al. very recently [3]. Copper sulphate also applicable in synthetic fibre industry, used as an electrolyte in copper refining, the paint industry uses it in anti-fouling paints and it plays a part in the coloring of glass, antiseptic agent, and antifungal agent. The goal of this paper is to give report about the growth of pure and L-Histidine doped copper sulphate single crystals by slow evaporation method, the structural and optical properties of the grown crystals were identified by powder X-ray diffraction data, Fourier Transform Infrared and UV-Vis spectral analysis.

## 2. EXPERIMENTAL

### 2.1 Crystal Growth

Analytical Reagent (AR) grade copper sulphate salt was used for the growth of pure and L-Histidine doped copper sulphate single crystals. Accordance with the solubility test suitable amount of copper sulphate salt was weighted. The mixture of deionised water and copper sulphate salt placed in a magnetic stirrer and is stir well with applying mild

temperature about 40°C. The stirring was continued until a homogeneous saturated solution was obtained. For doping purpose 0.002 wt% L-Histidine was added to the same solution. Then the solution was optimally covered using a plane thin paper sheet. Good quality transparent crystals were grown within 10-15 days. The grown crystals were carried out powder X-ray diffraction, Fourier Transform Infrared and UV-Visible spectral analysis. Transparent good quality crystals with perfect shape and free from macro defects were used for characterization.



**Fig -1:** Photograph of pure copper sulphate single crystal



**Fig -2:** Photograph of 0.002 wt % L – Histidine doped copper sulphate single crystal

## 2.2 Material Characterization

The grown pure and L – Histidine doped copper sulphate single crystal were characterized by Powder X-ray diffraction using XPERTPRO diffractometer. FTIR spectra of the grown crystals were characterized by Jasco 4100 Spectrophotometer equipped with ATR. UV-Vis absorption spectra of the grown crystals were recorded using Systronics UV-Vis double beam spectrophotometer.

## 3. RESULTS AND DISCUSSION

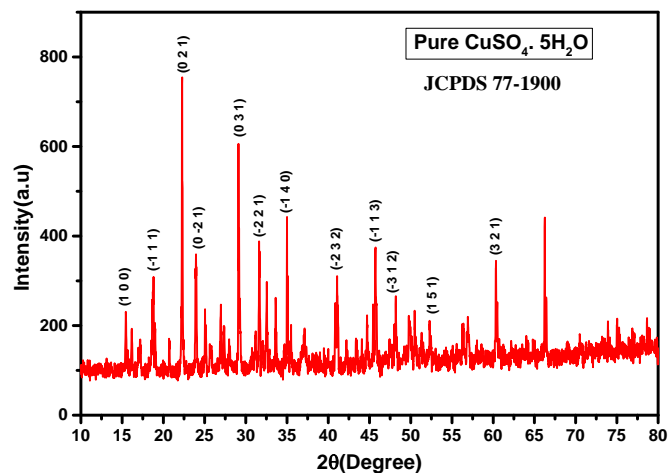
### 3.1 Powder X-ray Diffraction Analysis

The powdered forms of the grown crystals were subjected to powder X-ray diffraction analysis. The X-ray diffraction data were recorded using monochromatic  $\text{CuK}\alpha$ -1 radiation of wavelength  $1.54060 \text{ \AA}$  recorded between  $2\theta$  ranging from  $10^\circ$  to  $80^\circ$ . The diffraction pattern contains various reflections corresponding to various crystallographic planes. The narrow peaks in the XRD pattern show the good quality crystalline nature of the grown crystals [4, 5]. The data obtained is good agreement with standard JCPDS file (JCPDS 77-1900) of copper sulphate pentahydrate single crystal. The reflection planes were indexed accordance with the JCPDS file. The presence of the doping in the composition of copper sulphate crystal increases the intensity of peaks comparing to pure copper sulphate crystal. This reveals that the doping has brought a change in the internal structure of the crystal [6]. From powder X-ray diffraction analysis, it has been confirmed that the grown pure and doped copper sulphate crystal belongs to triclinic system. The calculated lattice parameters show in table (1).

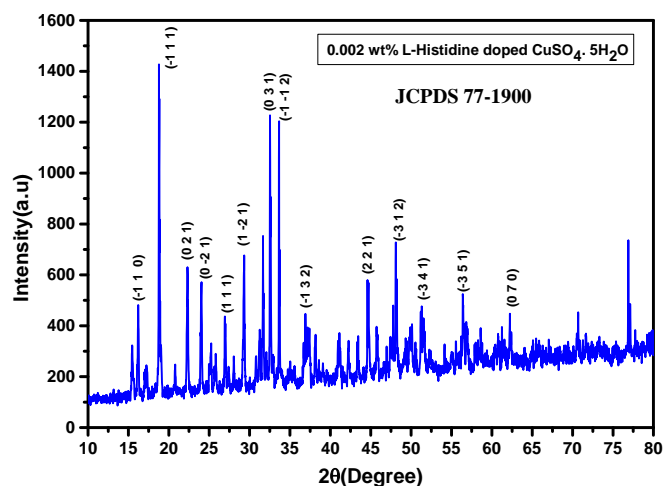
**Table -1:** Lattice parameters of pure and 0.002 wt% L - Histidine doped copper sulphate single crystals

system	Pure copper sulphate	0.002 wt% L-Histidine doped copper sulphate
a ( $\text{A}^\circ$ )	6.121	6.101
b ( $\text{A}^\circ$ )	10.678	10.737
c ( $\text{A}^\circ$ )	5.931	5.987
$\alpha$ ( $^\circ$ )	81.82	82.73

$\beta$ ( $^\circ$ )	107.41	107.17
$\gamma$ ( $^\circ$ )	102.41	102.72
Volume( $\text{A}^\circ$ ) <sup>3</sup>	360.056	364.766

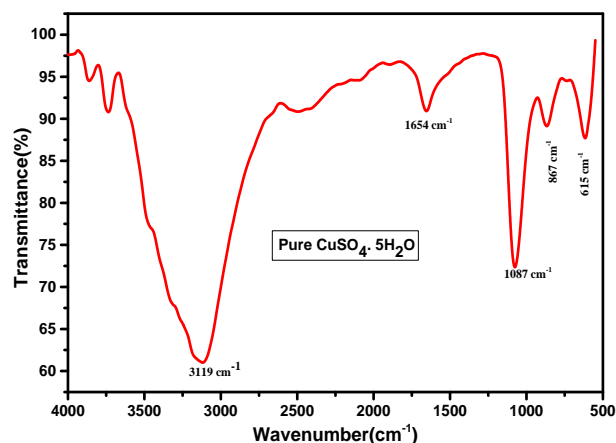


**Fig -3:** Powder X-ray diffraction pattern of pure copper sulphate single crystal



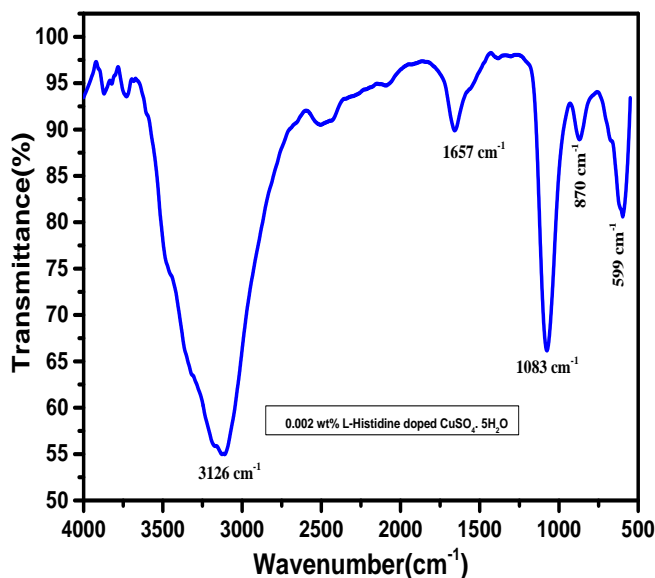
**Fig -4:** Powder X-ray diffraction pattern of 0.002 wt % L – Histidine doped copper sulphate single crystal

### 3.2 Fourier Transform Infrared (FTIR) Analysis



**Fig -5:** FTIR spectra of pure copper sulphate single crystal

Fourier Transform Infrared spectral analysis is one of the most prominent and broadly used spectroscopic methods for analysing the structure of unknown compounds quantitatively. It is useful to find out the functional groups, internal structure of the molecules and the nature chemical bonds of a compound. The Fourier Transform Infrared spectra of pure and L-Histidine doped copper sulphate crystals were recorded in the range of  $550 - 4000 \text{ cm}^{-1}$ . The FTIR spectra of pure and doped copper sulphate crystals were shown in figure (5) and figure (6) respectively.

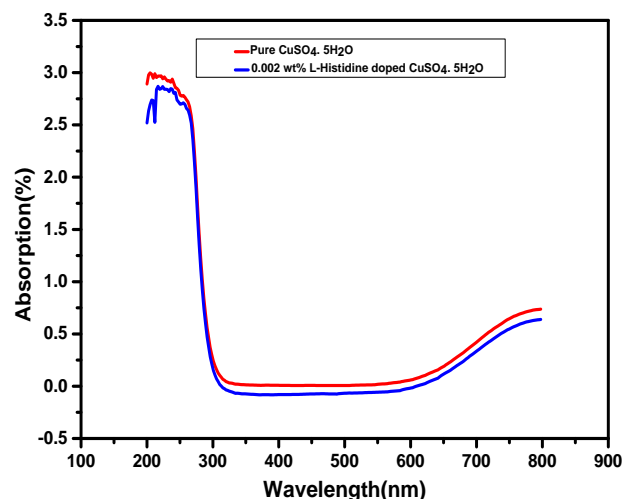


**Fig -6:** FTIR spectra of 0.002 wt % L – Histidine doped copper sulphate single crystal

For pure copper sulphate single crystal the band at  $3119 \text{ cm}^{-1}$  assigned to stretching vibration of O-H group [7]. The bending vibration mode of O-H group identified at  $1654 \text{ cm}^{-1}$ . The stretching vibration mode of S-O group found at  $1087 \text{ cm}^{-1}$ . The bending vibration mode of S-O group were identified at  $615 \text{ cm}^{-1}$ [8]. The vibration mode of metal ion  $\text{Cu}^{2+}$  may be observed at Cu-O-H vibration and is assigned to  $867 \text{ cm}^{-1}$ .

For L – Histidine doped copper sulphate single crystal the stretching and bending vibration mode of O-H group shifted to  $3126 \text{ cm}^{-1}$  and  $1657 \text{ cm}^{-1}$  respectively. The shifting stretching and bending vibration mode of S-O group observed at  $1083 \text{ cm}^{-1}$  and  $599 \text{ cm}^{-1}$ . Similar observations were previously reported by F. A. Miller and C. H. Wilkins for AR grade copper sulphate salt [9].

### 3.3 UV – Vis Spectral Analysis



**Fig -7:** UV – Visible absorption spectra of pure and 0.002 wt% L -Histidine doped copper sulphate single crystal

UV-Visible absorption spectra of pure and the L – Histidine doped copper sulphate crystals were recorded by using Systronics double beam spectrophotometer-2201 in the wavelength range 200 – 800 nm. The spectra were shown in figure (7).

From the UV-Visible absorption spectrum the absorbance is less than one unit in the entire visible region. The pure and L-Histidine doped copper sulphate crystals is very transparent in the wavelength range 320-600 nm. This is the essential property for NLO materials [2]. The UV cut-off wavelength of pure and L – Histidine doped copper sulphate crystals were almost same and is lies near 304 nm. The band gap value of copper sulphate crystal was found out using the relation  $E=h\nu$  and is found to be 4.08 eV. Similar results were reported by S.M. Delphine and etal. [10].

### 4. CONCLUSION

Single crystals of pure and L-Histidine doped copper sulphate were grown by slow evaporation method. The powder X-ray diffraction analysis revealed that the crystal belongs to triclinic system. Small variations in the lattice parameters for the doped crystal have been noted. The vibrational modes of functional groups were identified using FTIR spectroscopic technique. The UV-Vis spectral study highlights the excellent transparency of the crystal in the entire visible region.

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