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Studies on structural, thermal and optical characterization of glycine copper sulphate single crystal grown by slow evaporation method

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Abstract

The glycine copper sulphate single crystal was grown by solvent evaporation method using water as a solvent. A good quality crystal of dimension 20 × 10 × 3 mm^3 was harvested with well-defined morphology. The grown single crystal was characterized by powder XRD, thermal, UV-Visible and FTIR techniques. The structure of grown crystal was confirmed by XRD analysis and the thermal stability of grown crystal was found to be 240˚C from TG/DTA analysis. The UV-Visible absorption spectrum indicates the optical absorption cut off wavelength at 288 nm in the ultraviolet region and the corresponding band gap energy was found to be 4.30 eV. The FTIR study reveals the presence of functional groups in the investigated sample. The detailed investigation on the structural, thermal and optical properties of grown glycine copper sulphate crystal has been reported.

Keywords: Glycine Copper Sulphate, XRD, TGDTA, UV-Visible absorption, FTIR Analysis

1 Introduction

In recent years, semi-organic non-linear optical (NLO) single crystals are gaining momentum and also versatile in nature than their inorganic counterparts due to their large electro-optic co-efficient with low frequency dispersion and high non-linearity [1]. The major disadvantage of organic crystal for optical applications is poor mechanical strength [1,2]. The inorganic crystals have excellent mechanical and thermal properties but exhibits moderate NLO property. In order to overcome this issue, single crystals are grown with the combinations of organic and inorganic materials which have excellent mechanical as well as NLO properties compared to crystals originated from organic compounds [1-4]. Glycine contains 20 amino acids which are found to be difficult in dissolving aqueous solutions under ambient conditions [5]. Among the three crystalline phases namely α, β and γ found in glycine, the NLO characteristics is found to be absent in α glycine phase due to its centrosymmetric nature whereas the other two phases β and γ are non-centrosymmetric leading to NLO properties [6]. Single crystals grown from copper sulphate (chalcanthite) can be used as optical band pass filter in the visible range [7]. In the present investigation, the structural, thermal and optical properties are evaluated for the single crystal grown by slow evaporation technique taking proper combination of organic and inorganic compounds using aqueous solution. The XRD analysis reveals the triclinic structure of the grown crystal and lattice parameters are determined. The TGDTA study discloses the thermal stability and the UV-visible study confirms the absence of absorption in the visible region. This characteristic allows the grown crystal can be used as optical band pass filter applications. This behaviour has not been reported in the available literature.

2 Experimental

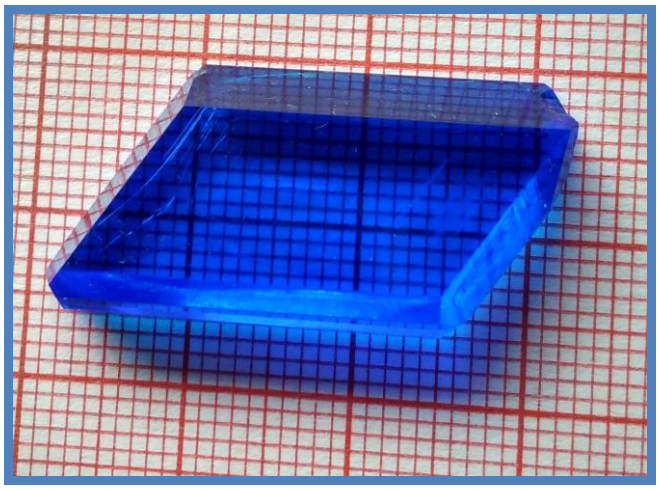

Fig.1 Grown Glycine Copper Sulphate Single crystal

The glycine copper sulphate single crystal is grown by solvent slow evaporation method using water as solvent. The glycine and copper sulphate (analytical grade reagents) chemicals are used as the starting materials with Merck products purity of 99.9%.

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The deionised water is used for the synthesis and growth of glycine copper sulphate single crystal. The glycine and $CuSO_4$ chemicals are weighed in the ratio of 3.75 g: 7.95 g and taken in a cleaned borosilicate glass beaker with 50 ml capacity of deionised water. Then the solution is stirred until homogeneous solution has been obtained and filtered out using Whatmann filter paper. The clean and transparent solution is left to evaporate at room temperature in a beaker covered with a silver foil which has pores for the evaporation of water molecules. After a certain time period the entire solution is completely transformed into a crystal with optically transparent and well defined morphology of dimensions of 20×10×3 mm^3 is harvested. The grown crystal is depicted in Fig. 1.

3 Results and Discussion

The structure of the grown crystal is studied by powder X-ray diffraction measurement using PANalytical X- ray diffractometer with characteristic of Cu K_α (λ = 1.54060Å) radiation from 10˚ to 60˚ and minimum step size of 0.001˚. Fig.2 shows the powder XRD pattern of the grown glycine copper sulphate crystal. The high intensity of diffraction peaks confirms that the grown crystal has good crystalline nature. The obtained diffraction peaks are indexed by JCPDS. The strong diffraction planes (111), (001) and (111) indicates that the grown crystal has triclinic crystal structure. The lattice parameters are found to be *a* = 5.95(8), *b* = 6.12(1) and *c* = 10.74(3) Å. The indexed XRD planes and lattice parameters are compared with the existing literatures of Glycine [8], copper sulphate pentahyderate [9] and Glycine copper sulphate crystals [10]. The reported lattice parameter values are found to be in good agreement with the existing literature [8-10].

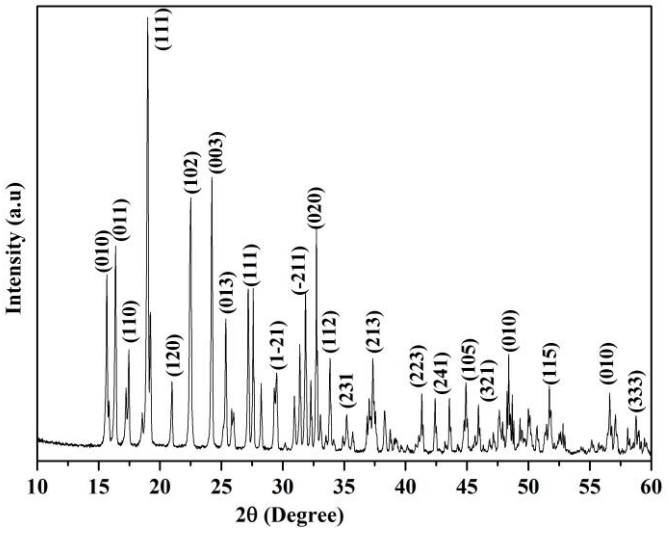

Fig.2 Powder XRD analysis of glycine copper sul

Thermal behaviour of the grown crystal is studied by thermogravimetric (TG) and differential thermal (DTA) analysis using SDT Q600V 8.3 build 101 simultaneous DTA/TGA analyzer in nitrogen atmosphere.

The sample is heated up to a temperature of 450˚C at the rate of 20˚C/min. Fig.3 shows the TGDTA curve of glycine copper sulphate single crystal. The grown crystal has two stages of decomposition which have been determined from the TGA curve. During the first stage the maximum weight loss of studied sample occurs at 100˚C which is attributed to the removal of the water (H_2O) molecule from the sample. In the second stage of TG curve, the weight loss occurs at 225˚C due to the complete decomposition of the compound. The total weight loss up to 75% is observed in the present investigated sample. The DTA curve shows the strong endothermic peak in the temperature range 70˚-130˚C and maximum peak at 100˚C which corresponds to the decomposition of glycine copper sulphate crystal. The second endothermic peak in the temperature range 230˚- 260˚C is due to the sharp melting of the investigated sample.

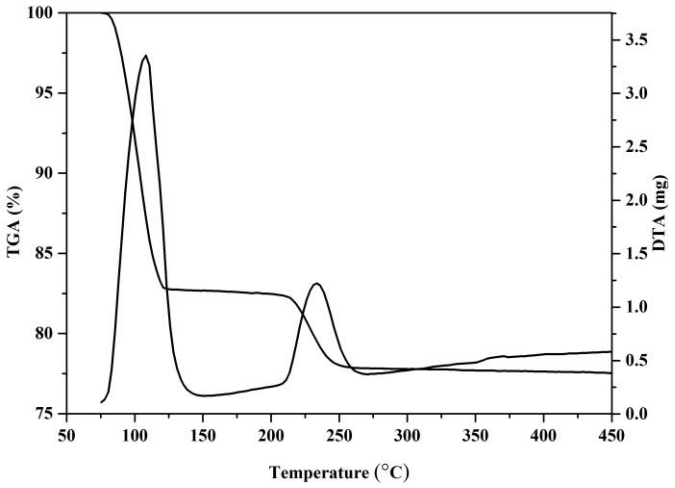

Fig.3 TGDTA curve of Glycine Copper Sulphate single crystal

Fig.4 shows the UV visible absorption and Fig.5 depicts the transmission spectrum of glycine copper sulphate single crystal. The optical absorption spectrum has been recorded from 200 nm to 800 nm using a UV-VIS-NIR Perkin Elmer spectrophotometer. From the UV-visible spectrum, it is observed that the absorbance is less than 1 unit in the entire visible region. Interestingly, it is observed that the optical absorption begins to decrease at 600 nm and there is a transparent region in between 500 nm and 350 nm. Further, the absorption starts to increase in the region of 350 to 200 nm. Moreover, the optical transmittance spectrum as displayed in Fig.5, 85% of transmittance takes place within the range of 400 to 500 nm. This can be attributed to the highly transparent region in the visible range. This observed result confirms the potential of the grown crystal as an efficient NLO material [6] and the crystal can be utilised as an optical band pass filter in the visible spectrum [7,12]. The optical absorption cut off wavelength is found to be 288 nm and the band gap energy is evaluated using the following relation ,

$$E = 1.2/\lambda,$$

where, λ is the fundamental absorption wavelength. The estimated band gap energy is 4.305 eV (288 nm) which is in good agreement with the band gap energies (3.7 - 4.67 eV) reported for single crystals grown from glycine with other metals [12].

Fig.6 shows the FTIR spectrum of grown Glycine Copper Sulphate single crystal. The peak observed at 607 cm^{-1} is due to vibration of amino group and the peak at 658 cm^{-1} indicates the presence of carboxylate group. The bending of COO gives a peak at 735 cm^{-1} and the C-C-N asymmetric stretching is evident from the medium intensity peak at 987 cm^{-1}. The band at 1162-1206 cm^{-1} is due to the N-H group and this confirms the existence of glycine in zwitterionic form. The strong absorption band observed at 1625 cm^{-1}. The peak at 2893 cm^{-1} and 2920 cm^{-1} are assigned to the C-stretching vibrational modes and the O-H stretching vibrational mode present at 3595 cm^{-1} is due to water of crystallization.

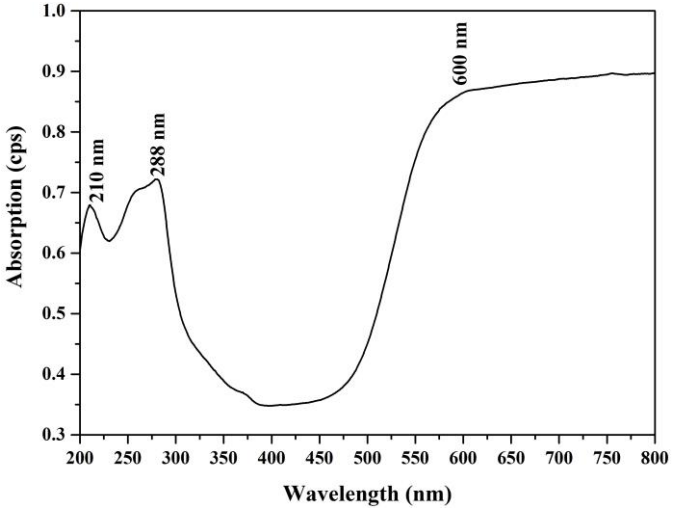

Fig.4 UV-Visible absorption study of Glycine copper sulphate crystal

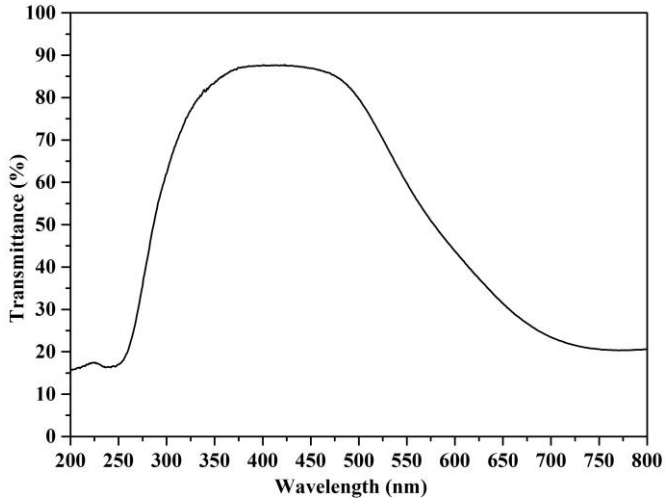

Fig.5 UV-Visible transmission study of grown glycine copper sulphate single crystal

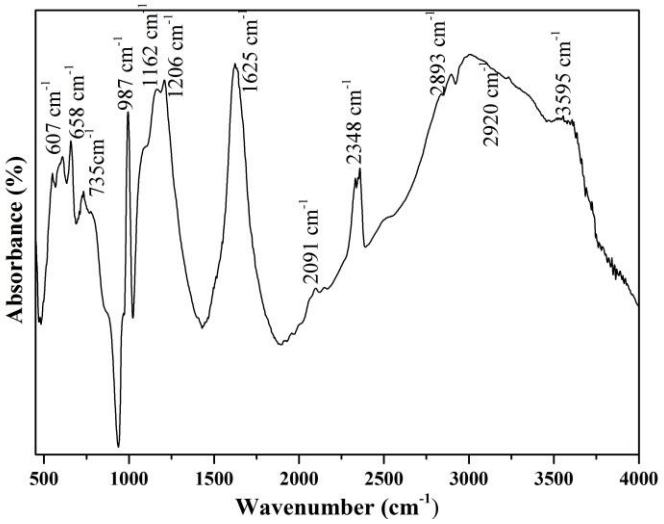

Fig.6 FTIR spectrum of grown Glycine Copper Sulphate single crystal

4 Conclusions

The glycine copper sulphate single crystal is successfully grown by slow evaporation technique with water as a solvent. The grown crystal belongs to triclinic structure and lattice parameters values are determined. The TG/DTA study reveals that the sample is stable up to a 240˚C and strong endothermic peak observed at 100˚C indicates the decomposition of the sample. The UV visible absorption spectrum shows an absorption band at 288 nm with optical band gap energy of 4.305 eV (288 nm). The 85% transmittance spectrum observed in the visible region confirms that the grown glycine copper sulphate single crystal can be utilized in optical band pass filter related applications.

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