SYNTHESIS AND CHARACTERIZATION OF COPPER SULPHATE CRYSTALS WITH GLYCINE

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ABSTRACT

Copper Sulphate single crystals, doped with Glycine were grown using slow evaporation method from an aqueous solution. The prepared crystals were analyzed with UV-visible spectroscopy and FTIR. The UV visible spectrum was utilized to estimate the optical transparency of the grown crystals. The UV spectrum indicated low absorbance in the visible region and high absorbance in the UV region, especially in 200nm to 290nm. The Energy gap (Eg) of crystals was observed to decrease from 3.77 eV to 2.49 eV when 5gm of Glycine was added in a ratio with Copper Sulphate. The FTIR studies confirmed the presence of various functional groups in the prepared crystals.

Key words: Slow evaporation, FTIR, UV, Energy gap, Tauc plot

1.INTRODUCTION

All naturally occurring crystals have been synthesized successfully in the laboratories using advanced crystal growth techniques. The growth characteristics of crystals vary, based on their physical and chemical properties including solubility, melting point, decomposition, phase change, etc. Crystal growth is the basis for various technological advancements. The control of crystal habits during the crystallization steps constitutes an important industrial challenge since the shape of a crystal affects solid-liquid separation characteristics, packaging, handling, drying, storage behaviors and end-use properties of the crystallized material. A change in solvent composition or the introduction of a small quantity of an additive is generally used to modify crystal habits.

In recent years, Amino acids and their complexes have been widely used for many applications to enhance the chemical stability, laser damage threshold, thermal physical properties and linear and non-linear optical properties [1]. Glycine (NH_2-CH_2-COOH) is the simplest amino acid. Unlike other amino acids, it has no asymmetric carbon atom and is optically inactive [2]. It has three polymeric crystalline forms α, β and γ. The α form of Glycine crystallizes in centro symmetric structure. Hence it does not exhibit second harmonic generation, whereas β and γ form crystallize in non- Centro symmetric crystal structure. The β form of Glycine is unstable. The γ form of Glycine exhibits a second harmonic generation. The Glycine molecule can exist in zwitterion form and hence it can form compounds. Some complexes of Glycine with HCl, $LiSO4_4$, H_2SO_4, H_3PO_3, $CaNO_3$, $CoBr_2$ form single crystals. Glycine (NH_2-CH_2-COOH) is the simplest amino acid. Unlike other amino acids, it has no asymmetric carbon atom and is optically inactive. In this paper, we present our findings on the growth of novel semi-organic Glycine Copper Sulphate (GCS) crystals, along with pure Copper Sulphate crystals using Fourier Transform Infrared Spectroscopy (FTIR) and UV-Visible Near IR analysis.

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2.Sample Preparation

Sample I

In the growth of any crystal, the selection of raw materials plays an important role. We have chosen copper sulphate and Glycine as the components.

To prepare pure Copper Sulphate crystals, anhydrous Copper Sulphate was dissolved in double-distilled water. The slow evaporation method was used for crystal growth. The 5gm of Anhydrous Copper Sulphate diluted with 10ml of Luke warm water. Stirred continuously until it dissolves completely. The solution was allowed to rest. After several days, the water evaporated, and the resulting transparent pure Copper Sulphate crystals (CSP) were collected and dried again for a couple of days. Finally grinded to the form the fine powders.

Sample II

The sample consisted of a mixture of 5gms of Glycine and Copper Sulphate dissolved in 10 ml of Lukewarm water. The solution was placed on the magnetic stirrer to ensure the complete dissolution of the powders. Using slow evaporation, crystals were formed within 10 days. The resulting crystals were then powdered, and the sample was sent for UV and FTIR studies.

3.Results And Discussion

3.1. UV VISIBLE SPECTRAL ANALYSIS

3.1.1. COPPER SULPHATE CRYSTAL(CSP)

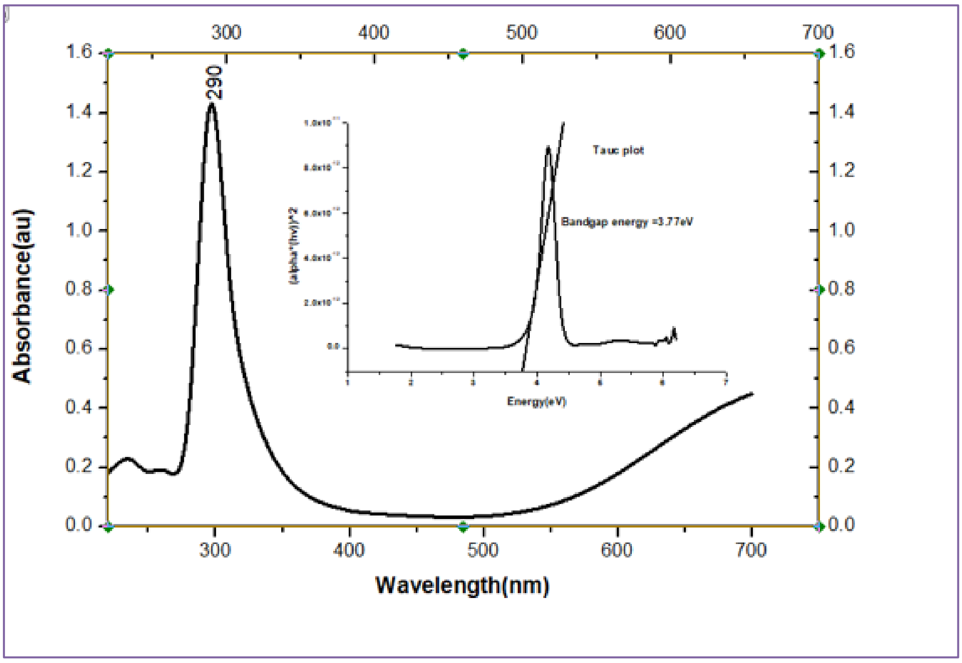
The UV-Visible absorption spectra of (CSP) crystals were recorded by using 'UV–Visible 1800 Double beam spectrophotometer' in the range of 200-800 nm as shown in Fig 11.

The absorbance of the sample increases as the wavelength increases in the range of (280nm-300nm) then decreases as the wavelength increases further nearly stable in the range of (400-500nm).

Figure 1: UV-Vis spectrum of Copper Sulphate crystal

The spectra show low absorbance in the visible region(300 to 700nm) and high absorbance in the infrared region(700 to 3300nm) and the ultraviolet region (200-290nm) this is in agreement with the results reported by Anne et al. [3]. The CSP crystals were transparent in the wavelength range between 300 nm and 530 nm, and they can be used as optical bandpass filters in this range. This result agrees with the results reported by Manomenova et al. [4] and Anne et al. [3]. The pure Copper Sulphate sample shows a peak in 290 to 340 nm(297nm) because of Jahn Teller effect. The Jahn Teller effect affects molecules with degenerate orbitals. In such a case, the molecule will distort in such a way that removes the degeneracy. The distortion in $CuSO_4.5H_2O$ involves the elongation of two Cu-O bonds and the shortening of other four. This distortion lowers the energy of the system by removing the electronic degeneracy, leading to a more stable system.

The indirect band gap energy was determined by Tauc plot by plotting $(\alpha hv)^2$ vs hv as shown in the graph and it found to be 3.77eV.



3.1.2. GLYCINE COPPER SULPHATE CRYSTAL

The previous studies indicated that the UV absorbance spectrum of Glycine is approximately between 240nm to 600 nm (264nm). The lower cutoff wavelength indicates the intrinsic property of glycine.

When an equal amount of Glycine is added to the Copper Sulphate, peak shift is observed from 297nm to 302 nm . A new peak appeared at 264 nm which indicates the presence of Glycine. According to theoretical predictions, the electronic absorption spectra of Glycine aqueous solution are mainly characterized in the near- ultraviolet or visible region . Here the observed data matches with the theoretical aspect. In the case of pure $CuSO_4$, a peak was initially observed at 297nm. Due to the interaction of glycine with copper, this peak shifted to 302 nm [5,6].

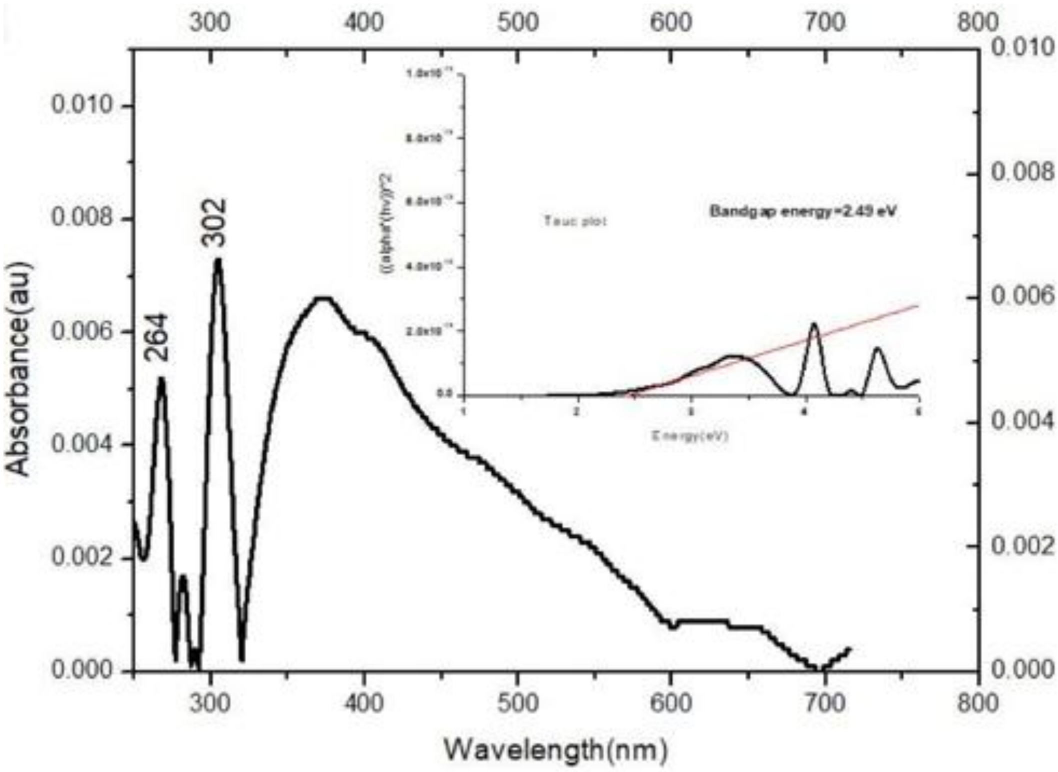


Figure 2: UV-Vis spectrum of Glycine Copper Sulphate crystal

The band gap energy was determined using Tauc plot $(\alpha hv)^2$ vs hv as shown in the graph and it found to be 2.49 eV. There will be a single linear region in direct transition and two linear portions in indirect transition. The Tauc's plot for GCS crystal has a single linear region, hence it corresponds to direct optical transition[7]. Because of the wide band gap the grown crystal has large transmittance in the visible region. Generally, Glycine reduces the bandgap energy. Here we observed, bandgap energy reduces from 3.77 eV to 2.49 eV by applying glycine.

4.FTIR SPECTRAL ANALYSIS

4.1. COPPER SULPHATE CRYSTAL

Fourier transform infrared spectroscopy (FTIR) is valuable tool for examining the molecular structure of chemical compounds.Each chemical compounds have distinct IR spectrum.The grown Copper Sulphate crystal was charcterized by Perkin Elmer spectrometer in the range of 500cm^{-1} .The$CuSO_4.5H_2O$ spectrum showed characteristic peaks at 2934, 1700 and 1515cm^{-1},indicating O-H stretching and absorption bands, though 2900 cm^{-1} ,peak appeared in less intense. The peaks at lower band values are associated with the vibration between O and H bonding. The bending vibration of O-H appeared at 1700cm^{-1}and the stretching vibration of S-O group appeared at 1515cm^{-1}.

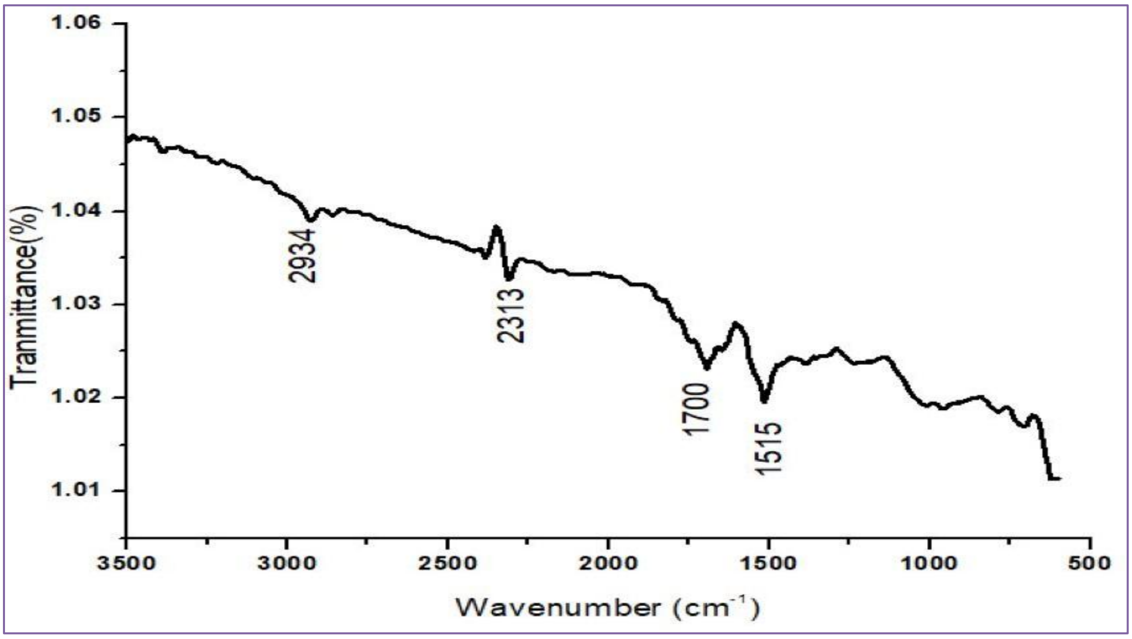


Figure 4: FTIR Spectrum for Copper Sulphate crystal

4.2. GLYCINE COPPER SULPHATE CRYSTAL

The N-H stretching at 3746 cm^{-1} is intense and broad. The spectrum shows that the C-H stretching occurred at 2982cm^{-1} and 2315cm^{-1}, while C=O stretch at 1685cm^{-1} , it was intense and sharp. In glycine, carboxyl group forms a carboxylate ion, and the amino group becomes an ammonium ion[7]. The absorption peaks in 1124cm^{-1} and 929cm^{-1} were shifted to 1060cm^{-1} and 950cm^{-1}.

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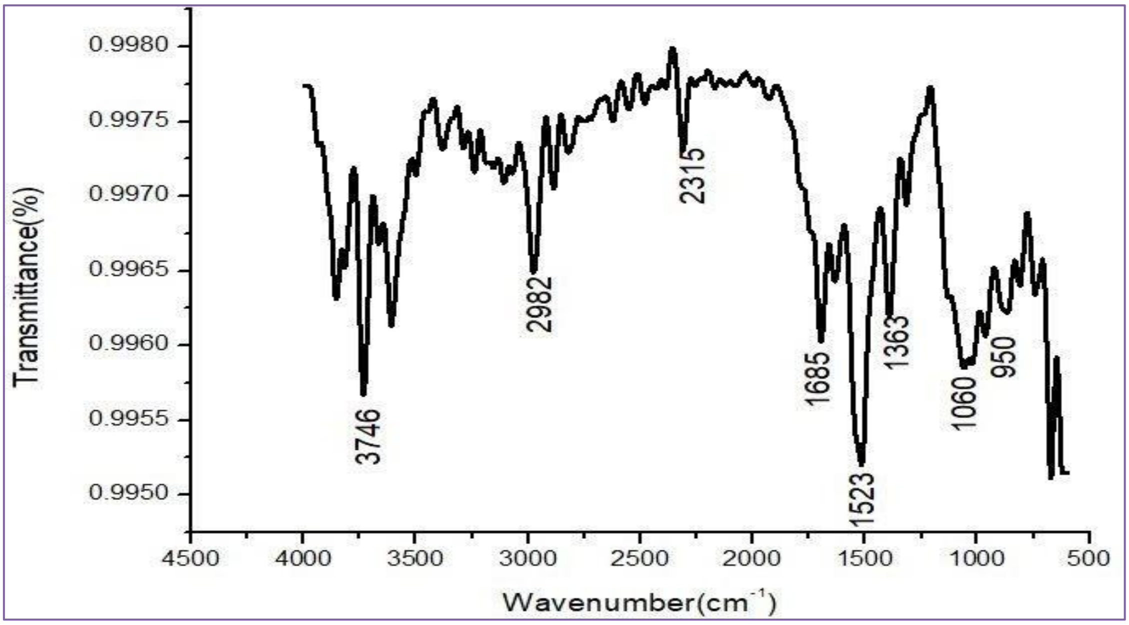

Figure 4: FTIR Spectrum for Glycine Copper Sulphate crystal

Vibrational group assignment of GCS crystal.

Wavenumber(cm^{-1})	Vibrational groups
3746	N-H stretching
2982	C-H stretching
2315	C-H stretching
1685	C=O stretching
1523	C=O symmetric stretching
1363	C-N symmetric stretching 1060
	NH_2 Wagging
950	S-O stretching

5. Conclusion

The Copper Sulphate & Glycine doped crystals were grown from the aqueous solution by the method of slow evaporation at room temperature. Good quality crystals were formed within 2 weeks (14 days). The UV-Vis spectral study of Copper Sulphate crystals highlights the excellent transparency of the crystal in the entire visible region. The band gap energy was found to be 3.77eV. CSP crystal can be used as optical bandpass filters. The vibrational modes of functional groups were identified using FTIR spectroscopic studies of Copper Sulphate crystals.

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