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Structural, Optical And Functional Group Analysis Of Ammonium Sulphate Doped L-Glutamic Acid Hydrochloride Single Crystal

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A novel Ammonium Sulphate-doped L-Glutamic acid Hydrochloride crystal was synthesized using a renowned method of slow evaporation technique. The synthesized crystal was analyzed by some characterization methods, such as XRD, Optical analysis, Scanning Electron Microscope (SEM) Analysis; The XRD analysis indubitably discovers its structure and space group. The title compound has well transmission in the visible region and an energy band gap was calculated. The functional group was ascertained from Fourier Transform Infra Red (FT-IR) analysis. Eventually, the morphology of the grown crystal was studied by SEM Analysis.

Keywords: L-Glutamic acid Hydrochloride`, Structural, XRD, FT-IR.

1. Introduction:

Amino acids are a collection of 20 different molecules used to build proteins. Proteins contain one or more chains of amino acids that are of enormous biological importance and are necessary for human life [1]. Lysine and glutamic acid are biologically important organic compounds [2-3]. L-Glutamic acid (LGlu) is an amino acid widely used in pharmaceutical and food industry. Amino acids based on several semi-organic series crystals are crystallized recently and their various properties have been analyzed [4-7]. L-Glutamic acid is a phase matchable NLO material that has high transparency in the UV region [8]. Already reported that the different concentration of (0.01/100ml, 0.04/100ml and 0.08/100ml) L-Glutamic acid single crystal in the presence of ammonium sulphate [9].

In this present investigation illustrates the growth and characterization of (0.02/50 ml) of Ammonium Sulphate Doped L-Glutamic acid Hydrochloride Single crystal by slow evaporation solution growth technique. This grown crystal was analyzed with X-ray Diffraction, FT-IR, UV-Vis and SEM characterization.

2. Experimental procedure:

2.1 Synthesis:

In the inception stage required amount of the sample L-Glutamic acid Hydrochloride (0.98) and 25 ml of Hydrochloric acid (HCl) taken and mixed aid of stirring 2 hours. Then (0.02) Ammonium Sulphate was blended to that preceding solution. After that this aqueous solution was dissolved in 50 ml De-ionized water again this mixture was persistently stirred for furthermore 1 hour, in the ensuing stages which solution was filtered using Whatsmann filter paper for removing impurities. This filtered solution was placed in the undisturbed place for evaporation. After 17 days we get the title compound of Ammonium Sulphate Doped L-Glutamic acid Hydrochloride Single crystal. Thereafter, the title compound was stored in air tight container for further characterization. The photograph of Ammonium Sulphate Doped L-Glutamic acid Hydrochloride Single crystal was shown in Figure 1.

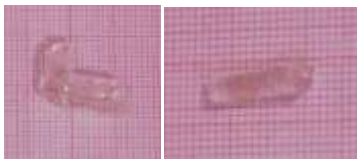

Figure 1: The photograph of Ammonium Sulphate Doped L-Glutamic acid Hydrochloride Single crystal

2.2. Characterization Studies:

The powder X-ray diffraction pattern of the grown crystal was recorded on the Xpert^3 powder X-ray diffractometer using CuKα (λ=1.54060 A^0) radiation. The transmission spectrum of the grown crystal was recorded by a Perkin Elmer (Lamda 35) UV-Vis Spectrometer. Using a Jasco Spectrometer (FT-IR model -4700) with a scanning speed of 2mm/sec, FT-IR spectrum was recorded, and various functional groups were discovered. Grown crystal morphology was quantitatively analyzed by using a JEOL-JSM –IT 200 Scanning Electron Microscope (SEM).

3. Results and Discussion

3.1 Powder X-Ray Diffraction (PXRD) Analysis:

The PXRD study confirmed its crystallographic structure and space group. The entitled compound was powdered and put through into the powder diffractometer and which was scanned, range 2θ from 0^o to 80^o with a scan rate of about 1^o/min. The obtained data were compared to JCPDS no.25-1925 and which peaks were indexed as shown in figure 2.

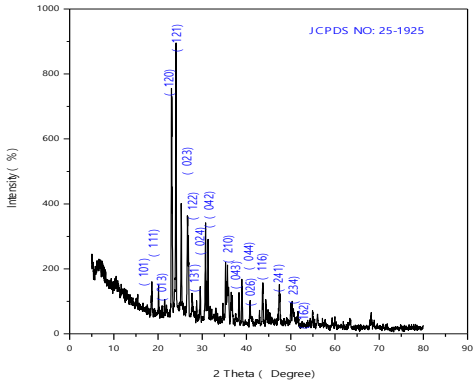

Figure 2: The recorded PXRD of Ammonium Sulphate Doped L-Glutamic acid Hydrochloride Crystal

From the PXRD pattern, we observed that the entitled compound belonged to orthorhombic structure and the $p2_12_12_1$ space group. The estimated lattice parameters are tabulated in table 1. The prominent diffraction peaks were located at 2θ, which harmonies the plane.

Table 1: The estimated lattice parameters of Ammonium Sulphate Doped L-Glutamic acid Hydrochloride Crystal

Cell Parameters	Ammonium Sulphate doped L-Glutamic acid hydrochloride crystal
a(A^o)	5.6399
b(A^o)	11.2866
c(A^o)	12.5935
α =β =γ	90°
Crystal system	Orthorhombic
Space group	p212121
Volume (A^{o3})	801.6429

The estimated lattice parameter values are slightly varied to intrinsic L-Glutamic acid hydrochloride crystal, and volume has changed due to the changes in lattice parameters.

3.2 UV- Visible Analysis

Figure 3 shows the transmission spectrum of Ammonium Sulphate Doped L-Glutamic acid Hydrochloride Crystal. It is discerned that the lower cut-off wavelength discovered in the 277 nm is about 80% respectively and there is no significant absorption in the whole region of the spectra. This lower absorption behavior in the region above 277 nm to 1100 nm manifests that the entitled compound was propitious for the second harmonic generation of Nd: YAG laser of wavelength λ = 1064 nm [10].

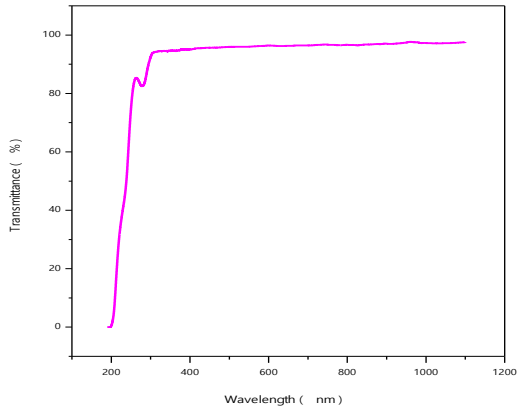

Figure 3: The transmission spectrum of Ammonium Sulphate Doped L-Glutamic acid Hydrochloride Crystal

In figure 4, the energy band gap of the grown crystal was determined to be about E_g= 5.6 eV. This high-energy band gap is needed for optical applications. According to Tauc's relationship, the optical energy band gap and the absorption coefficient (α) were calculated and is given by,

$$\alpha = \frac{2.303}{t}\log\frac{1}{T} \quad (3)$$

Where T is the transmittance and t is the thickness of the crystal.

$$\alpha h\nu = A(h\nu - Eg)^n \quad (4)$$

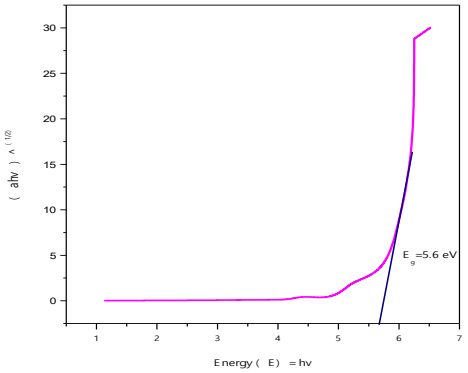

Figure 4: Band gap estimation of Ammonium Sulphate doped LGH crystal using Tauc plot

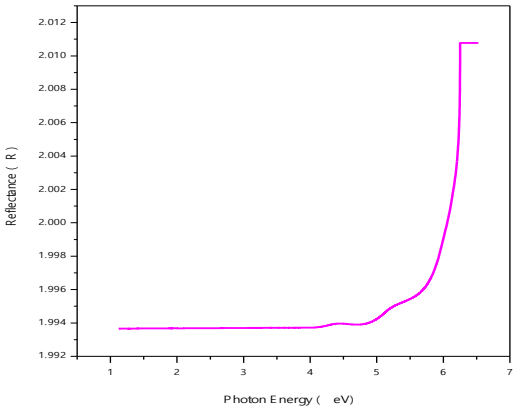

Figure 5: Reflectance Spectrum of use full compound

The skin depth of grown crystal was decreases with an increase in photon energy, which is clearly seen in figure 8. Due to the propagation of the electromagnetic wave, the absorption loss (K) is exhibited in figure 6 and this shows that K is not constant, although even now robust on the frequency of the light. The given formulas are used to quantify Skin depth (δ) and Extinction coefficient (K).

$$K = \frac{\lambda\alpha}{4\pi} \longrightarrow (1)$$

and skin depth (δ) is,

$$\delta = \frac{1}{\alpha} \longrightarrow (2)$$

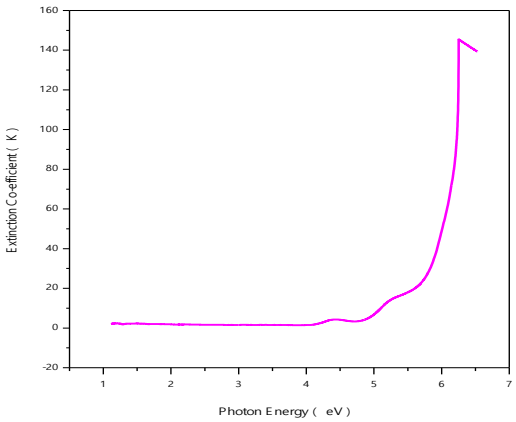

Figure 6: Extinction Co-efficient of grown crystal

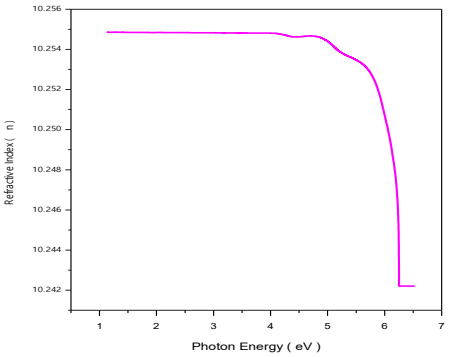

Figure 7: Refractive index of grown crystal

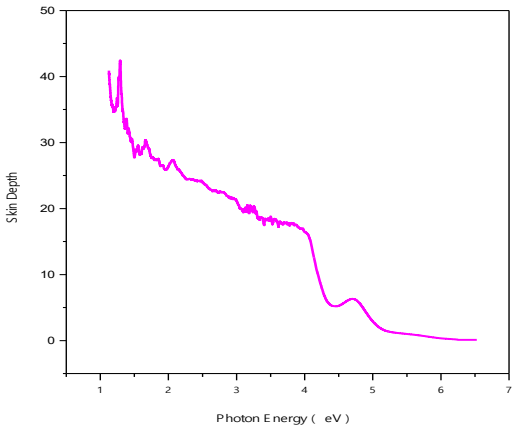

Figure 8: Skin depth of grown crystal

The reflectance (R) of the entitled compound was calculated using equation 5 and the variation of reflections with photon energy is shown in the figure5. The reflectance absolutely depends on photon energy.

$$R = 1 \pm \frac{1-\sqrt{1-\exp(-\alpha t)+\exp(\alpha t)}}{1+\exp(-\alpha t)} \longrightarrow \quad (5)$$

3.3 Fourier Transform Infra-red Spectroscopy (FT-IR) Analysis:

Figure 9 shows the recorded FT-IR of entitled compound of Ammonium Sulphate doped LGH crystal. This present study apparently explained that it's possible stretching and bending vibration modes. Some viable stretching modes are occur, such as C=O, C-O, O-H, N-H and C-H whereas the N-H, C-H, and O-H were the feasible bending vibration modes. These possible stretching and bending vibration modes corroborate its molecular structure. Moreover, it's possible interaction betwixt the carboxylic group and the amine group of the neighboring molecules is in the crystal structure. The C=O vibrations of stretching show an

absorption peak at 1719.23 cm^{-1} and the C=O vibrations of stretching occur at 1371.14 cm^{-1}, 1321 cm^{-1}, 1272.79 cm^{-1}, 1252.54 cm^{-1}, 1206.26 cm^{-1}, 1144.54 cm^{-1}. The peak was observed at 3137.61 cm^{-1}, 3013.23 cm^{-1}, corresponding to O-H Stretching mode of vibrations. N-H stretching and bending vibrations modes were occurs at 2813.63 cm^{-1},2854.13 cm^{-1},2643.93 cm^{-1} and 1608.34 cm^{-1}, 1504.2 cm^{-1}. C-H stretching vibration modes occur at 2854.13 cm^{-1}, 2813.63 cm^{-1}. Similarly, the C-H of bending vibration modes occurs at 1422.24 cm^{-1}. The prominent peaks ensure that the carboxylic group and the amine group of the L- Glutamic acid are in the crystal structures.

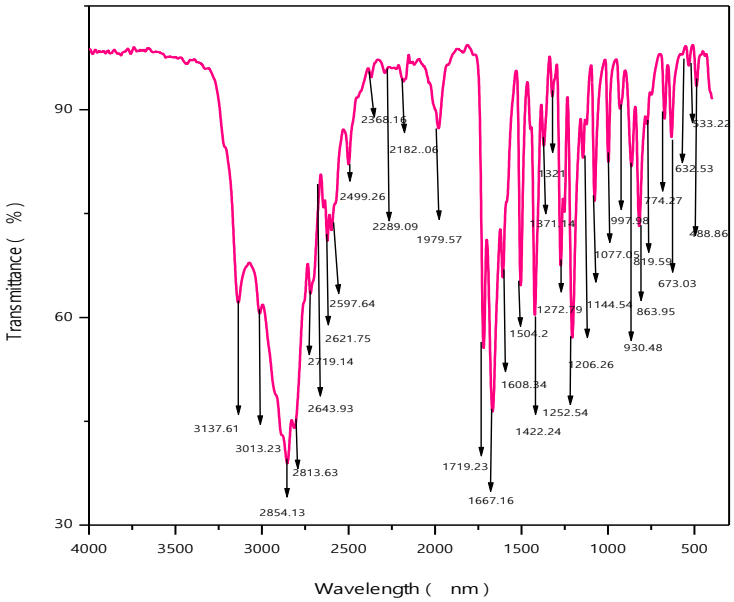

Figure 9: the recorded FT-IR of entitled compound of Ammonium Sulphate doped LGH crystal.

When L-Glutamic acid is doped with ammonium sulphate, around 3300-3000 cm^{-1} for N-H stretching is observed due to the presence of ammonium ion (NH_{4+}) ion. The same way, around 1400-1100 cm^{-1} for S-O stretching is due to the presence of Sulphate ion. Likewise, an ammonium ion being disclosed the vibrations occurred 3137.61 cm^{-1}. S-O stretching vibrations were observed at 1422.24 cm^{-1}, 1144.54 $cm^{-1,}$ which divulges the Sulphate ions. A few more functional group assignments of Ammonium Sulphate doped LGH crystal as shown in Table 2.

Table 2: FTIR functional group assignment of Ammonium Sulphate doped LGH crystal

Mode of vibrations	Standard reference [11,12] Wavenumber (cm^{-1})	Ammonium Sulphate doped LGH (Wavenumber) cm^{-1}
O-H Stretching	3400-2400	3137.61,3013.23
N-H Stretching	3500-3100	2813.63,2854.13,2643.93
C-H Stretching	3000-2850	2854.13,2813.63
C=O Stretching	1725-1700	1719.23
N-H bending	1640-1550	1608.34, 1504.2
C-H bending, O-H Plane	1465	1422.24
C-O Stretching	1300-1000	1371.14, 1321, 1272.79, 1252.54, 1206.26, 1144.54,
O-H bending	930	930.485
C-C Stretching	871	863.953
O-H in Plane deformation	812	819.598
COO- Wagging	538	533.221
S-O Stretching	1400-1100	1422.24,1144.54

3.4 Scanning Electron Microscope (SEM) Analysis:

The titular compound of the grown crystal's surface morphological features was assayed by SEM it different magnifications and its upshots were shown in 10. From the figure, we discern that the particle size and shape were assorted. The particle size was disparate from 10 µm to 5µm. Figure 10 (a) shows that the irregular surface morphology as well as Figure 10 (b) portrays that the elliptical and irregular surface morphology. Also, Figure 10 (a) was smoother than the other three (b), (c) and (d) Figure's.

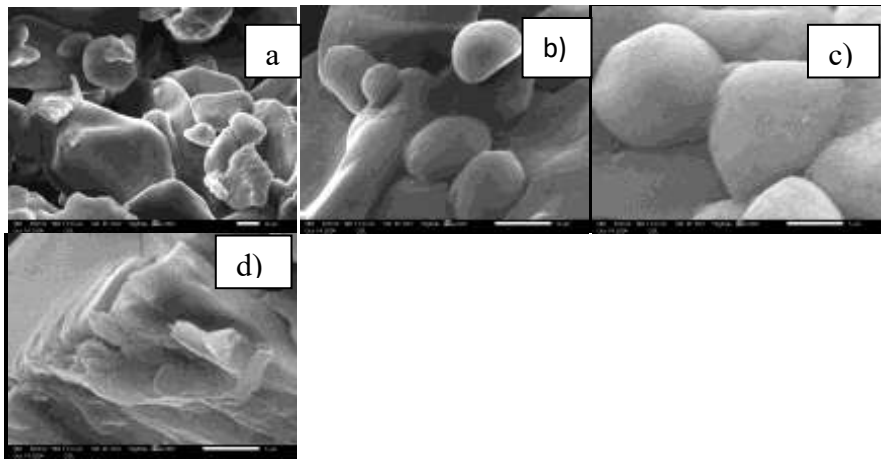

Figure 10: SEM image of the different magnification of Ammonium Sulphate LGH crystal

The spherical shape of the morphology was depicted in Figure 10 (c). Similarly, the irregular rock shape was illustrated in figure 10 (d). Uniquely, figure 10 (d) discloses the rough morphology. Moreover, we noticed that the tiny particles were jointly together to produce an enormous morphology. An additional observation is that from figures 10 (a) and (b) had a same particle size as 10µm. Likely, Figures 10 (c) and (d) had a particle size of 5 µm. Hence, the crystalline nature of the grown crystal portrays those different sizes and various morphology likely smooth and rough surfaces.

4. Conclusion:

A new semi-organic material of L-Glutamic acid doped Ammonium sulphate crystal was grown using a renowned method also its structure was confirmed by XRD analysis. Various functional groups were discerned by FT-IR study. The titular material was 80% transmittance in the UV-Visible region and was identified by the UV-Visible Spectrum. Also, energy band gap was estimated. The surface morphological characteristics were discovered by SEM Analysis.

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