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Synthesis, Growth and Characterization of L-Lysine Nickel Maleate Crystal

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Abstract: ***A good quality, single crystal L-Lysine Nickel Maleate (L-LNM) has been grown by slow evaporation solution growth technique. The crystalline nature of the grown crystal has been analyzed by powder X-ray diffraction analysis. The presence of elements in the compound has been identified by EDAX analysis. FTIR spectrum confirms the presence of functional groups in the grown crystal. The optical nature of the grown crystal has been studied byUV-Visible spectrum. The thermal stability and decomposition stages of the grown crystal were studied by TG-DTA analysis***.

Keywords: Nickel nitrate and Maleic acid, Slow evaporation solution growth, FTIR and Optical studies, Thermal Studies, EDAX Analysis

1. Introduction

Crystal growth is a frontier area of science and technology which plays a major role in modern technology and photonics. Research on organic and inorganic functionalized non linear material plays a crucial role because of their molecular interactions, bond strength, high molecular polarizability, easy incorporation ofions in the crystal lattice [1, 2].Slow evaporation method yields small size crystals with different crystallographic faces [3].Amino acid is an organic compound that contains both amine and carboxyl functional groups and one can produce outstanding materials with the help of organic and inorganic counterparts[4]. Crystalline salts of optically active amino acids have been widely studied in various applications such as NLO applications[5, 6].Amino acids are used as the dopants and they enhance the material properties like nonlinear optical and ferroelectric properties [7,8].L-Lysine monohydrochloride dihydrate is a potential material to produce semi organic crystals [9].On consideration of the above ideas an attempt has been made to growL-Lysine Nickel Maleate crystals by slow evaporation solution growth method. The structural, spectral, thermal and optical properties of the grown crystal have been studied using different instrumentation techniques.

2. Experimental Procedure

Synthesis and Crystal Growth

L-Lysine Nickel Maleate crystals were grown, from aqueous solution by slow evaporation solution growth technique. TheFig.2 Powder XRD pattern of L-Lysine Nickel Maleate crystalsaturated solution of L-LNM was prepared by dissolving L-Lysine, Nickel nitrate and Maleic acid in distilled water at room temperature. The saturated solution was filtered and then closed with a pin holed polythene paper and I was kept in a undisturbed position. Within a period of 21 days a good quality of L-Lysine Nickel Maleate crystals were harvested from the mote solution. The photograph of as- grown L-LNM crystalis shown in Fig.1.

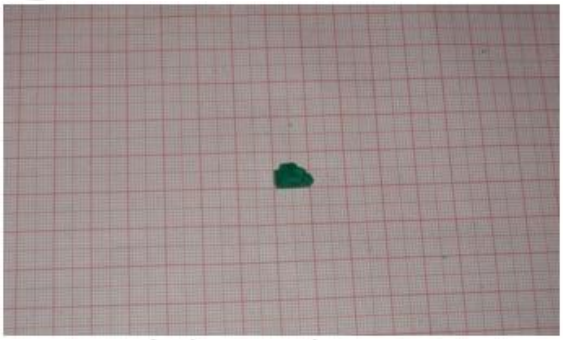

Figure 1: Photograph of as- grown L-LNM crystal

3. Characterization Techniques

3.1 Powder X-Ray Diffraction studies

Powder XRD is useful for confirming the identity of a solid material and determining crystallinity and phase purity. The grown sample was subjected to X-Ray Powder diffraction method using DIMAX ULTIMA-III with $\lambda = 1.5406A^{\circ}$ radiation. The observed powder XRD pattern ofL-Lysine Nickel Maleate crystal is shown in Fig.2.

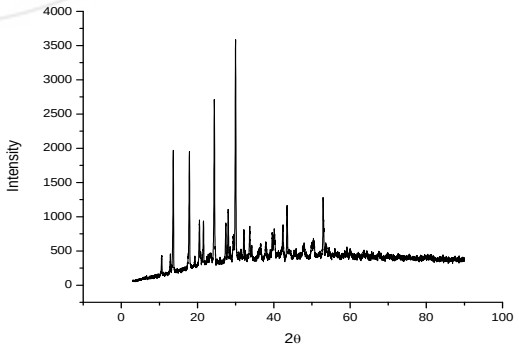

Figure 2: Powder XRD pattern of L-Lysine Nickel Maleate crystal

Table 1: Powder XRD data for L-Lysine Nickel Maleate crystal

2theta (deg)	FWHM (deg)	FWHM (Radians)	Grain Size(nm)	Lattice strain
29.956	0.182	0.003174	0.531	0.065275
24.4	0.174	0.003035	0.6518	0.053175
17.87	0.188	0.003279	0.8899	0.03894
13.636	0.189	0.003297	1.166	0.029725
52.921	0.158	0.002756	0.3005	0.11535
43.462	0.153	0.002669	0.36584	0.09475

3.2 EDAX Analysis

Elemental Dispersive Analysis by X-rays (EDAX) is used for the quantitative analysis of metal element. Fig. 3 shows EDAX spectrum of L-Lysine Nickel Maleate crystal which confirms the presence of Ni in the grown crystal.

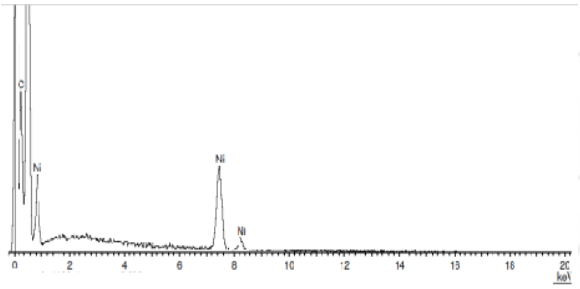

Figure 3: EDAX spectrum of L-Lysine Nickel Maleate crystal

3.3 FTIR Spectral studies

FT-IR spectral study is used to confirm the presence of functional group in the crystals. The IR spectral analysis was carried out to understand the chemical bonding and it provides useful information regarding the molecular structure of the compound. The FT-IR spectrum recorded for L-Lysine Nickel Maleate crystal is shown in Fig.4.The peak observed at3392cm^{-1} is due to N-H stretching vibration. The peak at 715 cm^{-1} indicates the presence of N-H bending. The peak corresponding to 770 cm^{-1} indicates NH_2 amine group. C-O Stretching (carboxylic acids) appears at 1222 cm^{-1} and 2580 cm^{-1}. The peak at 1536 cm^{-1} indicates the presence of NH_3^+ bending. Combination C-H stretching appears at 2580cm^{-1}.The presence of NH_3^+group confirms the protanation of amino groupL-Lysine in theL-Lysine doped Nickel Maleate crystal.

Table 2: Functional Groups of L-Lysine Nickel Maleate Crystal

Wave number (cm^{-1})	*Assignment*
715	N-H Bending(peptide)
770	NH_2 Wagging & Twisting(Amine)
1222	C-O Stretching(Carboxylic group)
1536	NH_3+ Bending
2359	Combination C-H Stretching
2580	O-H Stretching(Carboxylic group)
3392	N-H Stretching(Amine)

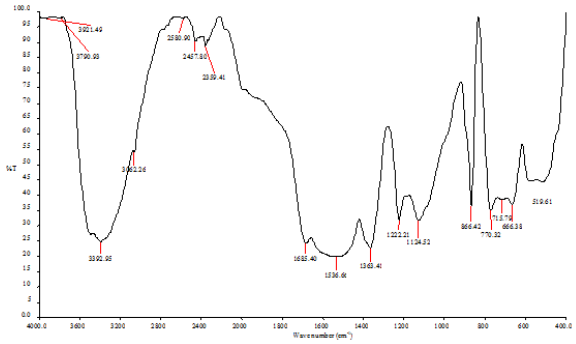

Figure 4: FT-IR spectrum for L-Lysine Nickel Maleate

3.4 UV-Visible Spectral Analysis

The linear optical properties of the grown crystal were studied by UV-Visible spectra using LAMBDA-35 spectrometer. The optical absorption spectral analysis of this crystal was carried out in the range between 200-1200 nm. It gives useful information about electronic transition of the compound. The lower cut - off wavelength of L-Lysine doped Nickel Maleate crystal is about 268 nm which is the most preferable region for electronic transition to occur in the grown crystal. From Fig.5, it was observed that the crystal has high transmittance (268-1200 nm) in the entire visible and near IR region. The transmittance above 85%led least absorption affirms high optical transparency and wide transmittance range of the grown crystal [10]. This optical property enables that this material is useful for optoelectronic application.

The dependence of optical absorption coefficient with the photon energy helps to study the band structure and the type of transition of electrons.

The absorption coefficient (α) were determined using Beer's law

$$\alpha = 1/d \log(1/T)$$

Where T is the transmittance and d is the thickness of the cell. As the indirect band gap, the crystal under study has an absorption coefficient (α) obeying the following relation for high photon energies.

$$\alpha = A(h\nu - Eg)^2/h\nu$$

Where A is constant, Eg is the optical band gap, h is the Planck's constant and ν is the frequency of the incident photon. The band gap energy was calculated from linear part of the Tauc's plot drawn between hν and $(\alpha h\nu)^2$ as shown in Fig.6

From the graph, the band gap energy forL-Lysine doped Nickel Maleatecrystalis found to be 4.2eV.

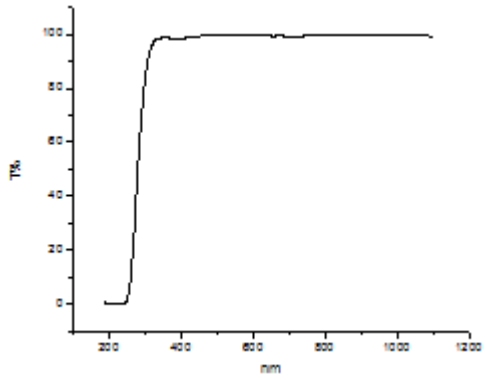

Figure 5: UV spectrum of L-Lysine Nickel Maleate Crystal

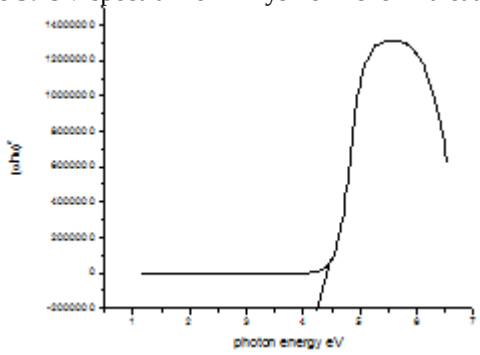

Figure 6: Band gap curve for L-lysine Nickel Maleate Crystal

3.5TG and DTA Analysis

To analyze the thermal stability and to confirm the melting point of the material, the Thermo Gravimetric and Differential Thermal Analysis was carried forL-Lysine doped Nickel Maleate crystal. The TG-DTA analysis of the grown crystal is shown in Fig.7. From the graphmajor weight loss starting at 110˚C. It observes that the L -LNM crystal has been found to be stable up to 110˚C,which confirms the absence of physically adsorbed water or lattice water in the grown crystal [11]. Steady decrease in weight observed up to 225˚C which may be due to the decomposition of the sample .The first stage of decomposition ends nearly at 225˚C with a weight loss of 45.52%.The DTA curve shows very sharp endotherm at 157.29˚C corresponding to the melting of L-LNM crystal.At temperature 600˚C, the final stage of decomposition occurs giving a total loss equal to 83.78%.

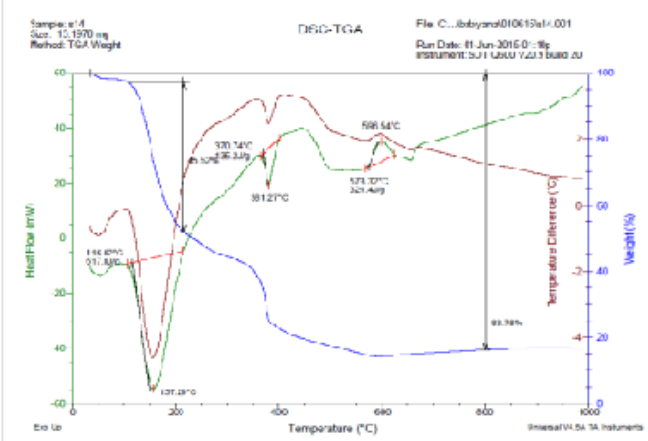

Figure 7: TG-DTA curve for L-Lysine Nickel Maleate crystal.

4. Conclusion

The good quality transparent L-Lysine Nickel Maleate crystal has been growns low evaporation solution growth technique. The powder XRD verifies the structure and the crystalline nature of the grown crystal. EDAX analysis confirms the presence of nickel in the crystals. FTIR spectrum confirms the incorporation of different functional groups present in the crystal. From UV-Visible spectrum, the lower cut-off wavelength is found to be 268 nm and the band gap of the grown crystal is calculated as 4.2 eV. The thermal stability of the crystal has been studied by obtaining Thermo gravimetric and Differential Thermal Analysis curve andit was found that the crystal is stable upto 110˚C.

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