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# Studies on Growth and Characterization of Undoped and L-Alanine Doped Ammonium Sulfate Semi Organic Crystals

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**Abstract:** ***Semi organic non-linear optical crystal of undoped and L-alanine doped ammonium sulfate have been synthesized and grown successfully from aqueous solution by slow evaporation technique. The grown single crystal was characterized by different instrumentation techniques such as FT-IR, UV-Vis, and Dielectric Hardness test. FT-IR studies reveal the functional groups and the optical characters were analyzed by UV-Vis spectral studies.***

**Keywords:** Growth from solution, FT-IR, Optical transmission, Mechanical, Dielectric properties

## 1. Introduction

Nonlinear optical (NLO) materials play a major role in nonlinear optics and in particular they have a great impact on information technology and industrial applications. In the last decade, however, this effort has also brought its fruits in applied aspects of nonlinear optics. This can be essentially traced to the improvement of the performances of the NLO materials [1]. The understanding of the nonlinear polarization mechanisms and their relation to the structural characteristics of the materials has been considerably improved. The new development of techniques for the fabrication and growth of artificial materials has drag magically contributed to this evolution [2]. The aim is to develop materials presenting large nonlinearities and satisfying at the same time all the technological requirements for applications such as wide transparency range, fast response, and high damage threshold [3-4]. But in addition to the process ability, adaptability and interfacing with other materials improvement is in nonlinear effects in devices, led the way to the study ofnew NLO effects and the introduction of new concepts. Optical solutions, optical switching and memory by NLO effects, which depend on light intensity, are expected to result in the realization of pivotal optical devices in optical fibre communication (OFC) and optical computing which make the maximum use of light characteristics such as parallel and spatial processing capabilities and high speed.[5].Currently, research on organic nonlinear optical (NLO) materials is strongly aggravated due to the demand of higher data rates in prospect optical communication technologies. Organic conjugated molecules are special candidates with nonlinear optical properties because of their high second and third order responses The π- conjugated systems having a donor and an acceptor show large NLO responses which enhance the study of these materials [6].

## 2. Experimental Procedure

**Synthesis and Crystal growth**

The undoped and L-alanine doped ammonium sulfate crystals has been grown from aqueous solution by slow evaporation method. The crystal was synthesized by dissolving ammonium sulfate in distilled water. This was stirred for 2 hours at room temperature ($32^0$C).

The solution was purified by repeated filtration. The saturation solution was kept in a beaker covered with filter paper and the solution was left undisturbed. As the result of slow evaporation, after 4 to 5 weeks white and pure crystals were obtained. The same procedure was followed to grow L-alanine mixed ammonium sulfate crystals. The grown undoped ammonium sulfate and amino acid doped ammonium sulfate crystals.

## 3. Characterization Techniques

### 3.1 FT-IR Spectral studies

The FT-IR spectroscopy studies were used to analyze the presence of functional groups in the synthesized compound. The FI-IR spectra of pure ammonium sulfate and L-alanine doped were recorded using KBr pellet technique employing Braker, IFS 66 FTIR spectrometer in the range 4000-400Cm$^{-1}$andare shown in fig1(a) (b). The characteristic vibrational frequencies of the functional groups of pure, L-alanine doped ammonium sulfate have been compared. Close observations of FT-IR spectra of pure and amino acid doped specimens reveal that doping generally result in of small shifts in some of the characteristic vibrational frequencies. It could be due to lattice strain as a result of metal doping.

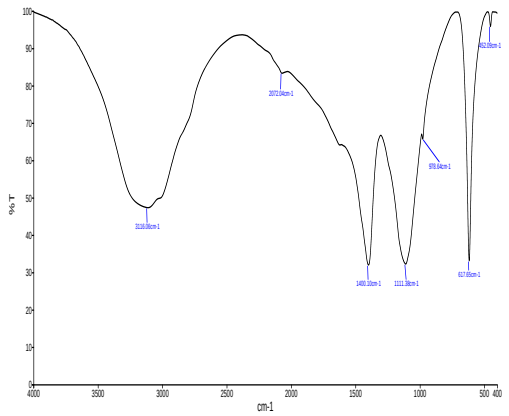


**Figure1 (a):** FT-IR Spectrum of Ammonium sulfate crystal

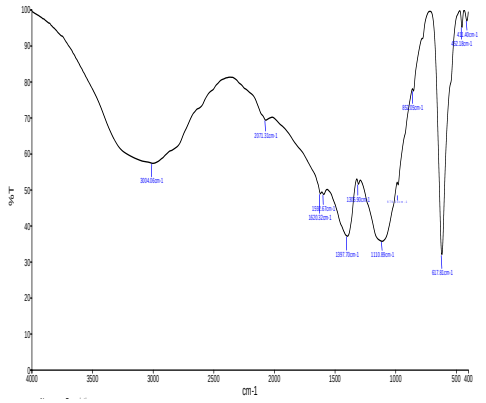

**Figure1 (b):** FT-IR Spectrum of L-alanine doped Ammonium sulfate crystal

**Table 1:** Function groups of undoped and l-alanine doped ammonium sulfate

| *Wave number $cm^{-1}$ undoped ammonium sulfate* | *Doped L alanine ammonium sulfate* | *Assignment* |
|---|---|---|
| 3116.06 | 3116.06 | C-H stretching(Aldehyde) |
| 852.05 | 852.05 | C-H stretching (Third overtone) |
| 1397.70 | 1397.70 | Aliphatic C=O stretching (Ketone) |
| 1400.10 | 1400.10 | Aliphatic C=O stretching (Ketone) |
| 1110.89 | 1110.89 | C-O-C stretching (Esther's) |

### 3.2 Optical studies: UV-Visible spectral Analysis

The optical transparencies of the grown crystals were analyzed by taking the UV-Visible spectral using LAMBDA-35 spectrometer wave length range between 200nm to 120nm. The observed spectra of undoped and L-Alanine doped ammonium sulfate crystals are shown in Fig2(a) and 2(b).The lower cutoff wavelengths of these crystals were found to be about 211nm, and 229nm. The percentage of transmission for ammonium sulfate is about 80% andL-alanine doped is 82% the transmittance Window range was obtained as 211 nm to 1100nm and 229nm to 1100nm. Hence these crystals can be used for opto electronic applications and in the second harmonic generation from the ND: YAG lasers

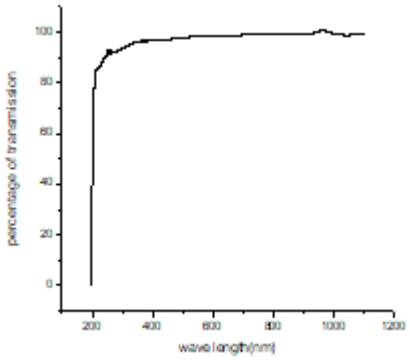


**Figure 2 (a):** UV-Visible spectrum of Ammonium Sulfate crystal

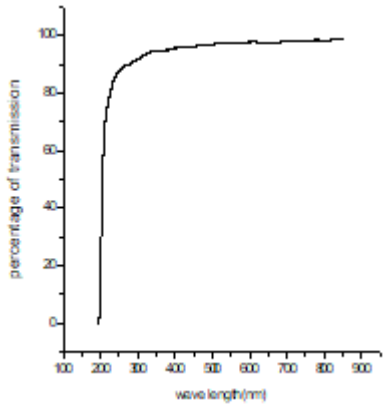


**Figure2 (b):** UV-Visible spectrum of L-alanine doped Ammonium Sulfate crystal

### 3.3 Optical energy band gap

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

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

$$\alpha = \frac{1}{d\log(\frac{1}{T})} \quad (1)$$

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

$$\alpha = \frac{A(h\nu - Eg)^2}{h\nu} \quad (2)$$

Where Eg is optical bandgap of the crystal and A is a constant. The plot of variation between photon energy (eV) and $(\alpha h\nu)^2$ shown in fig 3 (a) and (b).

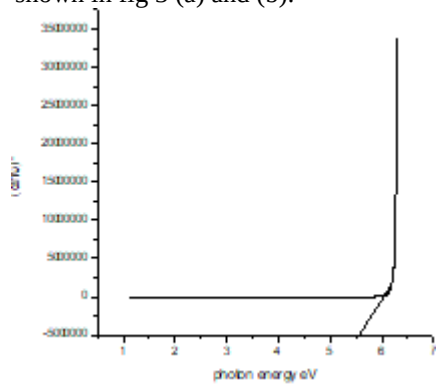


**Figure3 (a):** Band gap curve for ammonium sulfate

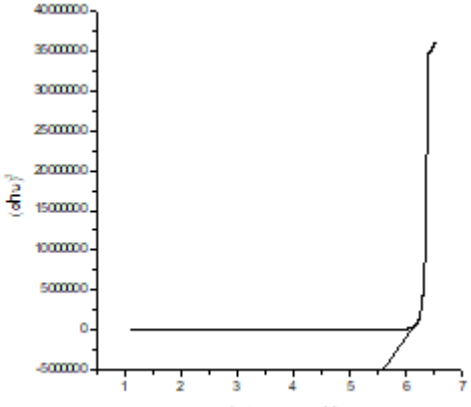


**Figure 3 (b):** Band gap curves for doped ammonium sulfate

From the graph the band gap for undoped ammonium sulfate and L-alanine doped ammonium sulfate crystals was found to be 5.6 eV and 5.57 eV.

### 3.4 Dielectric Studies

Dielectric measurement on dependent ammonium sulfate and doped ammonium sulfate single crystal were carried out a HIOKI 3532-50 LCR meter as a function of frequency (50Hz 20MHZ) and temperature ($32^0$C). From the dielectric measurement, the capacitance of parallel plate capacitor as a function of frequency and temperature was measured. The dielectric constant $\varepsilon^r$ and loss factor $\varepsilon`$ were calculated using the following relations.

$$\varepsilon^r = Cd/\varepsilon^0 A \quad (3)$$

$$\varepsilon` = \varepsilon^r \tan\delta \quad (4)$$

Here C is the capacitance of the parallel plate capacitor (pF), d is the thickness of the crystal. A is the area of the crystal acting as the dielectric,$\varepsilon_0$ is the permittivity of free space ($8.854x10^{12}F/m$) and tanδ is the dissipation factor. Plots of $\varepsilon^r$ and ε against log frequency at room temperature is shown in fig 4 and 5.

From the dielectric constant profiles, it is evident that the dielectric constant decreases with increasing frequency and increases with increasing temperature. The increase in dielectric constant low frequencies is attributed to the dependence of electronic, ionic, orientation and space charge polarization and the increase in dielectric constant with temperature leads to the conclusion that the thermal excitations of the atoms about their lattice points results in disordering the lattice. Here the space charge contribution to polarization is due to the purity of the material. Dielectric constant at low frequencies are comparable to optical frequencies which lead to minimization of the phase mismatch between optical and electrical pulses in high–speed travelling wave devices. In accordance with the Miller rule, the lower value of dielectric constant at high frequencies is a suitable parameter for the enhancement of SHG coefficient.

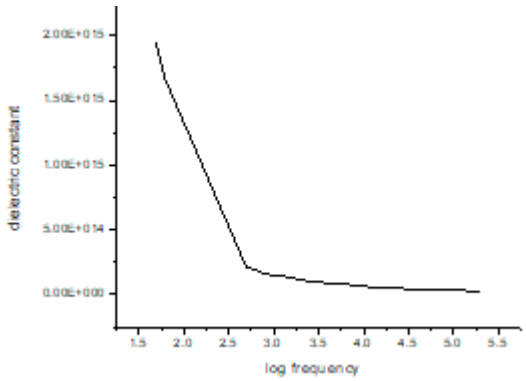


**Figure 4 (a):** Dielectric constant profile of undoped crystals

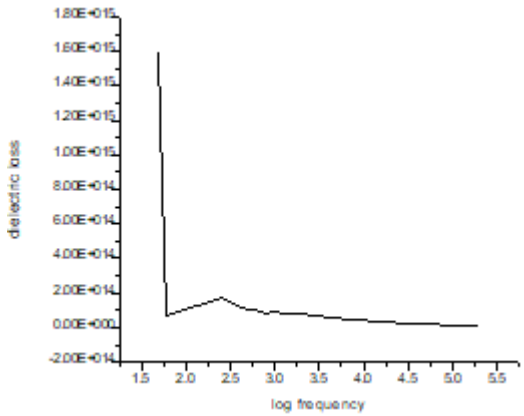


**Figure4 (b):** Dielectric loss profiles of undoped crystals

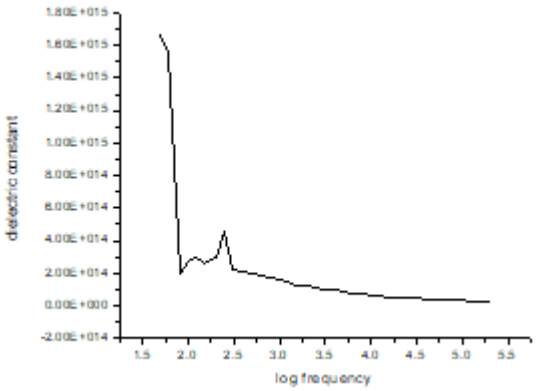


**Figure 5 (a):** Dielectric constant profile of L -alanine doped crystals

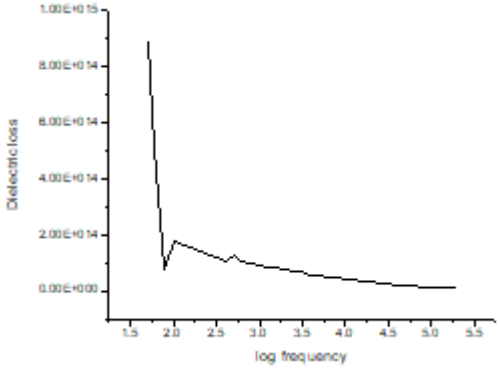


**Figure 5(b):** Dielectric loss profiles of L-alanine doped crystals

### 3.5 Mechanical studies: Vieker's Hardness Test

The Viekers micro hardness number Hv of the crystal was calculated using the relation

$$H_v = \frac{1.8544p}{d2}\ kg/mm^2 \quad (5)$$

The graph was plotted between loads Vs Hardness number is shown in Fig 2 (a) and (b) the vicker's hardness number increases for increasing loads. Work hardening coefficient 'n' and also calculated, by using Mayers' relation by plotting a graph between log p various log d which is shown in the Fig 6 (a) and (b) The data are given in the table 2 (a) and (b).

**Table 2 (a):** Hardness data for undoped Ammonium sulfate

| *Load p in grams* | *HV (kg/mm$^{2)}$* | *Log d* | *Log p* |
|---|---|---|---|
| 25 | 13.65 | -4.234 | -1.60206 |
| 50 | 23.9 | -4.2068 | -1.30103 |
| 100 | 34.1 | -4.18161 | -1 |

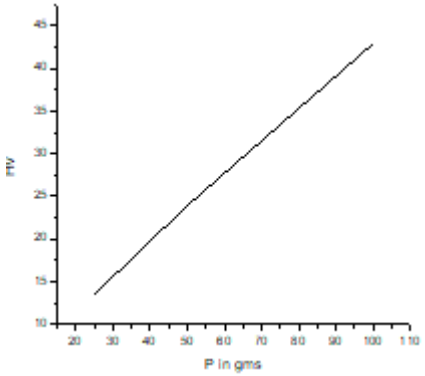


**Figure 6 (a):** Graph between Hv and load for the Ammonium sulfate

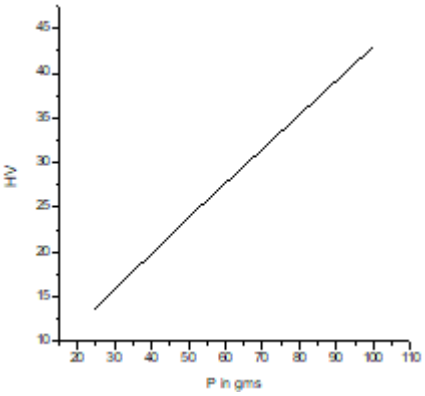


**Figure6 (b):** Graph between Hv and load for the L-alanine doped Ammonium sulfate

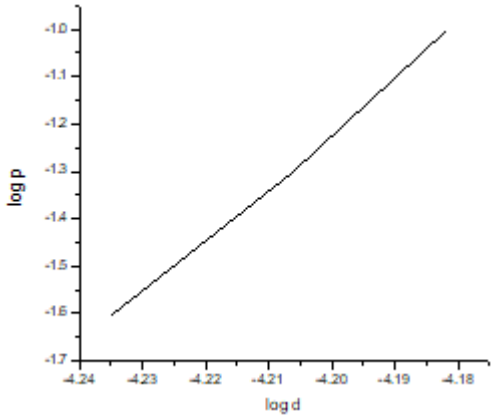


**Figure7 (a):** Graph between (log p) and (log d) for the Ammonium sulfate

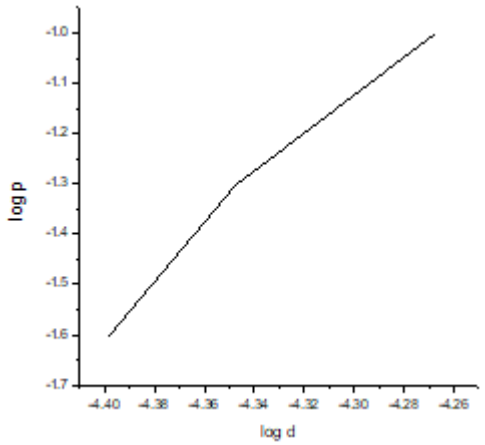


**Figure7 (b):** Graph between (log p) and (log d) for L-alanine doped Ammonium sulfate

**Table 2 (b):** Hardness data for L-alanine doped ammonium sulfate

| *Load p in grams* | *HV (kg/mm$^{2)}$* | *Log d* | *Log p* |
|---|---|---|---|
| 25 | 28.9 | -4.39808 | -1.60206 |
| 50 | 50.9 | -4.34739 | -1.30103 |
| 100 | 65.15 | -4.26694 | -1 |

From Meyer's law $p = ad^n$

Connecting the applied load (p) and diagonal length (d) of the indentation ,the work hardening coefficient 'n' is calculated. The calculated value of 'n' for ammonium sulfate and L-alanine doped ammonium sulfate is 4.5 and 11.26. Here 'a' is constant for a given material.

The work hardening coefficient 'n' is found for ammonium sulfate and L-alanine doped crystal by taking slope in the straight line of the graph drawn between log p and log d. which is shown in fig:7 (a) and (b).

From Onitsch [8] and Hanneman[9], 'n' lies between 1 and 1.6 for hard materials and is greater than 1.6 for soft material is [10,11]. `From the calculated values of 'n', it is suggested that the grown crystal are soft materials.

## 4. Conclusion

The potential semi organic NLO crystals of pure and amino acid doped ammonium sulfate were grown by slow evaporation method. The grown crystals were subjected to characterization techniques in order to assess the spectral, optical, mechanical and dielectric properties. The functional groups present in the crystals was determined by using FT-IR analysis and were tabulated which confirms the compounds. UV-Vis analysis was used for the determination of the optical quality of the crystals. The optical behaviors are analyzed using UV-Vis Spectrometer. It reveals that the crystals have an extended transparency down to UV and the bandgap estimated for the pure & amino acid doped ammonium sulfate crystals. From the optical response curve, the bandgap energy was found. By tailoring the absorption coefficient and tuning the band gap of material we can achieve the desired material suitable for fabricating various layers of optoelectronic devices. Vickers Hardness studies were performed in order to evaluate the mechanical strength of the grown crystals. The dielectric studies proved that the

crystals possessed a low dielectric constant values at higher frequencies, this suggests an enhanced optical quality with less defects. Owing to all these properties undoped and amino acid doped ammonium sulfate could be a promising material for NLO applications.

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