Growth and Characterization of L-Histidine doped Ammonium Sulphate Crystals

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ABSTRACT

Single Crystals of L-Histidine(LH) doped Ammonium Sulphate(AS) Crystals have been successfully grown from aqueous solution, by slow evaporation solution growth technique. Single crystals were grown in a period of 3 weeks. The lattice parameters were determined by PXRD analysis. The UV-Vis spectral study reveals that the material has a wide optical transparency in the entire region. The mechanical properties of the grown crystals have been studied using Vicker's Microhardness tester.

Key words: Crystal growth, X-Ray diffraction, L-Histidine, Ammonium Sulphate, UV, Microhardness.
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INTRODUCTION

Crystal growth from solution is a very important process that is used in many applications from the laboratory to the industrial scale. An innumerable number of organic and inorganic crystals are grown in this fashion [1]. Organic crystals have large nonlinear susceptibilities compared to inorganic crystals. Most organic NLO crystals have usually poor mechanical and thermal properties and are susceptible to damage during processing. It is difficult to grow large optical quality crystals of these materials for device applications [2-4]. In order to keep the merits and overcome the short comings of organic materials, some new classes of NLO crystals such as metal organic or semiorganic crystals have been developed [5]. Combining the high optical nonlinearity and chemical flexibility of organics with temporal and thermal stability and excellent transmittance of inorganics, semiorganic materials have been proposed and are attracting a great deal of attention in the nonlinear optical field [6-7]. Several reports of microscopic and macroscopic responses for organic-inorganic (NLO) systems have appeared in recent literature [8-13]. The crystals were grown by slow evaporation technique [14-15] and characterized by powder X-ray diffraction, UV- Vis and Microhardness studies.

MATERIALS AND METHODS

2. Experimental details
2.1 Crystal Growth
Analytical Reagent (AR) grade of L-Histidine doped Ammonium Sulphate in different molar ratios such as 0.01, 0.03, 0.05, 0.07 and 0.09 was grown. For this the required quantities of L-Histidine and Ammonium Sulphate were taken in a beaker and dissolved in double distilled water and stirred atleast for three to four hours using a heater cum magnetic stirrer. Now cover the beaker with a lid containing some holes. Evaporation take place, nucleation occurs

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for all crystals within four days. After a period of three weeks high quality single crystals are obtained and are harvested.

3. Characterization Studies
The grown crystals of L-Histidine doped Ammonium Sulphate crystals were analysed by powder XRD analysis. Powder XRD analysis was carried out to study the crystallinity by using an X-ray diffractmeter, MODEL RICH, SEIFERT, XRD 3000 P , with monochromatic nickel filtered CuKα (λ= 0.15406 nm) radiation. The sample was scanned over the range 10 - 70^0 at the rate of 1^0/min. The linear optical properties of the crystals were examined between 200 and 2000 nm using the VARIAN CARY 5E UV- Vis – NIR spectrophotometer.

RESULTS AND DISCUSSION

4.1. Powder X-RAY diffraction analysis
The freshly ground powder sample of Ammonium Sulphate and L-Histidine was subjected to powder X-ray diffraction analysis with a monochromated CuKα radiation (λ=0.15406 nm). The recorded PXRD pattern of L-Histidine doped Ammonium Sulphate is shown in figs 1.1, 1.2, 1.3, 1.4, 1.5 and 1.6. The observed sharp peaks confirm the crystalline nature of the grown L-Histidine doped Ammonium Sulphate crystals.
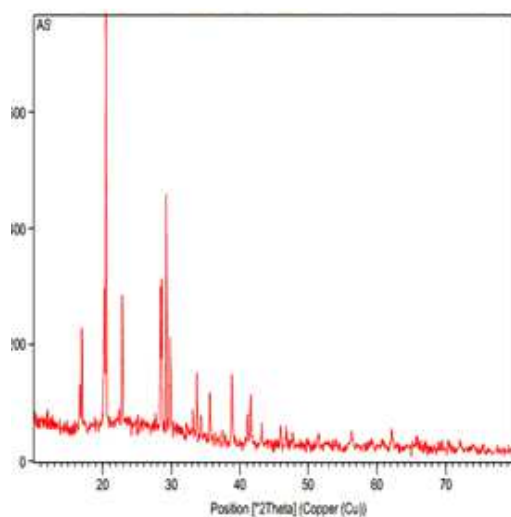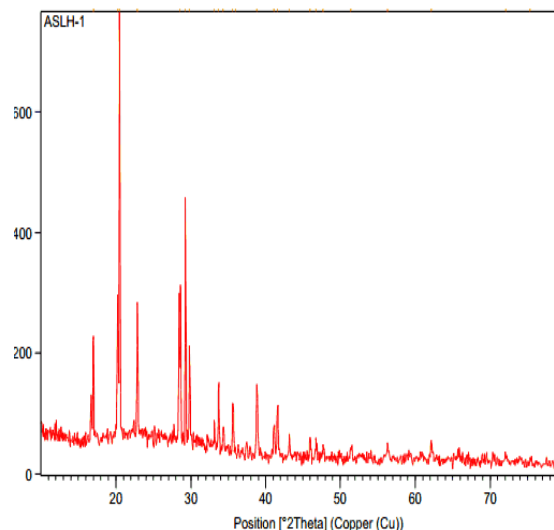

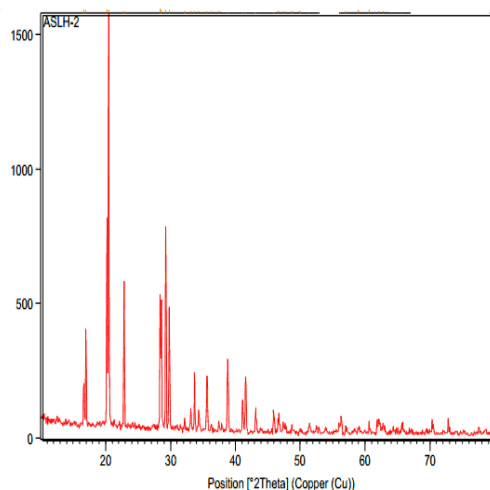**Fig 1. 1. PXRD pattern of Pure AS**   **Fig 1. 2. PXRD pattern of 0.01mol%**

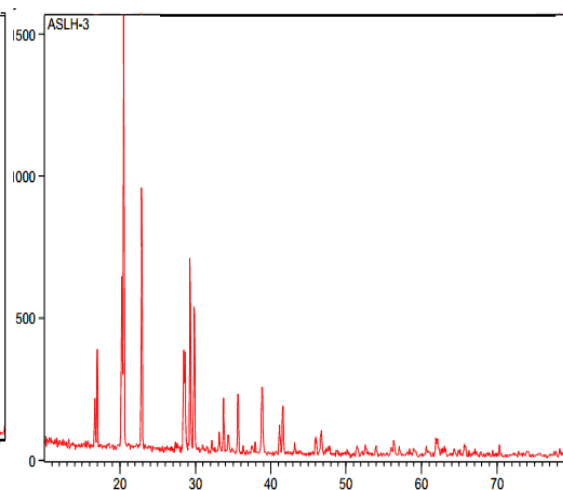**Fig 1. 3. PXRD pattern of 0.03 mol%**   **Fig 1. 4. PXRD pattern of 0.05 mol%**
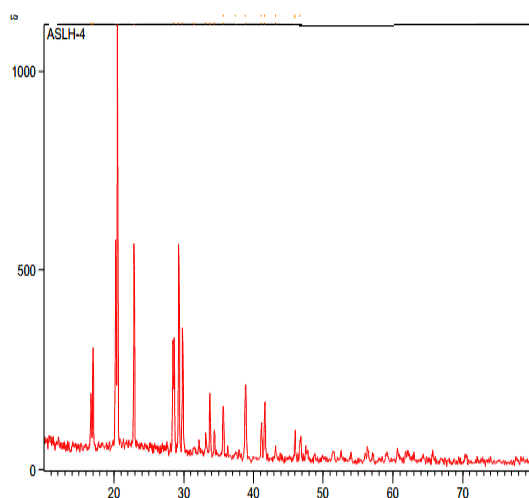
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Fig 1. 5. PXRD pattern of 0.07 mol%   **Fig 1. 6. PXRD pattern of 0.09 mol%**
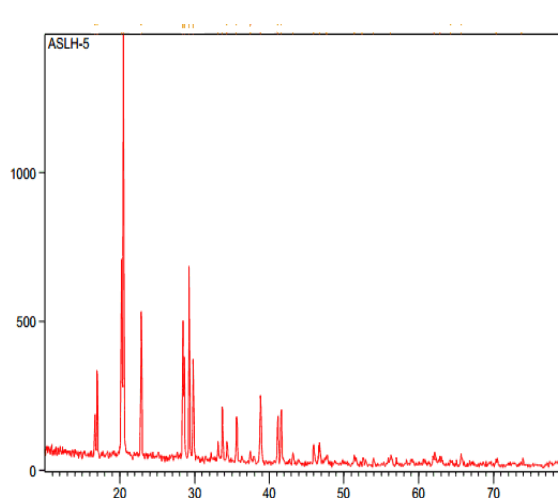
Table.1 Lattice parameters of the grown Crystals.

System	a A^0	b A^0	c A^0	Vol$(A^0)^3$
Pure AS	5.970	10.60	7.760	491.06
0.01 mol%	5.987	10.62	7.767	494.28
0.03 mol%	5.994	10.63	7.789	496.77
0.05 mol%	6.121	9.941	7.773	473.05
0.07 mol%	6.118	9.940	7.771	472.70
0.09 mol%	5.989	10.63	7.770	494.84

When the dopant concentration increases the lattice parameters a, b and c values are found to increase up to 0.05 mol% then for further increase in the dopant concentrations the lattice parameters varies randomly.

4.2 Optical transmission studies
Fig 2 show the UV-Vis NIR spectrum of LH doped AS recorded with highly transparent and defect free single crystals. It is evident from the spectrum that LH doped AS has a wide transmission spectra between 190 nm to 1100 nm. From the spectra both Pure and LH doped AS crystals show good transmittance in the entire visible region. The cut-off wavelength for the Pure and the LH doped AS crystals are almost same.
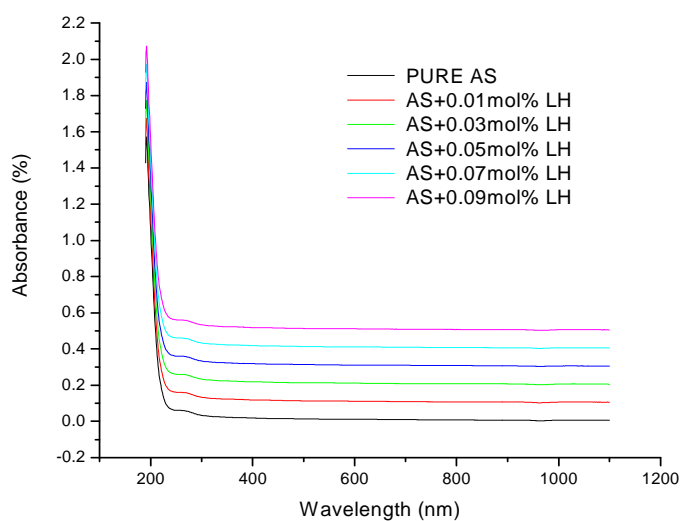

Fig 2: Absorbance spectra of Pure and LH doped AS crystals

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4.3 Measurement of Microhardness

Pure and LH doped AS crystals were placed on the platform of the microhardness tester and loads of different magnitudes (25, 50 & 100) were applied over a fixed interval of time (10s). The maximum applied load was restricted to 100g as micro cracks were developed at higher loads. The hardness was calculated using the relation

$$H_v = 1.8544 \frac{P}{d^2} \text{ Kgmm}^{-2}$$

Where P is the applied load in Kg and d is the average diagonal length of the indentation impression in micrometer. The relation between hardness number (H_v) and load (P) for LH doped AS crystals are shown in the Fig 3. The indentation hardness is measured as the ratio of applied load to the surface area of the indentation [16-17]. It is observed from the graph that the hardness value increases with increasing load .The increase of the microhardness with the increasing load is in agreement with the normal indentation size effect (ISE).

Table 2. Hardness number (H_v) and load (P) of the grown crystals.

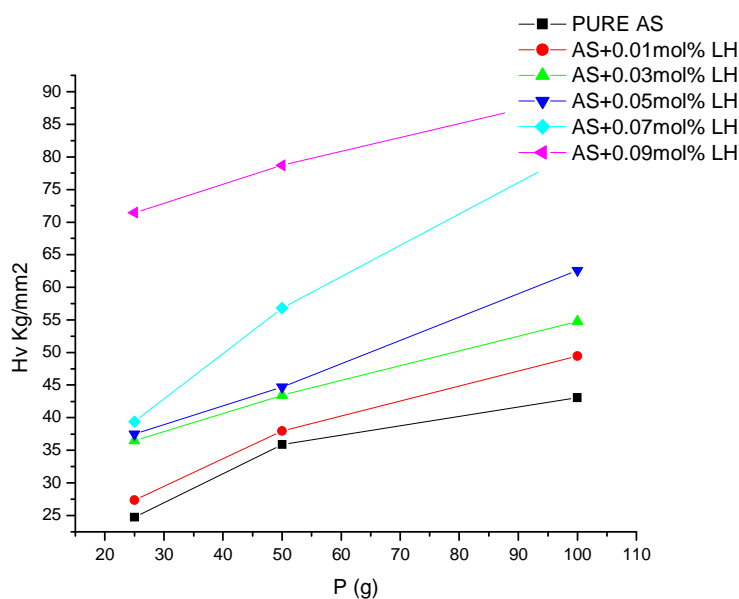| P (g)	H_v Kg/mm^2					
	Pure AS	0.01 mol%	0.03 mol%	0.05 mol%	0.07 mol%	0.09 mol%
25	24.72	27.37	36.44	37.48	39.40	71.43
50	35.89	37.93	43.40	44.73	56.80	78.68
100	43.08	49.45	54.77	62.60	80.87	89.32

Fig 3. load P (g) versus H_v (Kg /mm^2)

CONCLUSION

Optically clear single crystals of LH doped AS are conveniently grown by slow solvent evaporation technique. The single crystals PXRD studies confirm the orthohombic structure of the crystals. UV – Vis NIR spectrum of LH doped AS crystals recorded with highly transparent and defect free single crystals. The Microhardness study reveals the crystals are hard in nature.

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