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Effect of L-Histidine in Magnesium sulphate crystals

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ABSTRACT

Single crystal of L-histidine doped Magnesium Sulphate (LHMS) crystal has been grown from aqueous solution by slow evaporation technique. The grown crystals were subjected to powder X-ray diffraction analysis, confirming that the crystalline nature of the crystal. The mechanical properties of the grown crystals have been studied using Vicker's microhardness tester. The UV-visible transmittance studies show that the grown crystals have wide optical transparency in the entire visible region.

Key words: X-Ray diffraction, L-histidine, Magnesium Sulphate, UV, Microhardness

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INTRODUCTION

Nonlinear optics is playing a major role in the emerging photonic and optoelectronic technologies. Efforts have been taken to synthesize new materials for variety of nonlinear optical (NLO) applications such as optical signal processing, parametric amplification, optical phase conjugation, etc [1, 2]. 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 [3-5]. 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 [6]. 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 [7-8]. NLO material L-Histidine teterafluoroborate single crystal has been grown [9]. Recently the growth and characterization of NLO material L-histidinium bromide,L-histidine perchlorate and L-histidine hydrofloride dehydrate crystals were also reported [10,11,12]. In this work we report the growth of L-histidine doped Magnesium sulphate single crystals with different doping concentration. The grown crystals were characterized by PXRD, UV- Vis and Microhardness studies.

MATERIALS AND METHDOS

2.1 Crystal growth

Pure and L-histidine doped Magnesium sulphate crystals were grown from aqueous solution by slow evaporation. The crystals were synthesized by dissolving Magnesium sulphate and L-histidine in the molar ratio 1:0.01, 1:0.03, 1:0.05, 1:0.07 and 1:0.09 in aqueous solution. The solution was stirred well at a constant rate to get homogenity. Then the solution were taken in a crystallizing vessel and covered with a perforated sheet to facilitate the evaporation of the solvent at room temperature. Single crystals with perfect external shape were obtained by spontaneous nucleation after a period of 20 days. The grown crystals were found to be highly transparent, free from visible inclusions and non-hygroscopic in nature.

2.2 Characterization Studies

The grown crystals were powered by morter with a pestle. The X-ray data collection was carried out on X-ray diffractometer, MODEL RICH, SEIFERT, XRD 3000 P, with monochromatic nickel filtered CuKα (λ= 0.15408 nm) radiation. The samples were 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. Microhardness studies have been carried out on Pure and L-histidine doped Magnesium Sulphate crystals using a MVCCD-1000 Video Digital microhardness tester fitted with a Vickers diamond pyramidal indentor attached to an incident light microscope.

RESULTS AND DISCUSSION

4.1 Powder X-RAY diffraction analysis

The grown crystals were subjected to powder XRD to confirm the crystal structure. The pure and L-histidine doped crystals crystallize in Orthorohombic structure. The unit cell parameters of pure and L-histidine doped Magnesium sulphate crystals were shown in Table 1. The PXRD spectrums of the grown pure and doped crystals are shown figs 1.1, 1.2, 1.3, 1.4, 1.5 and 1.6. The observed sharp peaks confirm the crystalline nature of the crystals. The pure magnesium sulphate crystal has good agreement with the JCPDS value(75-0673). The spectra and lattice parameter of the L-histidine doped crystals revealed that the structures of the doped crystals are slightly distorted compared to the pure crystal. This may be due to the absorption or substitution of doped atom in lattice sight [13]. It is observed that the reflection lines of the doped Magnesium sulphate crystals correlate well those observed in the pure Magnesium sulphate.

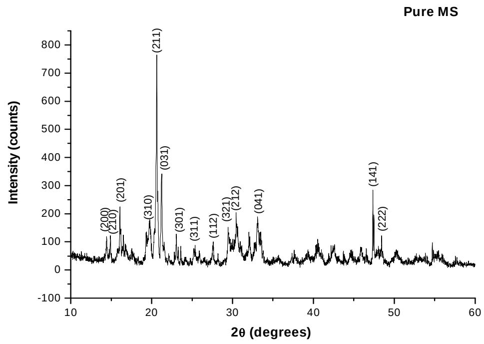


Fig 1. 1. PXRD pattern of Pure MS

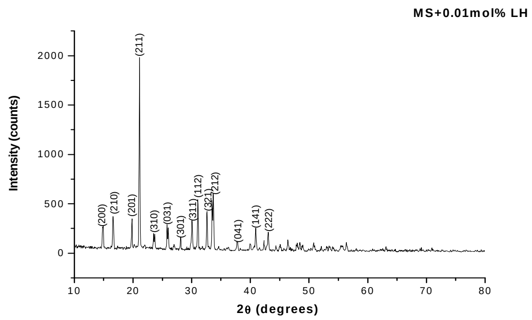


Fig 1. 2. PXRD pattern of 0.01mol%

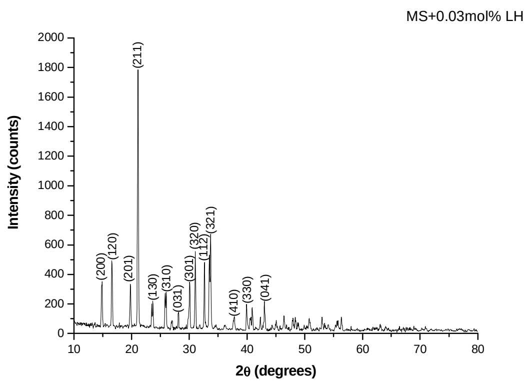


Fig 1. 3. PXRD pattern of 0.03mol%

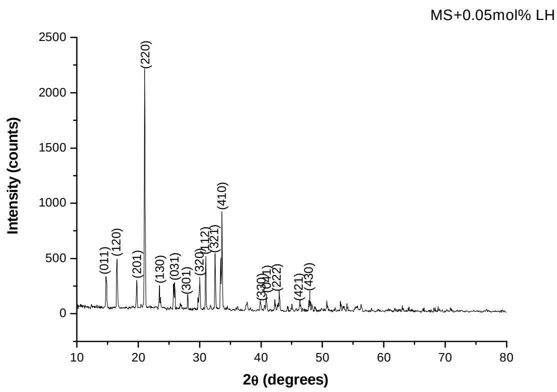
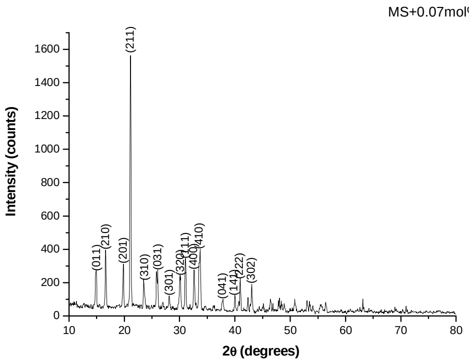


Fig 1. 4. PXRD pattern of 0.05mol%



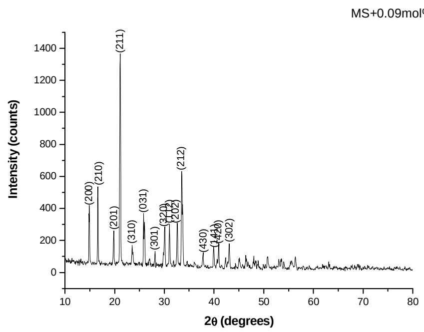


Fig 1.5. PXRD pattern of 0.07mol% **Fig 1. 6. PXRD pattern of 0.09mol%**

Table.1 Lattice Parameters of the grown Crystals

system	Pure MS	Concentration of L-histidine in Magnesium Sulphate				
		0.01 mol%	0.03 mol%	0.05 mol%	0.07 mol%	0.09 mol%
a(A^0)	11.86	11.900	11.875	11.904	11.900	11.966
b (A^0)	11.98	11.978	11.981	12.007	11.991	11.752
c (A^0)	6.847	6.847	6.855	6.866	6.803	6.852
Volume(A^0)^3	973.9	976.134	975.446	981.512	970.915	963.77

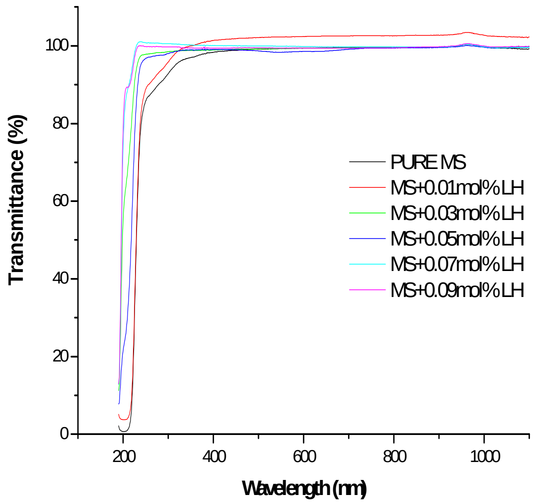


Fig 2.1 : Transmittance spectra of Pure and L-histidine doped MS crystals

3.2 Optical transmission spectral analysis

UV –Visible transmittance spectrum of pure and L-histidine doped Magnesium sulphate crystals are shown in Fig 2.1. Optical data were taken for the samples of thickness about 2 mm. UV-Vis studies also give important structural information because absorption of UV and visible light involves promotion of the electrons in π and σ orbital from the ground state to higher energy states [13]. The crystals have a good optical transmission in the entire visible region and the lower cut off wavelength (λ_{cut}) is observed to be at 210 nm. The larger transmission in the entire visible region and short cut off wavelength enables to be a potential material for second and third harmonic generation [14]. The percentage of transmission for pure and doped crystals have nearly 100%, and the crystal having molar ratio 1:0.09 has maximum percentage of transmission. The pure KDP crystal has about 70% of transmission [15] but L-histidine doped Magnesium sulphate crystals have 100% transmission. When increase in dopant concentration wavelength shifted to the lower cut-off region. The pure and L-histidine doped crystals are invariably have higher transmission percentage compared to pure KDP crystal. So it is more suitable for optical applications.

4.3 Microhardness studies

Microhardness measurements were carried out on pure and doped Magnesium sulphate crystals using a Leitz Weizler hardness tester fitted with a diamond indentor. The indentations were made using a Vicker's pyramidal indentor for various loads from 25 to 100g. The maximum applied load was restricted to 100 gm as micro cracks were developed at higher loads. Hence, readings were not taken for higher loads. Vicker's microhardness number was evaluated from the relation

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

Where H_v is Vicker's hardness number, P is the applied load in kg and d is the diagonal length of the indentation impression in micrometer. The indentation hardness is measured as the ratio of applied load to the surface area of the indentation [16, 17, 18]. Table 2 shows hardness number (H_v) and applied load (P) of the grown crystals. The maximum value of the hardness for pure crystal is found to be 21.235 Kg/mm^2 and maximum hardness for 0.09 mol% doped crystal is found to be 39.11 Kg/mm^2 .Addition of impurities in Magnesium sulphate modifies the hardness value. The doped Magnesium sulphate crystals have much more harder than the pure crystal. The plot (fig 3) between load P versus H_v shows that hardness of the crystal increases with increasing load. The work hardening coefficient (n) of the material is related to the load (P) by the relation $P = ad^n$ where 'a' is an arbitrary constant. This is well satisfied as shown by figure 3.The work hardening coefficient 'n' shown in table 2. Another well known NLO materials L-histidinium tetrafluorophthalate (LHFP) has a work hardening coefficient 4.3 [19]. According to Onitsch, $n \leq 1.6$ for hard materials and $n>1.6$ for soft materials [20]. So L-Histidine doped Magnesium Sulphate Crystals also hard materials.

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

P (gm)	Hv Kg/**mm²**					
	Pure MS	0.01 mol% LH	0.03 mol% LH	0.05 mol% LH	0.07 mol% LH	0.09 mol% LH
25	8.262	10.605	15.42	15.915	20.573	22.515
50	13.516	15.981	22.75	24.61	26.295	26.43
100	21.235	22.695	31.17	31.65	33.055	39.11

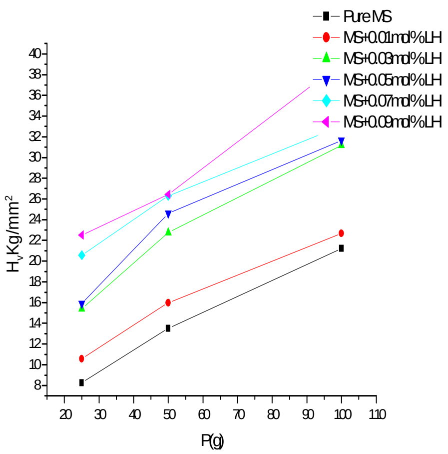


Fig 3. load P (g) versus H_v (Kg /mm²)

Table 3. Microhardness-Workhardening coefficient

No	Sample	Work hardening coefficient (n)
1.	Magnesium sulphate	0.159
2.	L-H (0.01 mol%) doped MS	0.225
3.	L-H (0.03 mol%) doped MS	0.240
4.	L-H (0.05 mol%) doped MS	0.236
5.	L-H (0.07 mol%) doped MS	0.328
6.	L-H (0.09 mol%) doped MS	0.322

CONCLUSION

Optically clear, pure and doped Magnesium sulphate (L-histidine) crystals have been grown by slow evaporation technique. Powder XRD was taken to analysis the structure of the crystals is Orthorhombic. It is also interesting that there is a change in lattice parameter values for the doped crystals minimum absorption in the entire visible region is interesting and maximum transmission is observed for all crystals. The crystals have a wide transparency range with a lower cutoff of 210 nm. Microhardness studies reveal that the hardness increases with increase of load and the crystals are hard materials.

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