Imperial Journal of Interdisciplinary Research (IJIR)
Vol-2, Issue-12, 2016
ISSN: 2454-1362, http://www.onlinejournal.in

Synthesis and Characterization of L-Alanine Magnesium Sulphate (LAMS) Single crystals

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Abstract: *Single crystal of L-Alanine doped Magnesium Sulphate (LAMS) 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 modes of vibration of different molecular groups present in the sample were identified by FTIR spectral analysis.*

1. 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-Alanine Magnesium sulphate single crystals with different doping concentration. The grown crystals were characterized by SXRD, PXRD, FTIR and Microhardness studies.

2. Experimental Procedures

Analytical reagents (AR) grade chemicals were used in the present study. L-Alanine Magnesium Sulphate crystals (LAMS) were grown from aqueous solution by slow evaporation technique. The crystals were synthesized by dissolving L-Alanine doped with Magnesium Sulphate in the molar ratio of 1:1. The solution was stirred well at a constant rate to get homogeneity. 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. Chemical Reaction,

$$C_3H_7NO_2 + MgSO_4 \rightarrow C_3H_7NO_2 .MgSO_4$$

2.1 Characterization Studies

Single crystal X-ray diffraction (SXRD) was collected at room temperature by using Bruker AXS kappa Apex 2 CCD. Single crystal X-ray diffractometer with $M_oK\alpha$ radiation (λ=0.7107Å) to indentify the crystals lattice parameters. Powder X-ray diffraction (PXRD) data were collected by employing a XPERT-PRO diffractometer with Cukα radiation (λ=1.54056Å) scanned over the 2θ range of 0° - 80° C at the rate of 1°/min to understand the crystallization of the crystals grown and characterize structurally. The Fourier transform infra-red (FTIR) spectra of all the seven crystals grown were recorded by a PERKIN EIMER RX1 spectrometer using the kBr pellet technique in the frequency range of 400-4000 cm^{-1} to identify the presence of functional groups. Microhardness studies have been carried out on L-Alanine doped Magnesium Sulphate crystals using a HMV-2T shimadzu microhardness tester fitted with a

Vickers diamond pyramidal indenter attached to an incident light microscope.

3. Results and Discussion

3.1 Lattice variations

Photographs of the sample crystals grown in the present study are shown in Figure 1.

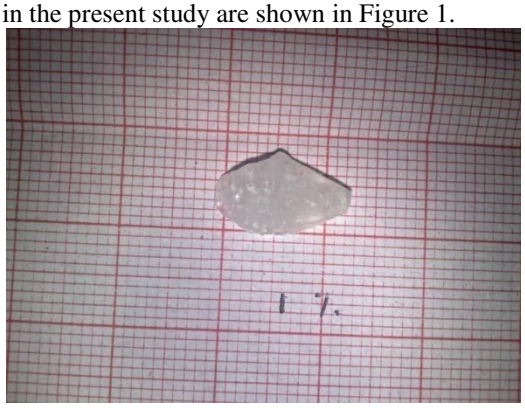

Figure 1. Grown LAMS crystals

The grown crystal belongs to orthorhombic system. The observed lattice parameters, space group and the indexed PXRD pattern recorded are shown in Figure 2.

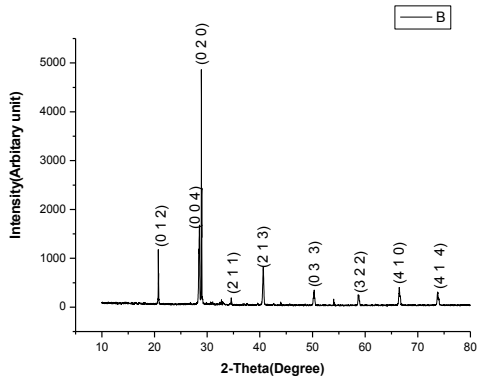

Figure 2. PXRD pattern of the grown LAMS crystals

The well-define peaks at specific 2-theta values show high crystallinity of the grown crystal. All the reflections of powder XRD pattern of the crystal of this work were indexed using unit cell software. The unit cell parameters of L-Alanine Magnesium Sulphate crystals were shown in Table 1.

Table 1: Lattice Parameters, space group and crystal system of LAMS Crystals

Lattice parameters				V	Space group	Crystal system
a	b	c	α β γ	(Å)^3		
5.8	6.06	12.40	90°	436	P22	Orthorhombic

3.2 FTIR spectroscopic analysis

Figure. 3 shows that the FTIR spectrum of LAMS.It is provide information about the chemical bonding or molecular structure of material. In the present study the spectrum is observed that the peaks at 2510.34 cm^{-1} are attributed to OH stretching. The C-CHO stretching vibration gives rise to absorption with 1150.88 cm^{-1} to 1019.74 cm^{-1}. The C-H out of plane bending is observed at 919.13 cm^{-1} to 772.90 cm^{-1} in both IR and Raman spectrum. The out of plane bending in the peaks at 412.80 cm^{-1} [13]. The COO^- symmetric stretching occurs at 1304.70 cm^{-1}. The $C \equiv C$ stretching vibration give rise to absorption with 2111.34 cm^{-1}. The sharp intense band in IR spectrum at 1455.85 cm^{-1} can assigned to $N = O$ stretching vibration. The peaks at 648.74 cm^{-1} is due to C-S stretching mode [14]. The NH_3^+ asymmetric and symmetric bending occur in the region 1625-1560 cm^{-1} and 1550-1500 cm^{-1} respectively [15]. The absorption peaks and their assignments are provided in Table: 2

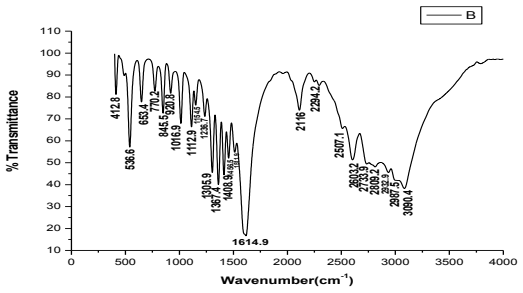

Figure 3 : FT-IR spectrum for LAMS Crystals

Table 2: Vibrational Assignments of L-Alanine Magnesium Sulphate crystals

Wave number (cm^1)	Assignments
412.80	Out of plane bending
648.74	C-S stretching
772.90	C-H out of plane bending
849.00	C-H out of plane bending
919.13	C-H out of plane bending
1013.66	C-CHO stretching
	C-CHO stretching
1150.88	COO^- symmetric stretching
1304.70	N= O stretching
1455.85	NH_3^+ symmetric bending
1512.42	NH_3^+ asymmetric

	bending
1616.17	C≡ C stretching
	OH stretching
	OH stretching
2111.34	OH stretching
2510.34	
2603.44	
2936.02	

3.3 Microhardness studies

Micro hardness measurements were done using a Vickers's micro hardness indenter using HMV-2T Shimadzu Hardness Tester. The mechanical properties of the grown crystal were studied by Vickers hardness test. Load 25, 50 and 100 g and the hardness was calculated using the relation

$$Hv = 1.8554\ 2\ p/\ d^2\ \text{Kg mm}^{-2}$$

Where P is the load applied in kg and d is the diagonal length of the indented impressions in mm. A plot between the load P and hardness number HV is shown in Figure 4a which shows that the micro hardness number increases with increasing load for the grown crystal. The graph between log p against log d for L AMS Crystals is shown in Figure 4b. The slope of the straight line gives the work hardening coefficient (n). The value for the work hardening coefficient for LAMS Crystal is 1.78. According to Onitsch[14], if n is greater than 1.6, the crystal belongs to soft category. As n is greater than 1.6, L-Alanine doped magnesium sulphate crystal is a soft material and the hardness number increases with the load is also observed. According to Hays-Kendall's approach load dependent hardness may be expressed by

$$P = W + A_1 d^n$$

Where W is the minimum load to initiate plastic deformation, A_1 is the load independent constant and the exponent n = 2. The value of W and A_1 can be calculated by plotting the experimental P against d^n plot. These two values have been estimated from the plot drawn between P and d^n shown in Figure: 4c. The corrected hardness H_v has been estimated using the relation

$$H_v = 1.8544 \times A_1$$

The calculated hardness parameters are given in Table: 3

Table 3: Hardness parameters of LAMS crystal

Hardness parameters	Value
Mayer's index number (n)	1.78
W (g)	16
A_1 (gm mm^{-2})	0.0479
Corrected hardness H_v (Kg mm^{-2})	88.825

The elastic stiffness constant (C_{11}) for different loads are calculated using Wooster's empirical formula $C_{11} = H_v^{7/4}$, and is shown in Table 4.The stiffness constant gives an idea about the tightness of bonding with the neighboring atoms. In the case of L-Alanine Magnesium Sulphate crystal the stiffness constant is found to increase with the applied load.

Table 4: The elastic stiffness constant for different load

Load (g)	C_{11} ($\times 10^{14}$ Pa)
25	**43.91**
50	**50.39**
100	**87.75**

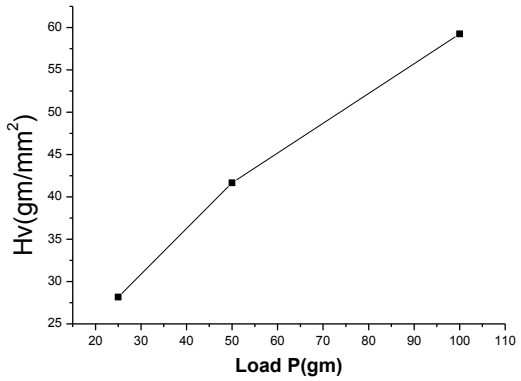

Figure 4a: Variation of H_v with load P

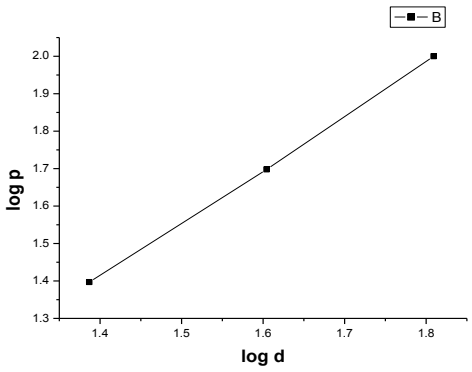

Figure 4b: Plot between log P and log d

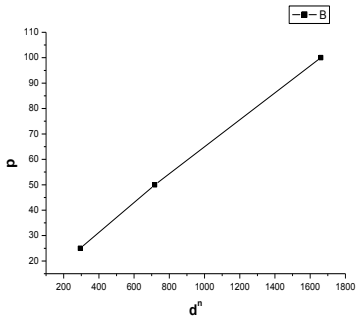

Figure 4c: Plot between log d^n and P

4. Conclusion

The L-Alanine Magnesium Sulphate crystal (LAMS) was grown by slow evaporation method and characterized from aqueous solution at room temperature. Result of single crystal XRD measurements show that the grown crystal belongs to orthorhombic system. The FTIR spectroscopic measurements confirmed the formation of functional groups of grown crystal. The microhardness studies shows that the crystal is soft and it can withstand a load of 100 gm which shows that the mechanical strength is high so that it can be used for device fabrication.

5. References

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