International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN : 0974-4290
Vol.6, No.6, pp 3248-3251, Aug-Sep 2014

ICMCT-2014 [10th – 12th March 2014]
International Conference on Materials and Characterization Techniques

Studies of L-alaninium maleate crystals admixtured with urea (LAMU) grown by slow cooling method

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Abstract: Nonlinear optical (NLO) crystals are the family of materials that have attracted the researchers and industries due to their wide applications in optical communication, optical computing, photonics, color display, electro-optic modulation and high energy lasers etc. In this work, L-alaninium maleate crystal is admixtured with urea (LAMU) to improve its physical and chemical properties. Single crystals of LAMU were grown by solution method with slow cooling technique. The grown crystals of LAMU were characterized by various studies such as XRD, FT-Raman, FTIR and hardness studies. The obtained results from various studies of LAMU crystals were discussed.
Key words: NLO; XRD; FTIR; FT-Raman; Hardness.

Introduction and Experimental

Amino acid family crystals are of great interest due to their attractive nonlinear optical properties. L-alanine is an organic material, in zwitterionic form both in crystal and in aqueous solution over a large range of pH. The growth and characterization of L-alanine and its complexes were reported by many authors earlier [1]. Maleic acid is a dicarboxylic acid with a large □-conjugation has attracted much attention. In the present study, bulk single crystals of L-alaninium maleate admixtured with urea (LAMU) have been grown by the slow cooling method with optimized growth conditions. The grown crystals were subjected to various characterization studies.

LAMU salt was synthesized using analar grade L-alanine, maleic acid and urea in 1:1:1 and the calculated amounts of the materials were dissolved using the double distilled water as a solvent. This solution was heated at 50 ^{o}C and left for evaporation to dryness. Slow cooling solution growth technique has been widely used for the growth of organic and inorganic materials. Optically transparent and defect free tiny crystals were obtained by the self nucleation of the saturated solution by slow evaporation and the seed crystals were immersed in the supersaturated solution at 40^{o}C. The sealed beaker with supersaturated solution was housed in Eurotherm controlled constant temperature bath (CTB) with accuracy of ±0.01°C. Then the temperature was reduced at a rate of 0.5 ^{o}C/day as growth progressed and the growth process was monitored carefully. Optical

quality crystals have been grown and shown in figure 1. Once the temperature 28 ^{o}C is reached the crystal is harvested.

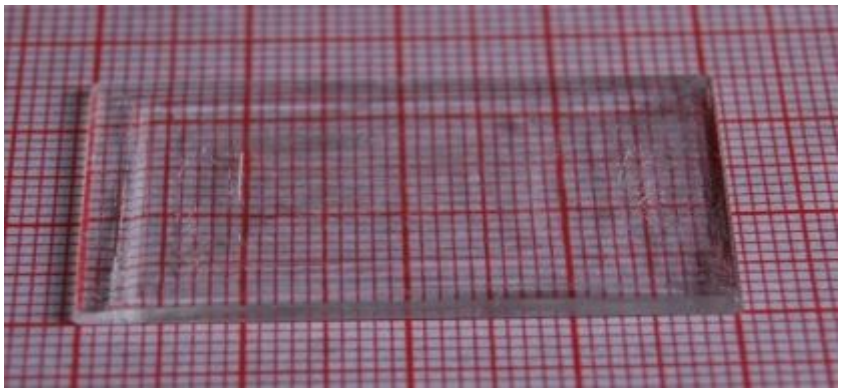

Fig.1: Grown crystal of LAMU

Results and discussion

From single crystal X-ray diffraction analysis, the lattice parameters of LAMU crystal were obtained as a (Å) = 5.591(12), b(Å)=7.377(17), c (Å)= 23.69(5), $\alpha = \beta = \gamma = 90°$ and hence volume (Å^3) = 977.09(4). Thus, LAMU crystallizes in orthorhombic system. The unit cell parameters agreed well with the reported values [2].

The functional groups were identified by Fourier transform infrared studies using Perkin Elmer Fourier transform infrared spectrometer. Raman spectra together with FTIR spectra form an indispensable tool for molecular analysis. The FT-Raman spectrum was recorded in the range of 50–4000 cm^{-1}. Vibrational (FTIR and FT-Raman) spectra of LAMU are shown in figure 2. The assignments for the vibrational peaks/bands of the spectra are given in accordance with the reported literature [3]. The complete assignments for characteristic absorption peaks/bands are listed in table 1.

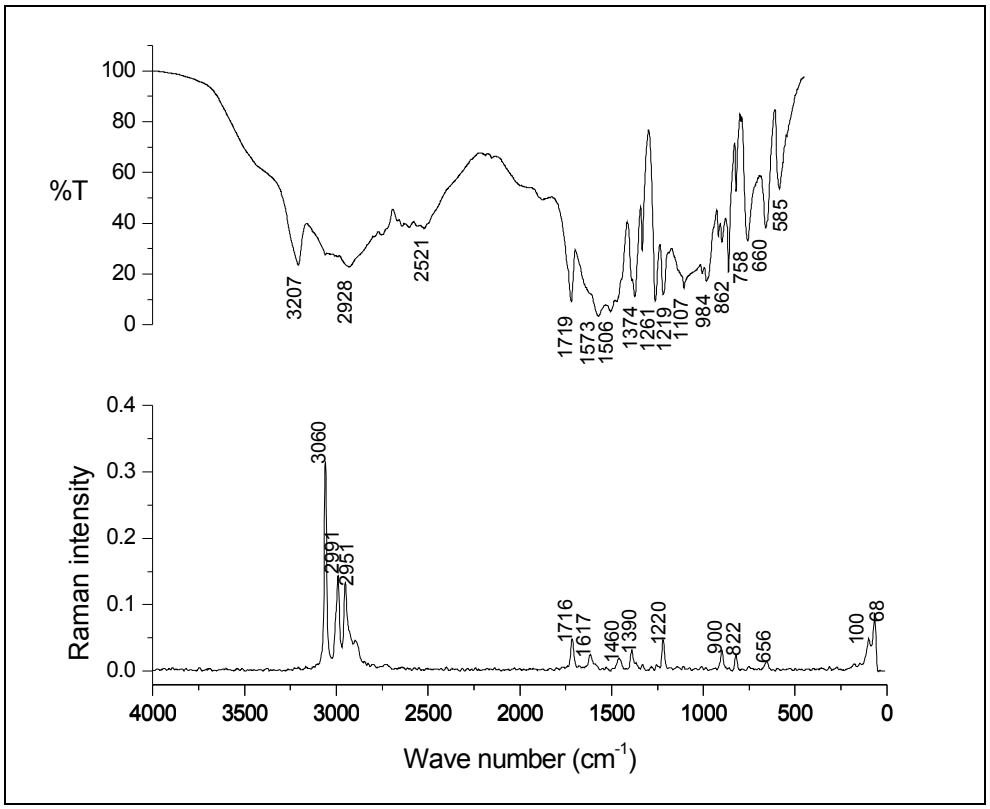

Fig.2: Infrared and Raman spectra of LAMU

Table 1: Assignments for the bands/peaks in FTIR and FT-Raman spectra of LAMU crystal

Wave number (cm^{-1})		Assignments
FTIR	FT- Raman	
3207		NH_3^+asymmetric stretching
	3060	NH_3^+ symmetric stretching
2928	2991; 2951	C-H stretching
1719	1716	C=O stretching
1506		NH_3^+ bending
	1460	Combination band
1374; 1261	1390	C-H deformation
1107		C-O stretching, NH_3^+ rocking
862	822	C-C-N vibration, NH_2 rocking
758		CH_2 rocking
660	656	O-C=O in plane deformation
585		NH_3^+ torsion

The LAMU crystal was tested for their microhardness property using Vickers's microhardness tester fitted with a diamond indenter. The Vickers hardness number (H_v) was calculated from the relation $H_v = 1.8544\ P/d^2\ kg/mm^2$ where 'P' is the load in kg,'d' is the length of the diagonal of the indentation impression in mm. The values of H_v versus applied load are plotted as shown in fig. 3. It was observed that the microhardness increases with increasing load. The Meyer's index 'n' was determined using the relation $P = ad^n$ where a is a constant [4]. A plot of log P versus log d is drawn (Inset in Fig.3) and the value of n was obtained as 4.278. Since the value of n is more than 1.6, the grown crystal belongs to soft category of materials.

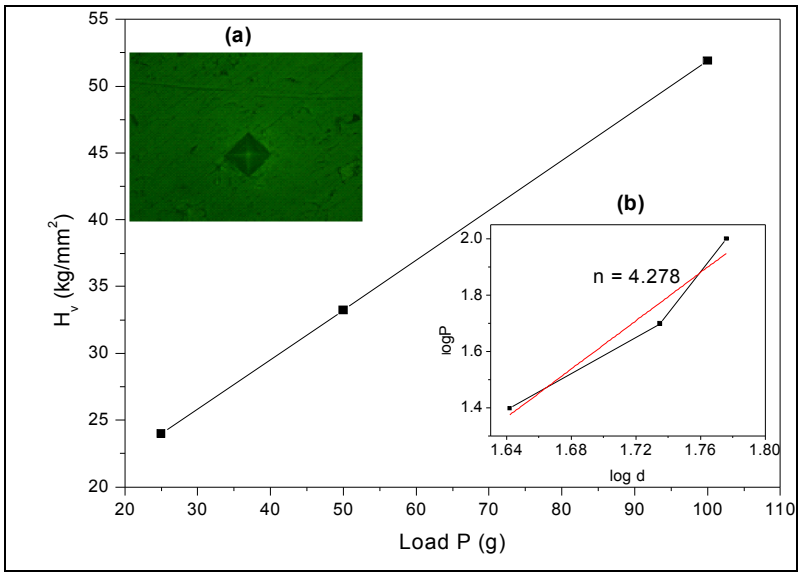

Fig.3: Variation of H_v with applied load for LAMU crystal.
Inset: (a) Indentation Impression made on the crystal and
(b) Plot of log P versus log d for the sample

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