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SYNTHESIS- OPTICAL, THERMAL, DIELECTRIC, HRXRD STUDIES OF L- ALANINE MALEATE

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Abstract— **Single crystals of a potential non-liner optical materials L-Alanine Maleate (LAM) were successfully grown by slow evaporation method. The grown crystals were characterized by X-ray diffraction (single crystals) to detemine the unit cell parameters.The grown crystals were also subjected to Fourier transform infrared spectroscopy(FT-IR) to confirm the presence functional groups in the compound. The optical quality of the grown crystals was analyzed by the UV-VIS Transmission studies. Thermo gravimetric and differential thermal analysis reveal the good thermal stability of the material. The grown crystals have also been subjected to non-linear optical property studies.**

Index terms- **organic compound, low temperature solution growth, Non-liner optical materials.**

I. INTRODUCTION

Non-Liner optics (NLO) has emerged as one of the most attractive fields of current research in view of its vital applications in areas like optical modulation, optical switching, optical logic, frequency shifting and optical data storage for the developing technologies in telecommunications and single processing (1,2). Among the materials producing NLO effects, in particular the second harmonic generation (SHG) the organic materials have been identified to be of considerable importance owing to their synthetic flexibility to design and produce many novel material (3).

The added advantages of organic materials are their large susceptibilities, inherent ultrafast response times and high laser damage thresholds as compared to the inorganic materials. Amino acids are interesting and useful materials for NLO applications. Maleic acid, basically a dicarboxylicacid with large π-conjugation has attracted a great deal of attention. On the basis of earlier reports on L-alanine salts, we have successfully grown L-alanine maleate (LAM), a new organic compound. In this paper growth and its characterization by XRD, FT IR, UV–Visible, dielectric, Hrxrd, NLO and differential thermal analysis are reported for the grown compound

II. EXPERIMENTAL

A. CRYSTAL GROWTH

Good quality single crystal of L-alanine maleate was grown from aqueous solution by slow solvent evaporation method. Highly pure L-Alanine and maleic acid were taken in equimolar ratio and dissolved in double distilled water. The solution is filtered and set for slow evaporation. After 15 days transparent single crystals were obtained from the mother solution.

III. RESULTS AND DISCUSSIONS

A. SINGLE CRYSTAL X-RAY DIFFRACTION

The unit cell parameters are determined using ENRAF NONIUS CAD-4 single crystal X-ray diffraction (XRD) with MoKα radiation. It is observed that L-Alanine maleate single crystal belongs to orthorhombic crystal system with a space group of $P2_12_12_1$.The lattice parameters of LAM are a=5.57Å, b= 7.35 Å, c=23.64 Å and $\alpha = \beta = \gamma = 90°$.

Figure :1 Photograph of single crystals of LAM

B. Fourier Transform infrared (FT-IR) Analysis

The FT-IR spectrum is shown in fig.2 The FT-IR spectrum is recorded using Perkin Elmer spectrum one in the range 450-4000 cm^{-1} by KBr pellet technique. The wavenumber assignment of the various absorption peaks of LAM are listed in Table.2

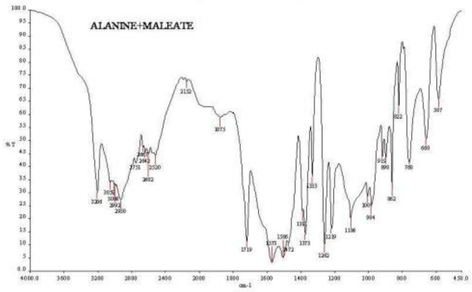

Fig .2 FT-IR spectrum of LAM crystals
Table 2 FT-IR spectrum band assignment of LAM

LAM	Assignment
3206	NH_3^+asymmetric stretching
2930	C-H stretching
1719	COO^- stretching
1506	NH_3^+symmetric bending
1373	C-H deformation in CH_3
1333	O-H plane deformation in COOH
1219	C-COO^- stretching
1106	C-O stretching, NH_3^+ rocking
862	O-H out of plane deformation
760	CH_2 rocking
660	O-C=0 in plane deformation

C. OPTICAL TRANSMISSION

Optical transmission study was carried out using a Shimadzu UV-VIS spectrophotometer. The transmission spectrum of LAM crystals were recorded in the range of 200-1200 nm (Fig.3). The crystal shows 75% transmittance in the range of 300 nm to 1100 nm and the lower out off is found to be low as 310 nm .

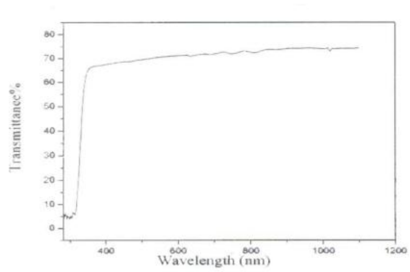

Fig:3 Optical transmittance spectrum of LAM Crystal.

D. THERMAL STUDIES

The Thermo –gravimetric analysis (TGA) and differential thermal analysis (DTA) were carried out for the LAM single crystal using NETZSHCH STA 490 C thermal analyser to study the thermal stability of the crystal. In TG the decomposition of LAM starts from 161˚C onwards An endothermic peak is observed at 160.5˚C in DTA. Fig .4 shows the characteristic curve of TGA and DTA

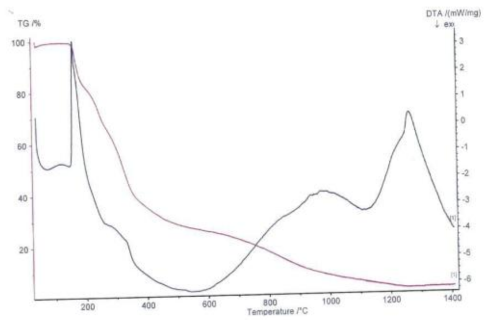

Fig. 4 TG/ DTA Cures of LAM

E. DIELECTRIC STUDIES

The Dielectric studies of LAM crystals were carried out using LCR meter in the frequency range of the 100 Hz to 1MHz for various temperatures. The variation of dielectric constant with frequency is shown in fig 6(a). The dielectric constant (ϵ_r) obtained for LAM is higher at the lower frequencies and then it decreases with the increasing frequencies and saturate for further increase in the frequency which can be attributed to space charge polarization mechanism of molecular dipoles (4). The dielectric constant has higher values at 368 K in accordance with miller rule, the lower value of dielectric constant of higher frequency is a suitable parameter for the enhancement of SHG Co-efficient (5). The variation of dielectric loss with frequency is shown in fig 6(b). The characteristics of low dielectric loss with high frequency for the sample suggests that the crystal possess enhanced optical quality with lesser defects and this parameter plays a vital role for the fabrication of non-linear optical devices(6).

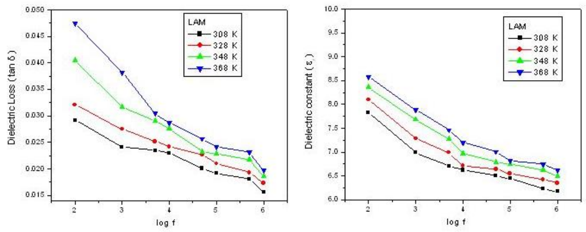

Fig 6(a) & 6(b)

F. HIGH RESOLUTION X-RAY DIFFRACTION (HRXRD)

Fig (7) shows the high-resolution diffraction cure (DC) recorded for a typical single crystal of LAM grown by slow evaporation method, using (200) diffracting planes in symmetrical Brag geometry by employing the multicrystal X-ray diffractometer with

Mokα₁ radiation. The DC (Fig 7) contains a single peak and indicates that the specimen is free from structural grain boundaries. The full-width at half-maximum (FWHM) of the curve is 12 arc seconds which is very close to that expected for an ideally perfect crystal from the plane ware theory of dynamical X-ray diffraction (7) .

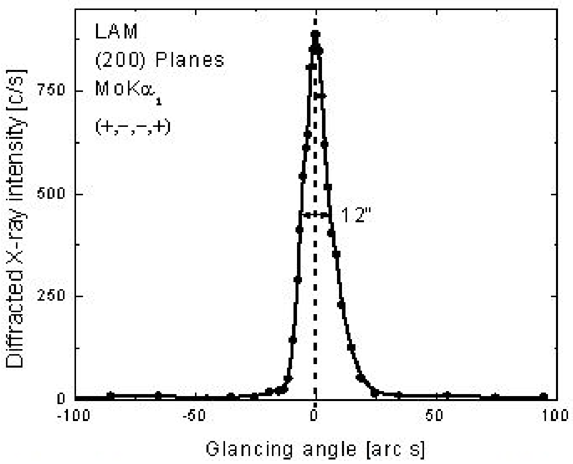

Fig. 7 HRXRD curve for LAM Crystal

G. SECOND HARMONIC GENERATION (SHG)

The powder SHG test was carried out for LAM using Kurtz and Perry technique. Powdered sample of LAM was tightly packed in the microcapillary tubes of uniform diameter (1.5mm) and irradiated by an incident laser radiation 1064nm of pulse width 10 ns and pulse energy of 10 – 800 mJ from a Q-Switched quanta ray of Nd: YAG laser.. The powder SHG efficiency of LAM was found to be 1.2 times that of standard KDP.

IV. CONCLUSION

The NLO crystal of L-alanine maleate was grown by slow evaporation method. Single crystal XRD results gives the unit cell dimensions which agree very well with reported values. FT-IR confirms the presence of functional groups in the compound. TG-DTA studies show the good thermal stability of the material. UV-VIS transmittance spectrum highlights the good transmittance. The value of dielectric constant is found to be as low as 6.2 at high frequencies. The HRXRD analysis conform the good crystalline perfection of the material. From the Kurtz –Perry powder technique the SHG efficiency of the crystal was found to be 1.2 times that of KDP crystal

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