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Growth and Characterisation of L-Aspartate Single Crystals

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ABSTRACT

In the present investigation, single crystals of L-Aspartate were grown successfully by slow cooling method. Powder X-ray diffraction(XRD) studies were carried out for the grown crystal and the lattice parameters were calculated. Absorption of the grown crystal was analysed using UV-VIS -NIR studies and it was found that these crystals possess minimum absorption from 360 nm to 2250 nm. The crystal was characterized by FT-IR and Thermal studies. Nonlinear optical property of the grown crystal was also studied by Kurtz-Perry powder technique.

Keywords: NLO, SHG, Thermal studies, XRD, DTA.

INTRODUCTION

In recent years, Non linear optical materials (NLO) for optical second harmonic generation have received much attention because of their practical application in the field of optoelectronics and photonics [1,2]. Frequency conversion is an important technique for extending the useful wavelength range of lasers [3]. New types of NLO materials have been grown from organic-inorganic complexes. The mechanical strength of these semiorganic NLO crystals are more than the organic NLO crystals [4]. The crystal engineering of this NLO material is based on the anchorage of organic molecules exhibiting a large NLO efficiency on the amino acid groups [5]. Aminoacids are very interesting materials for NLO applications because all of them except glycine contain chiral carbon atom and crystallize in non-centrosymmetric space groups [6,7]. Many number of natural amino acids are individually exhibiting the nonlinear optical properties because they are characterized by a proton-donating carboxyl (-COOH) group and the proton-accepting amino ($-NH_2$) group [8]. Hence they give rise to optical Second Harmonic Generation (SHG). In addition, amino acids have peculiar physical and chemical properties because of their dipolar nature. Already some of the amino acids like L-Arginine phosphate (LAP) have been reported to have NLO activity [9].

As one of the group of naturally occurring α-amino acids, L-Aspartate is a very important compound with molecular formula $C_4H_7NO_4$. In the present study, pure crystals of L-Aspartate are grown by slow cooling method. Good quality, transparent and defect free tiny crystals were formed due to spontaneous nucleation. Powder XRD studies were carried out and lattice parameters of the grown crystal are evaluated. It is observed that L-Aspartate belongs to monoclinic structure with space group $P2_12_12_1$. FT-IR, UV-VIS-NIR and thermal studies were carried out for the grown crystal. The SHG efficiency of the crystal was also studied using Nd:YAG Q-switched laser with the Kurtz Perry powder technique.

MATERIALS AND METHODS

An aqueous solution of L-Aspartate was prepared by dissolving L-Aspartate in distilled water. Pure seed crystals of L-Aspartate were grown by slow cooling method. Good quality seed crystals were used to grow

the single crystals of bigger size. The crystals were found to be transparent and free from defects. Figure1 shows the photograph of the grown crystal with dimensions 1.25cm x 0.5cm x 0.25cm.

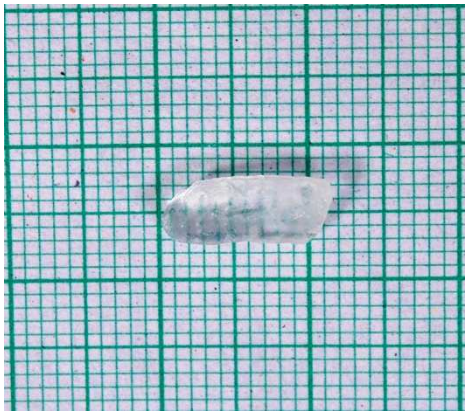

Figure 1 : Photograph of the grown L-Aspartate crystal

RESULTS AND DISCUSSION

3.1 Powder X-ray diffraction

Powder X-ray diffraction studies of pure L-Aspartate crystals were carried out using Siemens D500 X-ray diffractometer with CuKα (λ=1.5418A°) radiation. The L-Aspartate crystal crystallize into the monoclinic crystal system with space group of $P2_12_12_1$. The sample was scanned for 2θ values from 10°- 50° at a rate of 2°/min (Figure 2). The powder pattern were indexed and the lattice parameter values for the grown crystal were calculated. The calculated values and the reported values [10] are presented in Table 1.

Table 1 : Lattice parameter values for L-Aspartate crystal

Lattice parameters	L-Aspartate crystal	Reported Values
a(Å)	7.676	7.617
b(Å)	6.995	6.982
c(Å)	5.211	5.142
Crystal system	Monoclinic	Monoclinic
Space group	$P2_12_12_1$	$P2_12_12_1$
Volume(A^3)	275.93	273.46

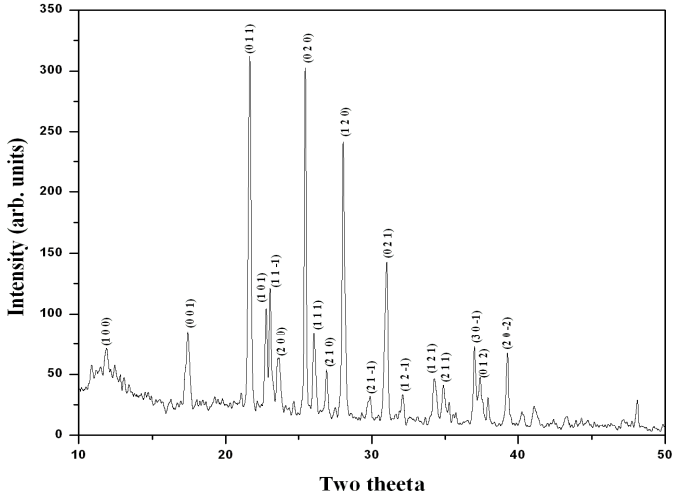

Figure 2 : Powder XRD pattern of L-Aspartate crystal

3.2 FT-IR Specrtoscopic analysis

In order to qualitatively analyse the presence of functional groups, the FT-IR analysis [11] of L-Aspartate was carried out between 400 cm^{-1} and 4000 cm^{-1} using the instrument FT-IR 4100 type A spectrometer. The

spectrum is shown in Figure 3. The broad envelope with few peaks between 1787 cm^{-1} and 3400 cm^{-1} includes overlapping of stretching modes due to N-H and C-H. The peak at 2036 cm^{-1} indicates the asymmetric bend of NH_3^+. The band at 1559 cm^{-1} is due to C=O stretching of carboxylic group, and at 1447 cm^{-1} is due to C-C stretching and at 1375 cm^{-1} is that of COO^- symmetric stretching mode. The peak at 1008 cm^{-1} indicates the C-N stretching mode.

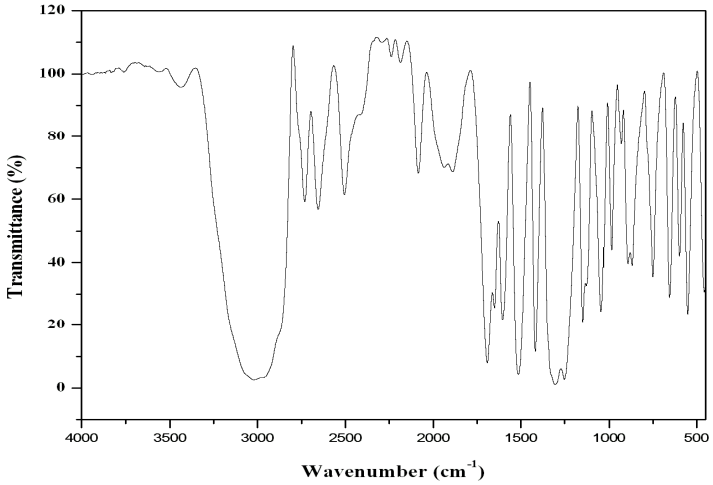

Figure 3 : FT-IR spectrum of L-Aspartate crystal

3.3 UV –VIS – NIR Spectrum

Absorbance spectra is very important for any NLO material because a nonlinear optical material can be of practical use only if it has a narrow absorbance window. To find the absorption range UV-VIS-NIR spectrum of L-Aspartate single crystal was taken in the range between 200 nm and 2500 nm using a varian carry 5E model dual beam spectrophotometer. The spectrum (Figure 4) indicates that the crystal has minimum absorption between 360 nm and 2250 nm. This transparent nature in the visible range is a desirable property for this material to be used for NLO applications. From the spectrum, it is also seen that the lower cut-off wavelength of the crystal is 360 nm. There is no transmittance of light to any appreciable extent in the visible range of the electromagnetic spectrum, which is an intrinsic property of all the amino acids. The transmittance within the UV range, is again a very important property of amino acids [12].

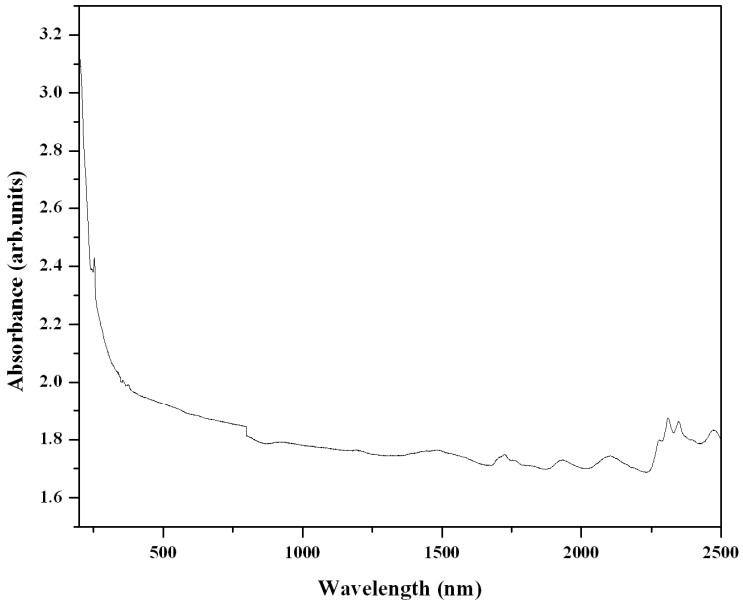

Figure 4 : Optical absorption spectrum of L-Aspartate crystal

3.4 Thermal studies

The thermo gravimetric analysis (TGA) of L-Aspartate single crystal was carried out in the temperature range of 30 °C – 870 °C in the nitrogen atmosphere at a heating rate of 20 °C/min using TGA Q500 instrument (Figure 5). The decomposition starts around 220 °C. The material is found to be thermally stable upto 220 °C. The DTA trace of L-Aspartate shows that, there is a sharp endotherm matching with the decomposition of L-Aspartate crystal. TGA analysis shows that the softening starts at 220 °C. Weight loss of about 35% are observed between 220 °C and 330 °C. The total weight losses of 48% observed between 330 °C and 850 °C corresponds to the decomposition of L-Aspartate single crystal. The decomposition of L-Aspartate single crystal is as shown in Table 2.

Table 2 : Decomposition of L-Aspartate crystal

Temperature range °C	Decomposition steps	Weight loss observed	Calculated
0 °C – 220 °C	$C_4H_7NO_4$	Nil	Nil
220 °C – 330 °C	C_3 H_4 O_2	34.6%	32%
330°C – 850 °C	Entire sample	48.06%	46%

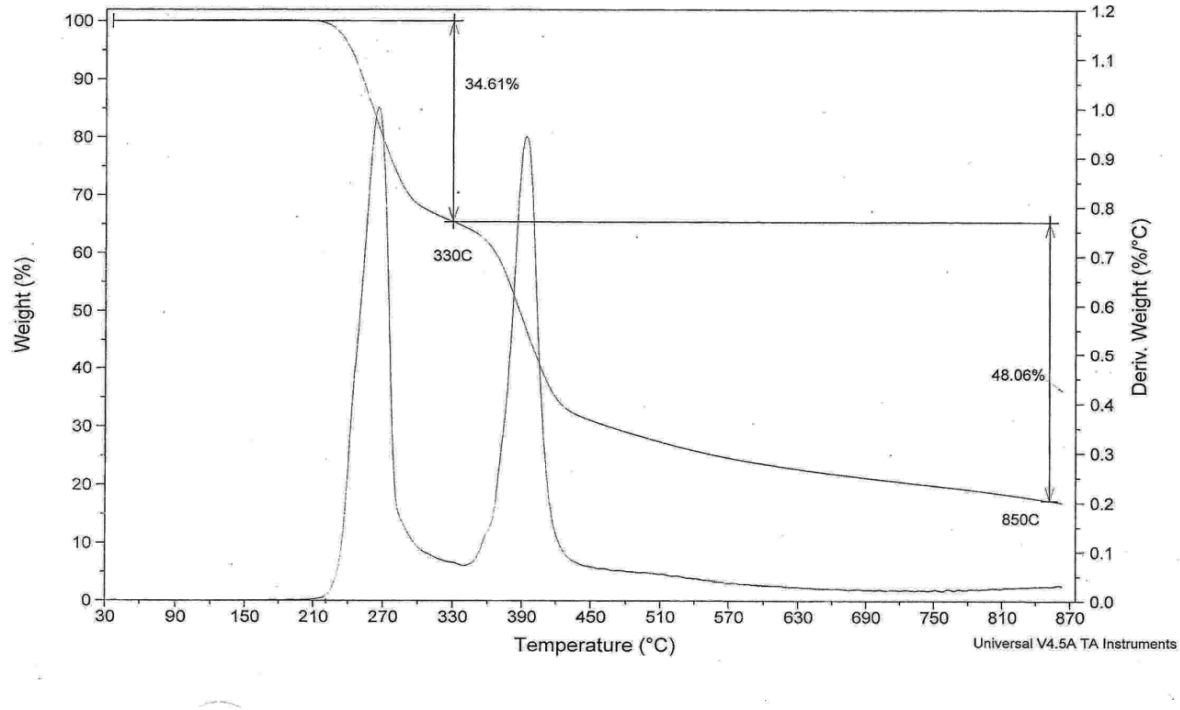

Figure 5 : TGA-DTA curve of L-Aspartate crystal

3.5 NLO studies

The grown crystal of L-Aspartate was subjected to Kurtz-Perry powder second harmonic generation test (SHG) using the Nd-YAG Q switched laser beam for the nonlinear optical (NLO) property [13]. The sample was illuminated with the laser input pulse of 6.8 mJ. The output pulse was obtained as 2.98 mJ. The second harmonic signal(532 nm) was obtained for the sample. The second harmonic generation in the crystal was confirmed by the emission of green radiation from the crystal. Hence it was confirmed that L-Aspartate single crystal is an NLO material. It was found that the efficiency of SHG is 0.44 times that of the standard KDP.

CONCLUSION

Single crystals of L-Aspartate were grown by slow cooling method. Powder XRD studies were carried out and the lattice parameters are calculated. It is found that the calculated values agree well with the reported values. The TGA studies show that the crystal is thermally stable upto 220 °C. The UV-VIS-NIR spectrum shows that the crystal is transparent in the entire visible range and has minimum absorption between 360 nm and 2250 nm. The functional groups present in the crystal was confirmed by the FT-IR spectrum. The second harmonic signal generation proved that the crystal is an NLO material.Thus L-Aspartate crystals can be used as a promising NLO material for the fabrication of photonic and optoelectronic devices.

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