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Growth and Characterizations of nonlinear optical L-arginine hydrobromide Single Crystal

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Abstract

L-arginine hydrobromide (LAHBr) single crystals had been grown by slow evaporation method using water as solvent. The structural parameters of the grown singles crystals were studied by single crystals X-ray diffraction. Thermal stability of the grown crystals was elucidated by differential thermal and thermos-gravimetric (TG-DTA) analysis. The various functional groups vibrations corresponding to LAHBr crystal were seen in FT-IR studies. The optical transparency of the grow crystals were measured by UV-Visible- NIR spectroscopy. The grown crystal mechanical stability and non-linear optical efficiency were observed through the microhardness and Kurtz and Perry tests.

Keywords: L-arginine hydrobromide, X-Ray Diffraction, TG-DTA analysis UV-Vis, microhardness

1. INTRODUCTION

Nonlinear optical (NLO) materials are attracting a great deal of attention because of their use in optical devices especially in frequency convertor. Potassium dihydrogen phosphate (KDP) group of materials remains the most widely used crystals for frequency conversion in spite of their modest nonlinearities [1]. This is primarily due to the low cost and the relative ease with which large volumes of optically homogeneous material can be grown. Hence new type of NLO materials have been built from organic–inorganic complexes in which the high optical non-linearity of a purely organic compound is combined with the favourable mechanical and thermal properties of inorganic materials[2-3]. An improved harmonic generator L-arginine phosphate monohydrate (LAP), one such semiorganic NLO material for laser fusion experiments to replace the conventional KDP crystals was discovered [4]. L-arginine hydrobromide monohydrate (LAHBr) is such salts which can be grown easily and in appreciable sizes [5]. Our intention to do this work was to improve the morphology of the crystals and to see how the optical and other physical properties of the parent crystals get changed due to the growth condition. In these communication detail studies on synthesis, crystal growth, X-ray diffraction, TG-DTA, SHG, transparency range, damage threshold measurement are presented.

2. EXPERIMENTAL PROCEDURE

2.1 Synthesis of L-Arginine Hydrobromide

Analytical grade (AR) L-arginine and hydrobromic acid were taken in equimolar ratio and dissolved in double distilled water to prepare the aqueous solution of LAHBr. The precipitate of synthesized product were collected and used for further growth procedure.

2.2 Solubility studies

L-arginine hydrobromide was taken and dissolved in distilled water. Solubility studies were carried out in a constant temperature bath with a cryostat facility. Stirring was achieved using an

immersible magnetic stirrer. The solution was stirred continuously for 6 h. Solubility studies for different temperatures (35, 40, 45 and 50 ºC) have been carried out. Solubility was determined by gravimetric analysis and the solubility curve of LAHBr is shown in Fig. 1. It has been found that the solubility of LAHBr was high in water. From the figure 57.6 g of LABHr salt was found to dissolve in 50 ml of water at 30 ºC for further growth. Using the solubility the crystals were grown by slow evaporation technique.

2.3 Growth of LAHBr single crystals

The starting material L-arginine hydrochloride was dissolved in Millipore water. 500 ml of saturated solution of LAHBr at 45 ºC was prepared. Using a glass filter of 1µm porosity, the solution was filtered using a peristaltic pump. Seeds obtained from slow evaporation technique which are defect free were selected for growth. The growth was carried out in a constant temperature bath of controlling accuracy ±0.01ºC. A cooling rate of 0.1–0.3 ºC/day was employed in the initial and final stages of the experiment. Once the room temperature is reached the crystal is harvested. A crystal of dimension 4.5x4x2 cm^3 has been grown and is shown in Fig. 2.

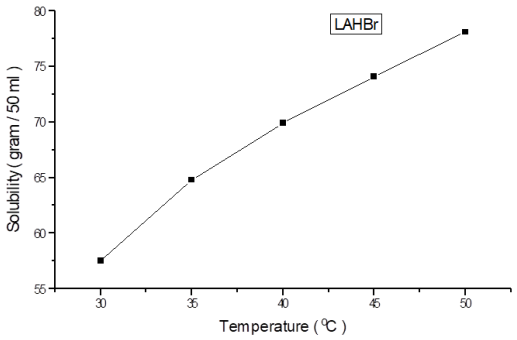

Fig. 1. Solubility of LAHBr in water at various temperatures

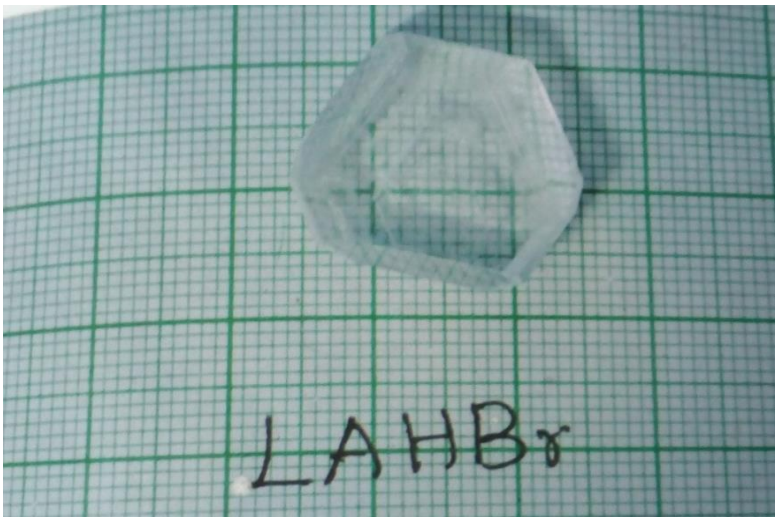

Fig. 2 Photograph of as grown LAHBr single crystal

3. RESULTS AND DISCUSSION

3.1. Single Crystal X-Ray Diffraction Studies

Single crystal X-ray diffraction studies of pure LAHBr crystal was carried out using MESSRS ENRAF NONIUS CAD4-F, single X-ray diffractometer. Both the pure and doped LAHBr crystals belong to monoclinic crystal system with the space group $P2_1$. The lattice parameter value of crystals were calculated to be a= 11.1931 Å, b=8.6149 Å, 11.2308 Å, α= 90º β= 91.717º and γ= 89.9986º and Z=2.

3.2. Fourier Transform-Infrared Spectroscopy (FT-IR)

The fine powdered samples of pure LAHBr crystal are taken for this experiment. The FT-IR spectrum is recorded for the pure and doped LAHBr single crystals at room temperature in the range of 400–4000 cm^{-1} by employing BRUKKER IFS 66V FT-IR spectrometer, using KBr pellet method. The FT-IR spectra of pure and doped crystals are shown in Fig. 3. It can be noted from the spectrum that the broad envelope in the higher energy region consists of peaks due to NH vibrations at 3333.4 and 3172 cm^{-1}. The broad envelope contains peak at 2955.7 cm^{-1} due to CH vibration. The peak observed at about 2100 cm^{-1} is due to the combination of asymmetric NH_3^+ vibrations observed at 1590.9 cm^{-1} and 1534.1 cm^{-1}. The C=O vibration of the carboxyl group is observed at 1669.9 cm^{-1}. The symmetrical NH_3^+ vibration is positioned at 1523.3 cm^{-1}. The CH_2 bending modes produce peaks at 1468 and 1354 cm^{-1}. The C-N vibrations produce a group of peaks between 1290 and 1022 cm^{-1}. Our investigations were well compared with earlier reports [6-7].

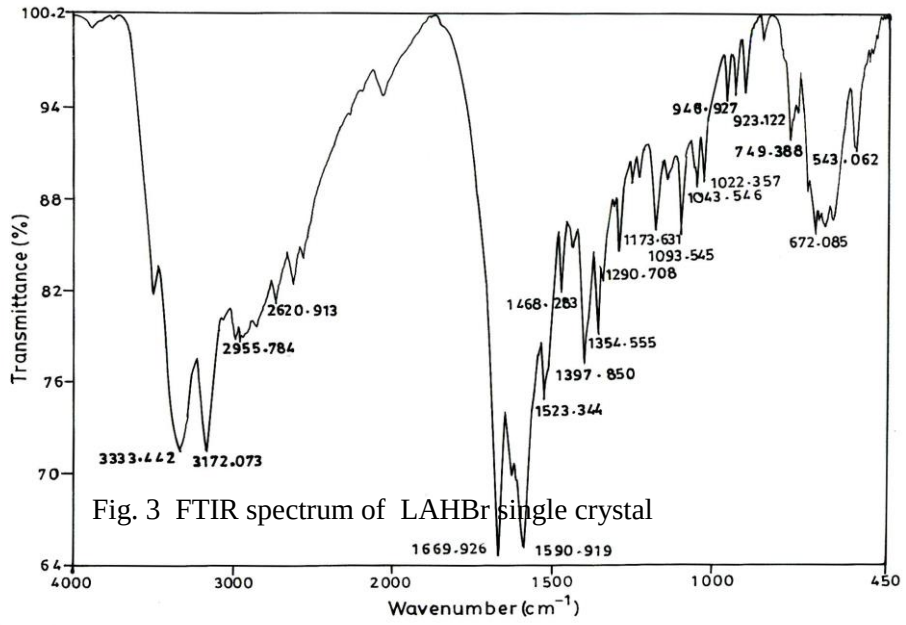

Fig. 3 FTIR spectrum of LAHBr single crystal

Fig.3.FTIR spectrum of LAHBr single crystal

3.3. UV Analysis

The UV absorption study of the pure and doped LAHBr crystals was carried out by UV-VIS-NIR spectrophotometer. Fig.4 shows that the absorption is high (≈3.5 arbitrary unit) in the near IR region,

which may be assigned to overtones and combinations. The entire visible region is highly transparent without any absorption and this is a desirable property of the materials chosen for NLO applications.

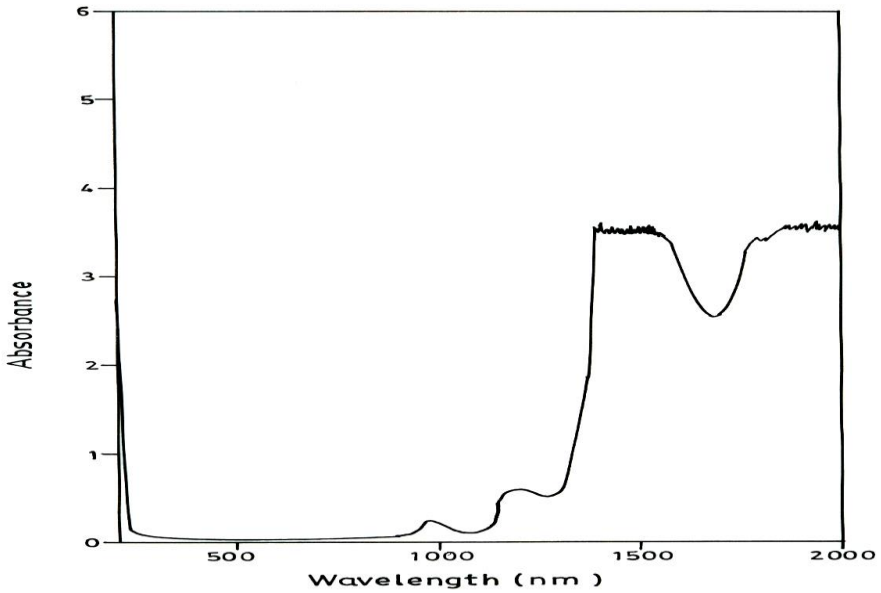

Fig.4 UV-Vis-NIR spectrum of LAHBr single crystal

3.4 Thermal Studies

The thermogravimetric analysis of LAHBr was carried out from room temperature and 1400°C in nitrogen atmosphere. The TG-DTA patteren are shown in the Fig. 5 and it explained the various weight losses corresponding reaction peaks.

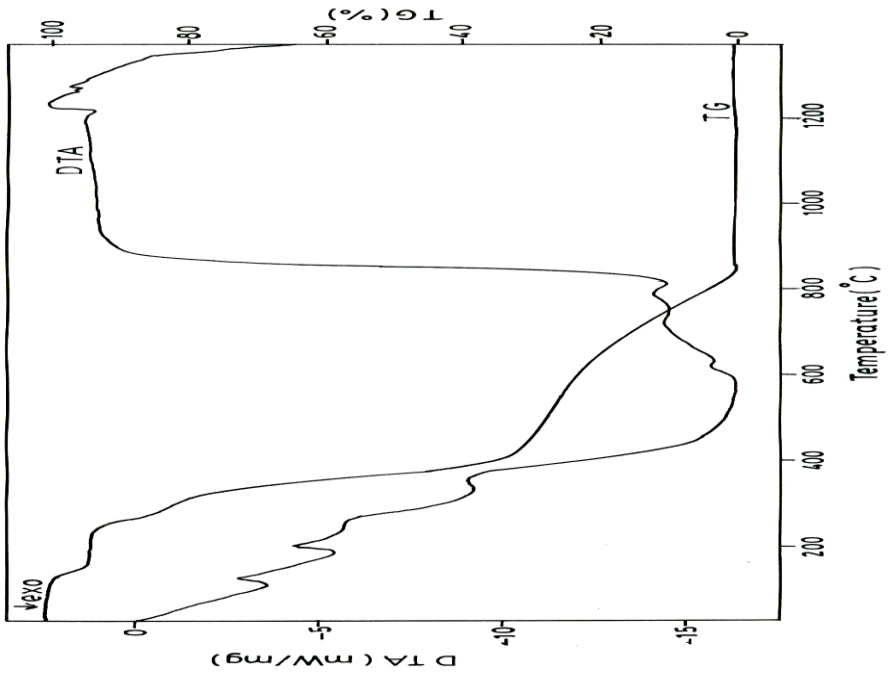

Fig. 5. TGA and DTA pattern of LAHBr single crystal

3.5 NLO Test

Kurtz SHG test was carried out on the LAHBr single crystal to study its NLO property. The sample was illuminated using Q–switched, mode locked Nd:YAG laser with the first harmonic output of 1064 nm. The emission of green radiation from the crystal confirmed the second harmonic signal generation in the crystal

CONCLUSION

LAHBr single crystals have been grown using water as solvent by the slow cooling method. Solubility of LAHBr has been carried out for different temperatures. The solubility of the solvent in the above solute is reduced considerably. Lattice parameter values were found using powder and single crystal X-ray diffraction analyses. TGA showed the presence and absence of water in the L-arginine hydrobromide monohydrate and L-arginine hydrobromide crystals, respectively. Optical transmission studies show an increased percentage of transmittance for LAHBr using water as solvent. FTIR studies confirmed the various functional groups present in the crystals. SHG was confirmed by Kurtz and Perry tests by produced the Nd:YAG 1064 nm output was up converted to 532 nm green light.

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