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Synthesis, Growth and Physicochemical Properties of a New Semiorganic Nonlinear Optical Crystal L-Arginine Barium Chloride

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Abstract: Semi organic nonlinear optical L-Arginine Barium Chloride (LABC) crystal has been synthesized using L-arginine and barium chloride for the first time. The LABC single crystals were grown from aqueous solution by slow evaporation technique at room temperature. The grown crystal was subjected to various characterizations. The grown LABC crystal was analyzed using single X-ray diffraction to identify the crystal system. From the calculated parameters, the LABC crystal was found to be orthorhombic in structure. The grown crystal of LABC has good crystalline nature which was observed from powder diffraction technique (XRD) study. The vibration frequencies of the functional groups are identified by FTIR spectroscopic study. The LABC crystal having good optical transmission in the entire visible region and hence LABC crystal may suitable candidate for optical device fabrication. Non linear optical property of the crystal was confirmed by SHG test using Kurtz Perry powder technique. Studies of dielectric properties like dielectric constant and dielectric loss as function of frequencies for varying temperatures has been carried out and the results suggested that LABC crystal is good candidates for electro optic modulators and frequency convertors.

Key words: XRD, Optical property, SHG, Dielectric

I. INTRODUCTION

Non linear optics plays an important role in the emerging photonic and optoelectronic technologies. Non linear optical materials find wide applications in the area of laser technology, optical communication and the data storage technology. Some organic compounds exhibit large NLO response, in many cases, order of magnitude larger than widely known inorganic materials. They also offer the flexibility of molecular design and the promise of virtually an unlimited number of crystalline structures. In this stimulating context, organic nonlinear materials have been recognized as a forefront candidate for fundamental and applied investigations involving, in a joint effort, chemists, material scientists and optical engineers [1-5]. This research is extended to semi-organic NLO material crystal so as to obtain superior NLO crystal by combining the advantages of organic and inorganic materials. Hence Semi-organic single crystals are attracting great attention in the field of non linear optics because of their high optical nonlinearity, chemical flexibility of ions, high mechanical strength, thermal stability and excellent transmittance in the UV-Vis region [6-8]. Moreover amino acid family crystals are still gaining importance because of its high second order nonlinear optical coefficients, wide optical transparency and multifunctional substitutions [9-10]. Most of the amino acids individually exhibit the NLO property due to donor amino group NH_3^+ and acceptor carboxyl group COO^- and intermolecular charge transfer [11]. Therefore, amino acid enhances the material properties such as nonlinear and ferroelectric properties [12]. Amino acid family with metal chlorides such as zinc chloride [13], calcium chloride [14], potassium chloride [15], sodium chloride [16], and lithium chloride [17] have been reported in the recent years. Interest have been centered on semiorganic crystal which have the combined properties of both inorganic and organic crystals which make them suitable for device fabrication [18]. In this present investigation we concentrated on synthesis, growth and characterization of a new semiorganic nonlinear optical crystal L-Arginine Barium Chloride (LABC). The grown crystal LABC were characterized using single crystal XRD, powder X-ray diffraction, Fourier transform infrared (FT-IR) analysis, UV-vis spectroscopy and also dielectric constant, dielectric loss have been determined for the first time.

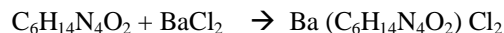
II. EXPERIMENTAL PROCEDURE

A. Synthesis

A analytical reagent grade of L-arginine and barium chloride were synthesized using double distilled water by adding equimolar ratio (1:1) of L-arginine and barium chloride to form a aqueous solution of LABC the solution was stirred using magnetic stirrer for

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6 hours to obtain the homogeneous solution at room temperature. The chemical reaction of synthesized compound was shown below,



B. Crystal growth

The saturated solution of L-arginine barium chloride (LABC) was filtered using good quality micro filter paper to remove the insoluble impurities. The filtered solution was kept into a beaker and sealed with a polythene sheet with few holes for slow evaporation. The beaker was kept in an undisturbed and dust free environment. After the time period of 35 days the LABC crystal were grown with dimension 9 mm × 6 mm × 4 mm. The as grown crystal of LABC is shown in the figure 1



Figure 1. As grown crystal of LABC

III. CHARACTERIZATIONS

A. Single crystal X-ray diffraction

Single crystal X-ray diffraction analysis of L-arginine barium chloride (LABC) was recorded using ENRAF NONIUS CAD-4 diffractometer. The calculated lattice parameters are $a=5.281\text{ \AA}$, $b=5.410\text{ \AA}$, $c=14.898\text{ \AA}$, $\alpha=\beta=\gamma=90^\circ$ and volume $V= 425.638\text{ \AA}^3$ which confirm the orthorhombic crystal system.

B. Powder X-ray diffraction analysis

Powder sample of LABC crystal was subjected to powder X-ray diffraction studies with $\text{CuK}\alpha$ ($\lambda=1.5406\text{ \AA}$) radiation. The powdered sample was scanned in the range $10\text{--}80^\circ$ at a scan rate of 1° per minute. A well defined Bragg's peaks observed in the powder XRD pattern reveals that the grown crystal has highly crystalline nature. The recorded powder XRD pattern of the grown LABC crystal is shown in Figure 2.

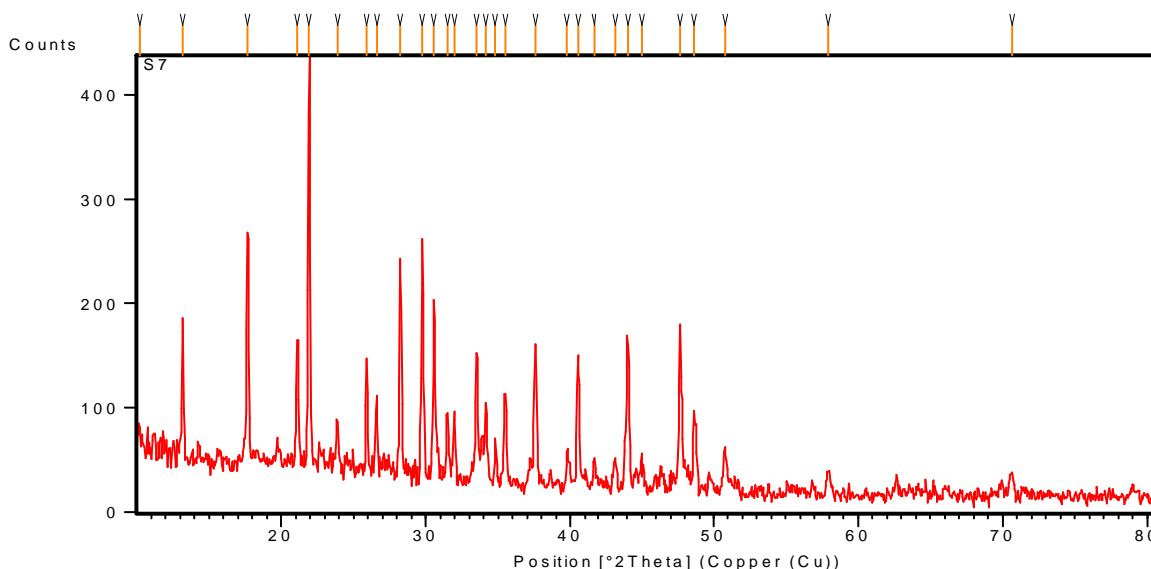


Figure 2. Powder XRD pattern LABC crystal

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C. Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR analysis is used to identify the functional groups present in the grown crystal LABC by using thermo Nicolect v-200 FTIR spectrometer by KBr pellet method in the range 500-4000 cm^{-1} . In order to analyse qualitatively the presence of functional groups, freshly crushed powder of LABC crystal was subjected to FTIR studies. Figure 3 shows FTIR spectrum of LABC crystal. The absorbed frequencies and their assignment of LABC crystals are shown in the Table 1. The broad band in the higher energy region around 3164 cm^{-1} is due to NH_3^+ asymmetric stretching. The strong but broad peaks observed at 2931 cm^{-1} due to presence of C-H asymmetric stretching. The C-H stretching and bending identified at 2620 and 1352 cm^{-1} respectively. The absorption band at 2113 cm^{-1} is due to the combination of NH_3^+ deformation and NH_3^+ torsion. The peak at 1615 cm^{-1} is attributed to NH_3^+ asymmetric deformation. The peak observed at 1508 cm^{-1} implies NH_3^+ stretching. The peak observed at 1282 cm^{-1} is due to C-N-stretching. The C-C stretching confirmed at 996 and 908 cm^{-1} respectively. The C-C-N symmetric and asymmetric stretching is found at 861 and 1096 cm^{-1} . The band appearing at 739 cm^{-1} infers the C-O-H stretching of the pure crystals which are all very well agreed with literature. The absorption peak at 668 cm^{-1} is due to the C-Cl stretching. The assignments confirm the presence of various functional groups present in the material.

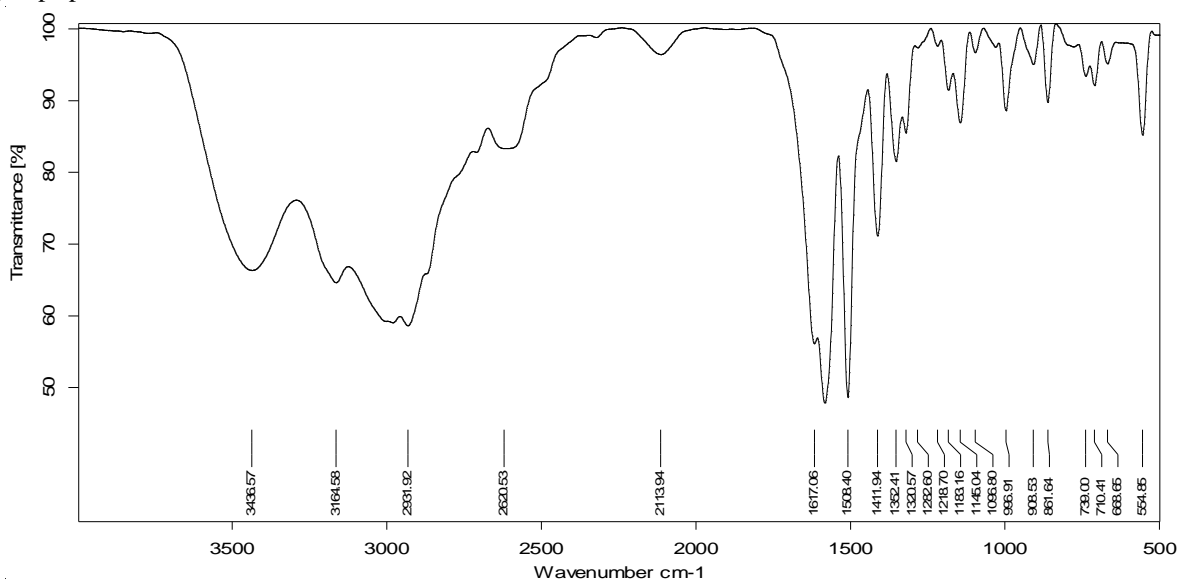


Figure 3 FTIR spectrum of LABC crystal

Table 1 Wavenumber Assignments of LABC crystal

| Wavenumber cm^{-1} | Assignments |
|-----------------------------|--|
| 3436 | O-H bending |
| 3164 | NH_3^+ asymmetric stretching |
| 2931 | C-H asymmetric stretch |
| 2620 | C-H stretching |
| 2113 | Combination of NH_3^+ deformation and NH_3^+ torsion |
| 1617 | NH_3^+ asymmetric deformation |
| 1508 | NH_3^+ stretching |
| 1411 | COO^- symmetric stretching |
| 1352 | C-H bending |
| 1282 | C-N stretching |
| 1145 | NH_3^+ rocking |
| 1096 | C-C-N asymmetric stretching |
| 996 | C-C stretching |
| 861 | C-C-N symmetric stretch |
| 739 | C-O-H stretching |
| 668 | C-Cl stretching |

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D. UV-vis – NIR spectrophotometer analysis

The optical transmission spectrum was recorded using DOUBLE BEAM UV-vis Spectrophotometer:2202 in the region 200-1000 nm and the optical transmission spectrum of L-arginine barium chloride is shown in Figure 4. The transmission is maximum in the entire visible region and infrared region. In LABC crystal, the UV transparency cut-off wavelength lies at 228 nm and the percentage of transmission is high in the entire visible region from 228 nm to 1000 nm. The absence of absorption in the entire visible region makes the L-arginine barium chloride crystal as a potential candidate for second harmonic generation and various applications [19].

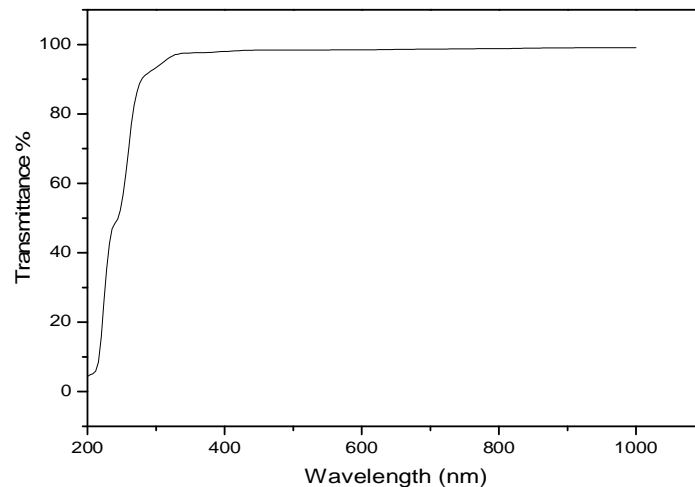


Figure 4. Optical transmission spectrum of LABC crystal

E. Nonlinear optical study

In order to confirm the non-linear optical property of powdered sample of LABC crystal was subjected to KURTZ and PERRY techniques, which remains powerful tool for initial screening of materials for SHG efficiency [20]. A Q-switched Nd: YAG laser emitting $1.06\mu\text{m}$ with power density up to $1\text{ GW}/\text{cm}^2$ was used as a source of illuminating the powder sample. The sample was prepared by sandwiching the graded crystalline powder with average particle size of about $90\mu\text{m}$ between two glass slides using copper splices of 0.4 mm thickness. A laser was produced a continuous laser pulses repetition rate of 10Hz. The experimental setup uses a mirror and 50/50 beam splitter. Here well known NLO crystal KDP is taken as a reference material.

The fundamental beam was splitted into two beams by the beam splitter (BS); one of them was used to illuminate the powder under study and the other constituted the reference beam of power P_0 . Half-wave plate (HW) placed between two parallel polarizers (P) and was used to pump the beam power.

The input power was fixed at 0.68 J and the output power was measured and compared to output 8.8 mJ of standard KDP. The diffusion of bright green radiation of wave length $\lambda=532\text{ nm}$ ($P_2\omega$) by the sample confirms second harmonic generation (SHG). The powder SHG efficiency of LABC crystal was confirmed by emission of green light. This indicates that the LABC crystals can be used as a suitable material for non-linear optical devices.

F. Dielectric studies

The dielectric constant and the dielectric loss of the LABC sample were measured using HIOKI 3532-50 LCR HITESTER. Dielectric constant and dielectric loss of the sample have been measured for different frequencies (100 Hz to 5 MHz) at different temperatures (308 to 368 K). Figure 5 and Figure 6 show the variations of dielectric constant and dielectric loss respectively as a function of frequency at different temperatures. It is observed from Figure 5 that the dielectric constant (at 308 K) decreases with increase in frequency from 100 Hz to 10 kHz and then attains a constant value of 27.82. The same trend is observed for other temperatures too. It is also observed that the value of dielectric constant increases with temperature. Such variations at higher temperature may be attributed to the blocking of charge carriers at the electrodes. The decrease of dielectric constant at low frequency region may be due to space charge polarization. Figure 6 indicates that as the frequency increases, the dielectric loss decreases exponentially and then attains a lower value of 0.049 at 308 K. The low value of dielectric loss confirms that the sample

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possesses lesser defects.

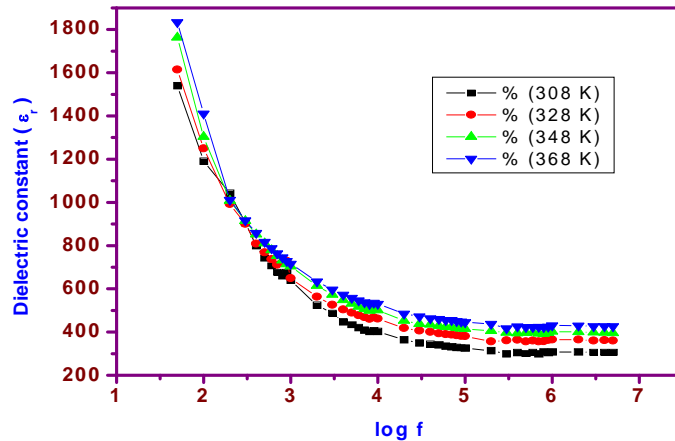


Figure 5. Variation of dielectric constant with log frequency at different temperatures for LABC crystal

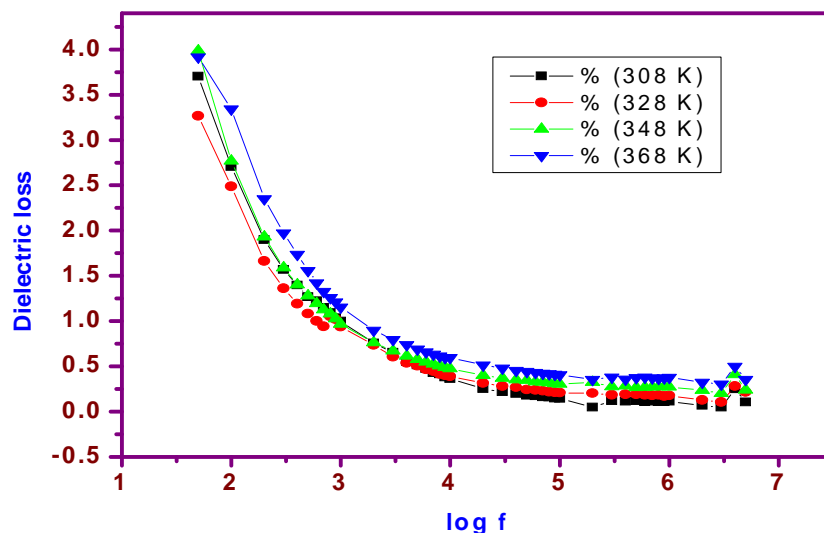


Figure 6. Variation of dielectric loss with log frequency at different temperatures for LABC crystal

IV. CONCLUSION

A new semi organic nonlinear optical (SONLO) crystal L-arginine barium chloride (LABC) were successfully grown employing slow solvent evaporation technique. A good quality crystal of LABC with dimension 9 mm x 6 mm x 4 mm was harvested in a period of 35. The various functional groups in LABC crystal was identified using FTIR analysis. The UV-vis- NIR spectral analysis showed good transparency in the UV visible region. The UV cut-off wavelength lies at 228 nm. The powder SHG efficiency of LABC crystal was confirmed by emission of green light. This indicates that the LABC crystals can be used as a suitable material for non-linear optical devices. The dielectric constant and dielectric loss measurements of LABC crystal revealed the normal behaviour. The low value of dielectric loss confirms that the sample possesses lesser defects.

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