International Journal of Engineering Technology Science and Research
IJETSR
www.ijetsr.com
ISSN 2394 – 3386
Volume 5, Issue 4
April 2018

Growth, Structural, Optical and Mechanical Properties Of L- Leucine Hydrobromide: A NLO Crystal

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Abstract:
L-Leucine hydrobromide, crystals were synthesis by taking L-Leucine(CDH) and Hydrobromic acid (Ranbaxy) in equimolar ratio and grown by slow evaporation solution technique. Growth conditions were optimized. Good quality crystals with high transparency were obtained after a span of 28-30 days. Nucleation kinetic studies was carried out to understand the nucleation process. The crystal structure and lattices parameters were analyzed by powder XRD and single crystal XRD and are found in good agreement with the available literature. The presence of various functional groups is confirmed by Fourier Transform Infra Red (FTIR) study. The grown crystals were characterized by TGA/ DTA analysis, Kurtz powder test and photoconductivity study. TGA study reveals that the grown crystals are stable up to 215^0 C. The crystals are having very high second harmonic generation (SHG) efficiency approximately 4 times more than KDP crystal. In the present work authors report the UV-VIS analyses to determine the transmission range and other optical constants. FTIR Spectroscopy study is also carried out to confirm the presence of various functional group in grown the crystals.

Keywords: XRD, FTIR, TGA/ DTA, UV-VIS.

1. Introduction:

In the present scenario, non-linear optical crystals are highly demanded by scientist and researchers because of their large application in photonic devices including optical communication data storage device, signal processing and instrumentation [1-2]. Semi- organic crystals are highly preferred as they possess property of both organic and inorganic material. Organic crystals have high value of second harmonic generation efficiency whereas inorganic crystal have high mechanical strength, high thermal stability range and high transmission range. Most of the amino acid family and their derivatives belong to organic and semi-organic non linear optical material. L-Leucine is the simplest molecule of amino acid family having SHG effienciency of about 4 times more than KDP crystal. In the present study, we attempt to syntheses and grow L-Leucine Hydrobromide crystal single crystal by slow evaporation solution technique. The grown crystals are analyzed by powder XRD, thermal studies, and mechanical strength by Vicker Microhardness test. NLO behavior has been confirmed by Kurtz and Perry powder test and second harmonic efficiency was measured.

2. Experimental Part:

2.1 Synthesis of LEHBr single crystal:

To synthesis LEHBr we take L-Leucine (CDH) and hydrobromic acid(CDH) of AR grade having purity 99 % are used as the starting material and taken in equimolar ratio 1:1. The calculated amount of L-Leucine and hydrobromic acid is dissolved in doubly distilled water till it completely dissolved and super-saturation achieves. The solution is stirred with the help of magnetic stirrer for 5-6 hours to make it homogenous. The solution is filtered and kept for evaporation in a oven at constant temperature 40^0 C. After 5-6 days the synthesize salt of L-Leucine hydrobromide is obtained which is used for crystal growth.

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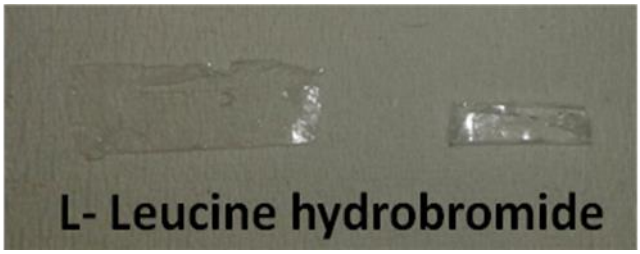
2.2 Crystal Growth:

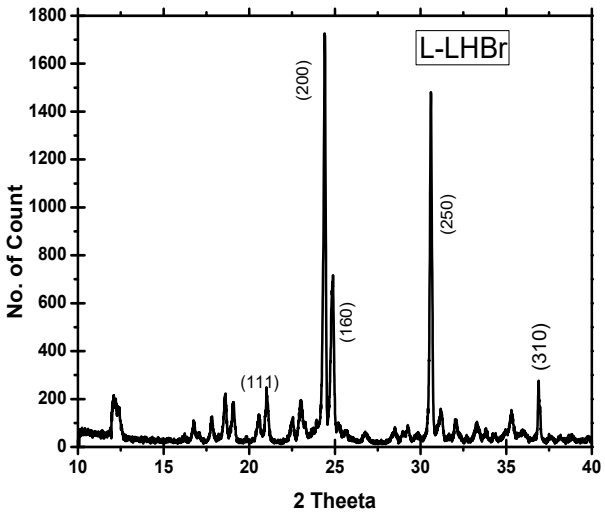
The synthesized salt of L-Leucine hydrobromide salt was taken and dissolved in doubly distilled water with the help of magnetic stirrer. To make the solution homogenous, solution is stirred for 5-6 hours. The solubility of synthesized salt is measured at room temperature and recrystallization process was carried out three times to reduce the impurity in the compound. The filtered solution is kept in a well cleaned beaker and placed in a constant temperature bath with a provision of constant evaporation. Due to spontaneous evaporation nucleation process starts and after a span of 25-28 day colourless needle shaped crystals have been made. Sevral attempts have been made to grow good quality crystals with appropriate larger size.

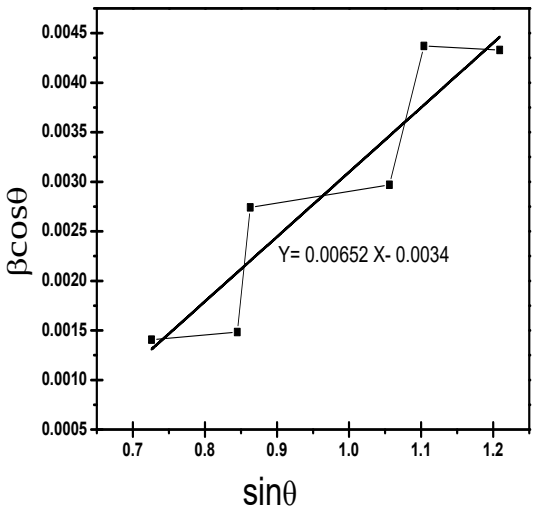


3. Characterization:

3.1 Powder XRD:

The powder X-ray diffraction measurement has been carried out for the powder specimen of L-Leucine hydrobromide. The powder specimen was subjected to Bruker D-8 Advance X-ray diffractometer. CuK-alpha source was used and measurement has been carried out between 10-80 degrees at ambient conditions. The recorded powder XRD pattern is shown in Fig. 1. From the measurement one can understand that the peaks are very sharp and indicate that the grown specimen is having good crystalline nature. The observed two theta values are given as the input in Chekcell software and the lattice dimensions were calculated. The calculated lattice dimensions are good in agreement with the reported literature values.





Using the data obtained from powder XRD analysis a curve is plotted between sinθ and βcosθ known as W-H plot. From the W-H plot, value of mechanical strain present in the crystal were calculated andfound equal to + 1.37 x 10^{-3}. Hence, the positive value of mechanical strength present in the crystal indicates that point defects are present in the grown crystal.

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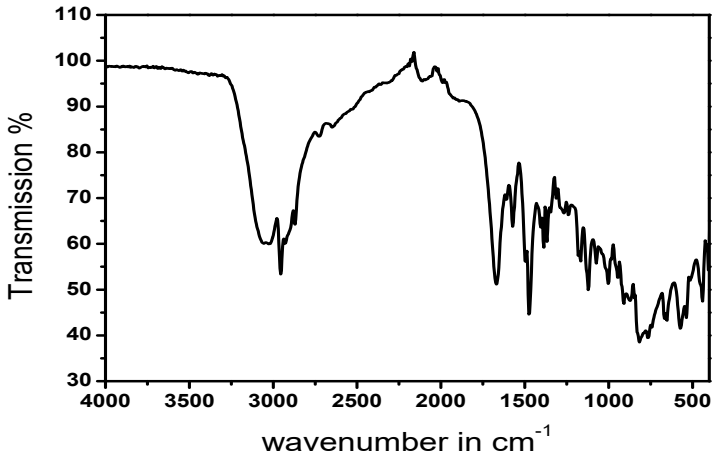
. 3.2 FTIR Analysis:

The presence of functional groups which is formed during the synthesis process was confirmed by fourier transform infrared analysis. The grown single crystal was crushed into fine powder and then mixed with potassium bromide by adopting Agilant Cary 630 FTIR spectrophotometer. The recorded FTIR spectra is shown in Fig. 2. From the pattern we have observed that 2849 cm-1 and 2956 cm-1 is assigned to symmetric and asymmetric stretching of CH2. The intense peak observed at 1482 cm-1 is assigned to NH3 symmetric deformation. The peak at 1244 cm-1 is assigned to NH3 rocking of the title compound. CH3 group is observed at 1340 cm-1. The peak at 824 is assigned to CH deformation.



4.5.7 UV-Vis NIR spectroscopy:

A good quality single crystal is first well polished with alumana powder in order to remove the surface disorder and then scanned by a UV-Vis-NIR spectrometer in transmition mode. The observed spectra is shown in figure 4.10. The figure clearly reveals that the grown crystal has a very high value of transmittance in the large wavelength range of 300 nm to 1200 nm. Hence, this much high value of observed transmitance indicates that the absorption is negligible small this further reveals that the grown crystal is suitable for second harmonic generation application of Nd:YAG laser of wavelength λ = 1064 nm and also suitable for frequency multiplier in the range 300 nm to 1200 nm. At wavelength 800 nm sharp decrease is observed due to light source switch and it is not the property of material.Furthermore, the obtained result confirm that LLHBr possess the property of extended UV-Vis transmittance and does not absorb light in a wide range of wavelength.

At 225 nm, there is rapid increase in transmittance and the transmission edge is existed at this value. The % transmission in the whole visible range is found good nearly 45 %. Besides this good value of transmittance, Tauc plot has been plotted and various other important optical parameters like optical band gap, reflectance, extinction coefficient, refractive index, electrical susceptibility and optical dielectric constant were also calculated. With the help of recorded UV-Vis data, absorption coefficient (α) is determined by the following relation:

$$\alpha = \frac{2.303 * \log\left(\frac{1}{T}\right)}{t} \qquad \ldots.(4.11)$$

here t assumed thickness of our speciman used. Using the value of absorption coefficient and transmission data, we plotted a graph between E_g and $(\alpha h\nu)^2$, known as Tauc plot. Tauc plot is shown in figure. A straigth line is drawn tangent to the curve and the corresponding intercept on E_g axis yields the value of band gap. The

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observed value of band gap is found 5.28 eV which is very near to the already reported value. Similarly, value of extinction coefficient is determined as

$$K = \frac{\lambda\alpha}{4\Pi} \qquad \text{....(4.12)}$$

The value of reflectance is assessed from the transmission data and absorption coefficient and it is determined as follow:

$$R = \frac{\exp(-\alpha t) + \sqrt{\exp(-\alpha t)T - \exp(-3\alpha t)T + \exp(-2\alpha t)T^2}}{\exp(-\alpha t) + \exp(-2\alpha t)T} \qquad \text{....(4.13)}$$

Refractive index of the material is also calculated using the value of reflectance by the equation

$$n = -(R-1) + \frac{2\sqrt{R}}{(R-1)} \qquad \text{....(4.14)}$$

Using the value of refractive index and extinction coefficieent, electrical susceptibility χ_c is calculated by the formula

$$\chi_c = \frac{n^2 - K^2 - \varepsilon_0}{4\Pi} \qquad \text{....(4.15)}$$

Using the above formula, value of reflectance and refractive index is found equal to 1.66 & 2.88 respectively. Whereas electrical susceptibility is 0.6640. The value of extinction coefficient was found 3.9756 x 10^{-4}. The real and imaginary part of dielectric constant are 8.339 and 0.00229 at 850 nm respectively.

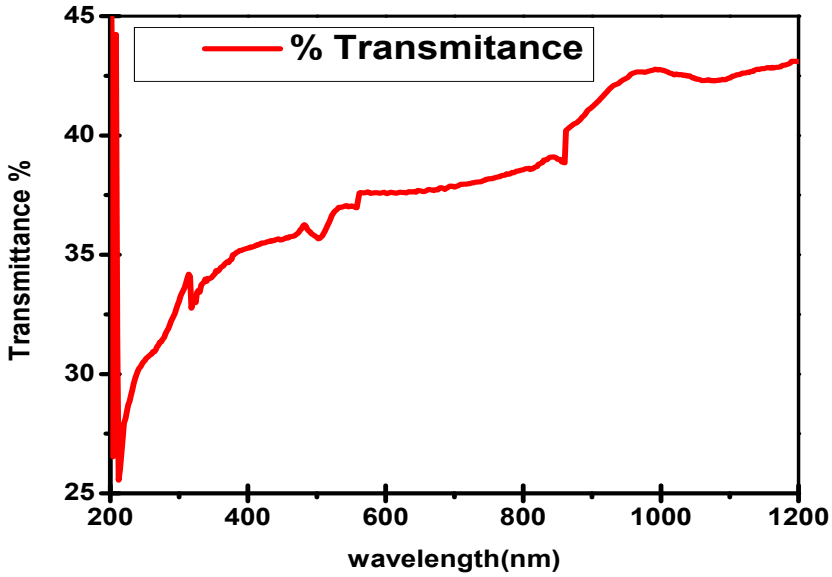

Figure 4.10 Transmission spectra of LLHBr

3.3 NLO Studies:

Finely cursed powder of the grown crystal is subjected to Kurtz and Perry Test in which a Q- switched laser Nd^{3+}:YAG emitting 1.06 μm radiations is used. The input laser beam was passed through IR reflector and made incident on powdered sample placed in a capillary tube of diameter 0.154mm. The bright green emission is confirmed that the title compound is having good second harmonic behavior and its second harmonic generation efficiency (SHG) is four times that of KDP..

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4. Conclusions:

Good quality single crystals of L-Leucine hydrobromide were successfully grown by slow evaporation solution technique at room temperature by using doubly distilled water as a solvent for growth process. The growth process is observed very minutely and found that due to presence of inorganic hydrobromide, there is no growth of bio fungus. The powder XRD confirms the single phase and lattice dimensions were calculated. Its functional groups were identified by FTIR analysis. The grown crystal is a hard material and mechanical stable that enhanced its application in device fabrication. Large SHG efficiency equal to 4 times than of KDP indicate that the grown crystal have better non-linear behavior.

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