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# **Investigation on new NLO material L-histidine potassium pentaborate (LHKB5)**

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*Abstract***-** A new nonlinear optical crystal L-histidine potassium pentaborate single crystal was grown from aqueous solution by the slow evaporation solution growth technique to analyse its suitability for NLO device applications. It belongs to the orthorhombic crystal system which is confirmed by single crystal X-ray diffraction analysis. The UV-visible analysis was carried out on LHKB<sub>5</sub> crystal to find out cut-off wavelength and the energy gap of the material was found to be 245 nm and 5.05 eV. The presence of L-histidine potassium pentaborate on grown material was confirmed by FTIR analysis. TG-DSC analysis determines that the material has two stages of decomposition and it has three stages of weight loss. From DTA curve a sharp endothermic peak at 192.45°C shows good crystallinity and corresponds to the melting of the grown crystal. The mechanical strength of the material is calculated using Vicker's microhardness. Grown crystal shows that the material is very much suitable for a second harmonic generation and frequency conversion applications.

*Keywords***:** X-ray diffraction, FT-IR, Optical, TG-DSC and microhardness.

# **I. INTRODUCTION**

In recent trends, the research on new NLO materials has aroused much interest in modern material science for the growth of novel single crystal which finds a variety of applications to perform functions like optical switching, electro optic shutters and optical memory storage devices, photonics, frequency conversion and optoelectronic technology [1-4]. The semi organic crystals are superior in high optical nonlinearity, resistance to laser induced damage, a high degree of design chemical flexibility and with good mechanical strength and have high thermal stability [6, 7].Amino acids are potential substance for NLO applications and were subjected to extensive analysis by several researchers for their nonlinear properties. Since they contain proton donor carboxylic group, proton acceptor  $(-NH<sub>2</sub>)$ group in them and Zwitter ionic nature favors crystal hardness and crystallizes with a non-centrosymmetric space group [8-10]. Among the amino acids family, L-histidine molecule consists of the planar imidazole ring in the histidine structure which helps in possessing high nonlinear optical efficiency [11]. A number of L-histidine complexes were reported earlier which shows good NLO property, namely L-histidine maleates  $[12]$ , L-histidinehydrochloride<sup>13</sup> and  $Tb^{3+}$  doped L-histidine hydrochloride monohydrate crystal [14]. The electronegative value between boron and oxygen are higher, they can exhibit different structure. A boron atom may coordinate to triangular or tetrahedral shape in complex formation. The borates crystals possess high damage threshold and high optical quality. Some of the borate crystals which have been reported earlier are  $(KB_5)$ ,  $(APB)$ ,  $(K_3CdB_5O_{10})$  and  $(LAKB_5)$  [15-19]. In the present investigation the new semi organic crystal of L-histidine potassium pentaborate crystals have been grown by slow evaporation solution growth technique from aqueous solution. The grown  $LHKB<sub>5</sub>$  crystals were characterized by single crystal XRD, FTIR, UV-visible, TG-DSC and microhardness properties were reported for the first time.

# **II. EXPERIMENTAL DETAILS**

The analytical reagent (AR) grade materials of potassium carbonate (Merck) and boric acid (Merck) were taken in the ratio 1:10 for the synthesis of potassium pentaborate using deionized water as solvent. The resultant product of potassium pentaborate was found as homogeneous mixture which is then mixed with 1 mole of L-histidine (LOBA). The solution was continuously stirred for 5hours to obtain homogeneous mixture of L-histidine pentaborate (LHKB $_5$ ). After that, colorless solution was obtained which reaches saturation point. The saturated solution was passed through Wattmann filter paper twice to remove impurities and allowed for crystallization at room temperature. After one week a good quality seed crystals were obtained. The seed crystals were immersed in a mother solution and allowed to crystallize at room temperature. The  $LHKB<sub>5</sub>single$  crystals were harvested from the mother solution over a period of 28 days. Fig. 1 shows the reaction of synthesized LHKB<sub>5</sub> crystal. The grown LHKB<sub>5</sub> crystal is depicted in fig.2.



Fig. 1. Reaction scheme of  $LHKB<sub>5</sub>$ 



Fig. 2. Grown crystal of  $LHKB<sub>5</sub>$ 

# **III. CHARACTERIZATION METHOD**

L-histidine pentaborate crystals were characterized using varioustechniques like single crystal X-ray diffraction, Fourier Transform Infrared (FTIR), UV-VIS spectral, TG-DSC, microhardness and nonlinear optical (NLO) studies. Single crystal X-ray diffraction is carried using BRUKER APEX 2 with MoKα  $(λ=0.71073Å)$  radiation The Perkin Elmer Spectrum1 FTIR spectrum by KBr pellet technique between the range 4000-400cm<sup>-1</sup>. The UV-VIS absorption was analyzed using Perkin Elmer LAMDA 950 between the ranges 200-800. TG-DSC was carried using NETZSCH STA 449 F3 Jupiter thermal analysis is used to measure the thermal stability of the crystals. The microhardness analysis was carried using a Vicker's microhardness analyzer. The second harmonic generation was detected using Kurtz Perry powder technique.

#### **IV. RESULTS AND DISCUSSION**

#### **A. Single crystal X-ray diffraction studies**

The grown  $LHKB<sub>5</sub>$  crystals were subjected to single crystal X-ray diffraction analysis affirms that the title material belongs to orthorhombic crystal system. The unit cell parameters of title compound are a=9.13Å, b=11.22Å, c=11.12Å,  $\alpha = \beta = \gamma = 90^{\circ}$  and volume (V) = 1138Å [3]. The cell parameters of some of earlier reported  $KB<sub>5</sub>$  crystals are compared with  $LHKB<sub>5</sub>$  and it is depicted in table.1. The





#### **B. Vibrational analysis of Potassium pentaborate**

The  $LHKB<sub>5</sub>$  crystal were subjected to FTIR spectrum to confirm the presence of various functional groups. Fig.3 shows the FTIR spectrum of  $LHKB<sub>5</sub>$ . The peaks observed at  $3445 \text{ cm}^{-1}$  is correspond to O-H stretching vibration  $\lceil 17, 22 \rceil$ . The peak at 3061 cm<sup>-1</sup> is mainly due to OH stretch of  $(B_5O_6(OH)_4)$  and confirms that all the OH groups are present in hydrogen bonding [18,22]. The B-O asymmetric stretching vibrations are observed at 1350 cm<sup>-1</sup> and 1249 cm<sup>-1 22</sup>. The band assignments at 1101, 1025 and 782 cm-1 are due to tetrahedral boron [23]. The symmetric stretching of B-O in  $BO<sub>3</sub>$  is assigned due to sharp peak at 925 cm<sup>-1</sup>[24]. The O-B-O ring bending vibration occurring at 455 and 593  $\text{cm}^{-1}[22, 25]$ .

#### **Vibrational analysis of L-histidine**

The presence of L-histidine in the LHKB $<sub>5</sub>$  crystals</sub> was analyzed using Vibrational analysis data, the peak observed at 3377 cm<sup>-1</sup> is due to  $NH^{3+}$  asymmetric stretching vibration. The band occurring at  $2663 \text{ cm}^{-1}$  assigned due to  $CH<sub>2</sub> stretching vibration [10]$ . The band observed at 2176 cm<sup>-</sup> <sup>1</sup> is due to asymmetrical bending vibration of  $NH_3^+$ . The peak observed at  $1654 \text{ cm}^{-1}$  was attributed due to C=N stretching vibration of the imidazole ring [1]. The presence of the carboxylic acid group in L-histidine was confirmed by band observed at  $1434 \text{ cm}^{-1}$  is due to weak COO stretching. The weak C-H bending vibration was observed by a sharp peak at 694 cm<sup>-1</sup>. Torsional oscillation of  $NH_3^+$  was observed at 508 cm<sup>-1</sup>. The obtained vibrations were compared with some standard FTIR spectra and are in good agreement with  $[26-28]$ . FTIR spectrum of LHKB<sub>5</sub> was depicted in Fig.3.



Fig. 3 FTIR spectrum of  $LHKB<sub>5</sub>$ Table.2 Tentative assignment of various functional groups

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#### **C. Optical analysis**

The optical absorption of a single crystal is essential for device fabrications. The title material was subjected to UV-VIS absorption spectrum analysis between the range 200-800 nm. The plot of absorption spectra of  $LHKB<sub>5</sub>$  crystal is depicted in fig.4. The lower cut-off wavelength of  $LHKB<sub>5</sub>$  crystal was found to be 245 nm from the absorption spectrum. The low absorption of title material shows suitability for optoelectronic device fabrications. The  $LHKB<sub>5</sub>$  has low absorption between 245 to 800 nm shows its suitability for second harmonic generation. The measured absorption (A) was used to calculate the absorption coefficient ( $\alpha$ ) utilizing relation [20].

$$
\alpha = \frac{2.3042A}{t}
$$
---(1)

Where t is a thickness of the crystal and A is the absorption. The optical energy gap can be measured using the expression [21].

$$
(\alpha h v)^2 = A (E_g - h v)
$$
  
........(2)

Where A is a constant,  $v$  is the frequency of incident radiation, h Planck's constant and Eg energy gap. A plot between the energy gap (hν) and with the product of the absorption coefficient and the energy gap is depicted in fig.5. The measured energy gap of title material was 5.05  $eV$ . The LHKB<sub>5</sub> has low absorption in the entire visible region and has a high value of energy gap shows that  $LHKB<sub>5</sub>$  crystal is suitable for device fabrications.





Fig. 4 The absorption spectra of  $LHKB<sub>5</sub>$ .



Fig. 5 Tauc's plot of  $LHKB<sub>5</sub>$  crystals.

#### **D. Thermal analysis**

In the field of optical switching and optoelectronic device fabrications there is a huge need for thermally stable materials, which can with stand at high temperature when they are exposed to laser continuously [29].Thermogravemetric(TG) and Differential scanning (DSC) studies were carried concomitantly on  $LHKB<sub>5</sub>$ powder sample 4.739 mg. Fig.6.shows the thermograms of LHKB<sub>5</sub>.From TG curve it shows three stage of decomposition first stage weight loss of 16.31% between the temperatures ranges  $169.44^{\circ}$ C to  $220.20^{\circ}$ C due to vaporization of water molecules in the lattice points of the crystals. From DSC curve a sharp exothermic peaks observed at 198.35°C due to decomposition of L-histidine and anhydrous potassium pentaborate. Second stage weight loss of about 7.01% between temperatures 220.20°C to 416.71<sup> $\degree$ </sup>C due to liberation of volatile substance such as CO<sub>2</sub>,  $H<sub>2</sub>O$  and NH<sub>3</sub> in L-histidine and potassium pentaborate. The decomposition of borate in potassium pentaborate was observed from DSC curve at 787.29°C due to sharp exothermic peak. Third stage of weight loss of 30.17% was obtained at the range of  $1084.44^{\circ}$ C to  $1400^{\circ}$ C due to removal of borate from potassium pentaborate. The total mass change of 53.40% is occurring between  $169.44^{\circ}$ C to  $1400^{\circ}$ C. The final residue of 47.52% was left about 1400°C. From TG-DSC curve confirms that material can tolerate heat of 198.35°C which is very much essential for optoelectronic device fabrication. DTA curve of  $LHKB<sub>5</sub>$  was depicted at

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fig.7. The endothermic peak of the DTA analysis was observed at 192.45°C which corresponds to melting point of the material. The good degree of crystalline nature and purity of  $LHKB<sub>5</sub>$  crystals were confirmed by sharp exothermic peak [2].



Fig. 6 TG-DSC curve of  $LHKB<sub>5</sub>$  crystals.



Fig. 7 DTA curve of  $LHKB<sub>5</sub>$  crystals.

# **E. Microhardness Measurement**

The Vicker's diamond pyramidal indenter microhardness analyzer was used to contemplate the mechanical quality of L-histidine potassium pentaborate crystal. In the field of optoelectronic device fabrication, the mechanical quality of the material plays a fundamental role. Using different loads from 10g to 100g with a consistent space-time of 10 s for all loads the indentations were made utilizing a Vicker's pyramidal indenter [30]. The hardness of the L-histidine potassium pentaborate crystal can be determined to utilize the relation [19].

 ⁄ **-------**(3)

Where d is the diagonal length in mm and P is the applied load in kg. Fig. 8. demonstrates the variety of connected load with a Vicker's hardness number for title material. Reverse indentation size effect (RISE) is noticed on  $LHKB<sub>5</sub>$  crystal due to the increase of hardness  $(H_v)$  as the load  $(P)$  is increased [31]. To distinguish the material quality the estimation of the work hardening coefficient is fundamental. Meyer's index (n) value for the soft material is more noteworthy than 1.6 and for hard material under 1.6. Meyer's index (n) can be resolved to utilize the relation [32].

$$
P = K d^n \qquad \qquad \text{---} \tag{4}
$$

$$
LogP = LogK + nLogd
$$
 \n-----(5)

Where n is Meyer's index and K is a constant for a crystal. Meyer's index (n) is assessed from the fig. 9. Variation between log d and log P, by a linear fit. The slope value n=3.09 was distinguished from linear fit, in this manner affirming that the LHKB5 crystal has a place with the soft category. L-histidine potassium pentaborate crystal has a great mechanical strength which is essential for optoelectronic device fabrications.



Fig. 8 Variation of Vicker's hardness number and applied load for  $LHKB<sub>5</sub>$  crystal.



Fig. 9 Variation of log d and log p for  $LHKB<sub>5</sub>$  crystal.

# **F. Kurtz-Perry Powder Second Harmonic Generation Studies**

The grown crystals of L-histidine potassium pentaborate was subjected to Kurtz-Perry [33] powder second harmonic generation (SHG) test to confirm the nonlinear optical (NLO) property. The sample were taken in a powder form and tightly packed between glass slides Nd: YAG Qswitched laser beam of wavelength 1064 nm was made to fall on the powder sample at a repetition rate of 10 Hz with a pulse width of 6 ns. The input energy of 0.61 J was made to fall on the powder samples which exposed to laser radiation. The second harmonic generation in the crystal was confirmed by the emission of green light from the sample. The reference material of potassium dihydrogen phosphate crystals was taken in the powder form to compare the SHG efficiency with  $LHKB<sub>5</sub>$ . The output power of standard KDP

is 7.501mJ and title material emitted output power of 15.752mJ. The efficiency of  $LHKB<sub>5</sub>$  crystals was found to be 2.1 times greater than that of the KDP crystals.

# **V. CONCLUSION**

The single crystal of L-histidine potassium pentaborate was grown from aqueous solution by employing a solvent evaporation technique at room temperature. X-ray diffraction studies affirm that the crystal belongs to the orthorhombic crystal system and lattice parameter values are reported. Presence of L-histidine potassium pentaborate functional groups vibrations were confirmed by the FTIR spectrum. From the UV absorption spectrum, energy gap and cut-off wavelength were found to be 5.05 eV and 245 nm. TG-DSC curve shows two stages of decomposition at 198.35°C and 782.29°C of the material and the endothermic peak of the DTA analysis are observed at 192.45°C which corresponds to the melting point of the material. The good degree at crystalline nature and purity of  $LHKB<sub>5</sub>$  crystals were confirmed by a sharp endothermic peak at 192.45°C from DTA curve. From the hardness study, material has Meyer's index n=3.04 which shows that title material belongs to soft crystal category.SHG efficiency confirms that the crystal has 2.1 times greater efficiency than reference material standard KDP. The studies carried out on title material conclude that the material has high thermal stability and SHG efficiency which is essential for device fabrication. Thus, various studies carried on L-histidine potassium pentaborate crystal suggest that the material has great potential for nonlinear optoelectronics device fabrication.

#### **Compliance with Ethical Standards:**

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