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# Investigation on new NLO material L-histidine potassium pentaborate (LHKB<sub>5</sub>)

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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>1</sup> and Tb<sup>3+</sup> 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<sub>5</sub>), (APB), (K<sub>3</sub>CdB<sub>5</sub>O<sub>10</sub>) and (LAKB<sub>5</sub>) [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<sub>5</sub>). 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 MoK $\alpha$  ( $\lambda$ =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



inclusior	ı of	L-histidine	in	$KB_5$	shows	small	variation	in
lattice pa	iram	eters value.						

Table .1 Cell parameters values of KB <sub>5</sub> and LHKB <sub>5</sub>							
Cell parameters	$\mathrm{KB_{5}}^{25}$	LHKB5 <sup>Present work</sup>					
a (Å)	11.13	9.13					
b (Å)	11.23	11.22					
c (Å)	9.10	11.12					
Volume $(\text{Å})^3$	1137	1138					

#### **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 cm<sup>-1</sup> is correspond to O-H stretching vibration [17, 22]. 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<sup>-1</sup> 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 cm<sup>-1</sup>[22, 25].

#### Vibrational analysis of L-histidine

The presence of L-histidine in the LHKB<sub>5</sub> crystals was analyzed using Vibrational analysis data, the peak observed at 3377 cm<sup>-1</sup> is due to NH<sup>3+</sup> asymmetric stretching vibration. The band occurring at 2663 cm<sup>-1</sup> 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 cm<sup>-1</sup> 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 cm<sup>-1</sup> is due to weak COO<sup>-</sup> stretching. The weak C-H bending vibration was observed by a sharp peak at 694 cm<sup>-1</sup>. Torsional oscillation of NH<sub>3</sub><sup>+</sup> 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 LHKB5 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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Assignments	Wavenumber cm <sup>-1</sup>
O-H stretching	3445, 3377
OH stretch of $(B_5O_6(OH)_4)^-$	3061
B-O asymmetric stretching	1350, 1249
tetrahedral boron	1101, 1025, 782
B-O in BO <sub>3</sub> symmetric stretching	925
O-B-O ring bending	455,593
NH stretching	2663
NH <sub>3</sub> <sup>+</sup> asymmetrical bending	2176
COO <sup>-</sup> strong band	1654
COO <sup>-</sup> weak band	1434
C-H bending	694
$\mathrm{NH_3}^+$ torsional oscillation	508

#### 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].

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



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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 LHKB5 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°C to 220.20°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 C due to liberation of volatile substance such as  $CO_2$ , 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°C to 1400°C due to removal of borate from potassium pentaborate. The total mass change of 53.40% is occurring between 169.44°C to 1400°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 LHKB5 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].

$$Hv = \frac{1.854p}{d^2} (kg/mm^2)$$
 -----(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].

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$$LogP = LogK + nLogd$$
 -----(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 Q-switched 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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