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# **Structural, Optical and Thermal Analysis of Valine Doped Potassium Pentaborate (LVPPB) Crystals**

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Abstract: In this present communication, I have grown Valine potassium pentaborate (LVPPB) crystals by slow evaporation solution growth technique. To know its suitability for device fabrication, different characterization analyses have been performed. by powder X-ray diffraction method the lattice constants have been calculated. It is found that it has orthorhombic structure by using powder-x computer software. FT-IR measurements calculate the functional groups present in the compound. The optical absorption property of the crystal was inspected by UV-Vis spectral analysis. The thermal stability of the crystal was examined by TGA analysis.

Keywords: Crystal growth, powder XRD, FT-IR, UV-Vis, TGA.

## I. INTRODUCTION

Borate crystals are superior in UV applications to other commonly used NLO materials such as potassium dihydrogen phosphate (KDP) or lithium niobate (LN) because of their high transmittance at wavelengths down to 155 nm combined with higher damage threshold. [1, 2]. Potassium pentaborate (KBO<sub>5</sub>) is a desirable inorganic nonlinear optical material which exhibits a low angular sensitivity and hence, proved useful for second harmonic generation. [3]. High damage threshold and wide transparency make it a better alternative for KDP crystals in frequency doubling and laser fusion experiments [4]. Kato attributed the primary advantage of KBO<sub>5</sub> over KDP to its slightly large birefringence, which permitted generation of shorter wavelengths. Further his experiments confirmed the fifth harmonic generation for KBO<sub>5</sub> crystals at room temperature, whereas the other NLO crystals like ADP and KDP can achieve the same only at high temperature [5, 6]. The growth of single and twinned crystals of KBO<sub>5</sub> by low temperature solution growth was reported by several workers [7, 8, 9]. The SHG efficiency of the pure and doped KBO<sub>5</sub> crystals were also studied using Nd:YAG Q-switched laser [10]. Nonlinear optical (NLO) crystals are widely used in harmonic generation, switching and other optical signal processing devices. So, the search for new materials possessing high optical nonlinearity is an important task. Some complexes of amino acids with inorganic salts are reported to be promising materials for optical second harmonic generation [11–13]. Amino acids with inorganic compounds are promising materials for nonlinear optical applications, as the high optical nonlinearity of the purely organic amino acid tends to combine with the favourable mechanical and thermal properties of the inorganic salt [14]. In this series, I have successfully grown Valine potassium pentaborate crystals and the results of characterization of these crystals have been reported in this communication.

#### II. EXPERIMENTAL

#### A. Crystal Growth

Grown pure potassium pentaborate and L-Valine (AR grade) were dissolved in the ratio 1:3 in double distil water until a saturated solution was obtained. The solution was stirred well thoroughly for three hours using a magnetic stirrer. The impurities were by successive recrystallization. The supersaturated solutions were filtered using Whatmann filter paper no. 1 and were kept in dust free atmosphere. In order to obtain single crystals of high quality, recrystallization was carried out for more than three times. Then the solution was filtered and crystallization was allowed to take place by slow evaporation under room temperature. Good quality optically transparent single crystal of KBO<sub>5</sub> and doped 3M-LVPPB with average size  $15 \times 9 \times 8 \text{ mm}^3$  and  $11 \times 6 \times 7 \text{ mm}^3$  were obtained in a period of 70 to 90 days and the grown crystals are shown in Figure 2.1 and 2.2.



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### B. Characterization

Powder X-ray diffraction pattern was recorded for pure KBO<sub>5</sub> and KBO<sub>5</sub> crystal doped with 3 mol% L-Valine (3M-LVPPB) using a Philips X-ray diffractometer with CuK $\alpha$  ( $\lambda = 1.5418$ Å) radiation. FT-IR spectrum of the grown crystals was recorded on SHIMADZU FT-IR 8400 spectrophotometer in the regions 4000–400 cm<sup>-1</sup> by KBr pellet method to assign functional groups present in pure KBO<sub>5</sub> and 3M-LVPPB. UV-Vis of pure KBO<sub>5</sub> and 3M-LVPPB are used to study the linear optical properties. The optical absorption spectra was measured in the range 210 to 800 nm using the SHIMADZU UV 1700 UV-Spectroscopy. Measurement of the thermo gravimetric analysis (TGA) were carried out on the pure KBO<sub>5</sub> and 3M-LVPPB crystals between 30 °C (room temperature) and 900 °C in the nitrogen atmosphere and alumina reference which provides an inert environment with the heating rate of 20 °C/min.

#### III. RESULT AND DISCUSSION

#### A. Single crystal X-ray diffraction analysis

The unit cell parameters of pure KBO<sub>5</sub> and 3M-LVPPB crystals were obtained from the powder X-ray diffraction analysis by using the powder–x computer software. *h*, *k* and *l* parameters as well as *d* and 20 values are generated in such a way that these values match with the X – ray powder diffraction values. From the powder X-ray diffraction studies it was found that the pure KBO<sub>5</sub> and 3M-LVPPB crystals have orthorhombic crystal structure with unit cell parameters a = 11.065, b = 11.171, c = 9.054 and a = 11.070, b = 11.169, c = 9.037 respectively and lattice parameter is  $\alpha = \beta = \gamma = 90^{\circ}$  which is matched with reported value [18,19]. The powder X-ray diffraction pattern of the pure KBO<sub>5</sub> and 3M-LVPPB crystals are given in figure 3.1 and 3.2. Experimental and calculated values of d (A°) for pure KBO5 and 3M-LVPPB are matched.



Figure 3.1 Powder X-ray diffraction pattern of Pure KBO<sub>5</sub>





Figure 3.2 XRD of 3M-LVPPB

# B. FT-IR spectral analysis

The FT-IR spectrum was recorded on SHIMADZU spectrophotometer in the regions 4000–400 cm<sup>-1</sup>, by KBr pellet method. Due to molecular vibrational modes, the molecules undergo a net change in their dipole moment and absorb the IR radiation. The molecules are related with each other due to the intermolecular forces connecting the hydrogen bonding. This hydrogen bonding which the molecules to be held together due to the hydroxyl groups of pentaborate anion and water. For pure KBO<sub>5</sub> the OH stretch of water is a doublet due to unequal H bonds for 2H of H<sub>2</sub>O and ring O<sub>2</sub> of pentaborate which occurs at 3368.49 cm<sup>-1</sup>. The OH stretch of B<sub>5</sub>O<sub>6</sub>(OH)<sub>4</sub> due to strong H bonding interaction with ring O<sub>2</sub> occurs at 3052 cm<sup>-1</sup>. The broad envelope between 1246.66 cm<sup>-1</sup> and 1330.35 cm<sup>-1</sup> is assigned to B-O stretching vibrations and the corresponding bending modes are assigned at 1022.45 cm<sup>-1</sup>. The O-B-O ring vibrations occur between 914.22 cm<sup>-1</sup> to 453.89 cm<sup>-1</sup>. For 3M-LVPPB the OH stretch of B<sub>5</sub>O<sub>6</sub>(OH)<sub>4</sub> due to strong H bonding interaction which occurs at 2924.09 cm<sup>-1</sup>. The OH stretch of B<sub>5</sub>O<sub>6</sub>(OH)<sub>4</sub> due to strong H bonding of pentaborate which occurs at 2924.09 cm<sup>-1</sup>. The OH stretch of B<sub>5</sub>O<sub>6</sub>(OH)<sub>4</sub> due to strong H bonding interaction when cocurs at 2924.09 cm<sup>-1</sup>. The OH stretch of B<sub>5</sub>O<sub>6</sub>(OH)<sub>4</sub> due to strong H bonding interaction when ring O<sub>2</sub> occurs at 2924.09 cm<sup>-1</sup>. The OH stretch of B<sub>5</sub>O<sub>6</sub>(OH)<sub>4</sub> due to strong H bonding interaction when ring O<sub>2</sub> occurs at 2856.68 cm<sup>-1</sup>. The broad envelope between 1246.02 cm<sup>-1</sup> and 1330.88 cm<sup>-1</sup> is assigned to B-O stretching vibrations and the corresponding between 1246.02 cm<sup>-1</sup> and 1330.88 cm<sup>-1</sup> is assigned to B-O stretching vibrations and the corresponding bending modes are assigned at 1028.06 cm<sup>-1</sup> in both. The O-B-O ring vibrations occur between 918.12 cm<sup>-1</sup> to 561.29 cm<sup>-1</sup>. The spectra of the pure KBO<sub>5</sub> and 3M-LVPPB crystals are shown in Figure 3.3 and 3.4.



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## C. Optical transmission studies

Amino acid doped KBO<sub>5</sub> UV–Vis characterization is an important study for NLO crystals to know the optical transmission range of the crystals. The grown crystals of KBO<sub>5</sub> and amino acid doped KBO<sub>5</sub> were subjected to absorption measurements in the spectral region of KBO<sub>5</sub> UV–Vis characterization is an important study for NLO crystals to know the optical transmission range of the crystals. The UV–Vis optical absorption spectra of pure KBO<sub>5</sub> and amino acid doped KBO<sub>5</sub> (3M-LVPPB) crystals are recorded in the range 200–900 nm. There is no absorption between 286 nm to 800 nm in pure KBO<sub>5</sub> and 286.5 nm to 800 nm in 3M-LMPPB respectively. The good transmission property of the crystal in the entire visible region suggests its suitability for NLO applications. In order to estimate the non linear optical property of the crystal, the grown crystal was subjected to second harmonic generation (SHG) efficiency test. An Nd: YAG laser beam was sent through the crystal and emission of green light was observed. This confirms the SHG efficiency of the crystal. Both of UV-Vis is given in figure 3.5 and 3.6.





Figure 3.5 UV-Vis. spectrum of KBO<sub>5</sub> crystal



Figure 3.6 UV-Vis. spectrum of 3M-LVPPB

# D. Thermal Analysis

For device fabrication, the information about the thermal stability is needed. The thermo gravimetric analysis of KBO<sub>5</sub> was carried out between 30°C (RT) and 900°C in nitrogen atmosphere and alumina reference at a heating rate of  $20^{\circ}$ C/min. It is observed that the dopant slightly increases the decomposition temperature and change the weight loss of KBO<sub>5</sub> crystals. The weight loss of pure KBO<sub>5</sub> is 25.45%. No further decomposition was observed in the temperature range 452- 900°C and the weight loss of 3M-LVPPB is 27.28%. No further decomposition was observed in the temperature range 452- 900°C The difference in decomposition temperature of pure KBO<sub>5</sub> and 3M-LVPPB crystals confirms the incorporation of dopant in the crystal lattice. From the TGA curve it is observed



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that there is no weight loss up to  $162^{\circ}$ C in pure KBO<sub>5</sub> and  $150^{\circ}$ C in 3M-LVPPB which indicates the good thermal stability of the crystal. Above  $210^{\circ}$ C, there is no weight loss in both pure KBO<sub>5</sub> and 3M-LVPPB. The TGA are carried out by using Model: TG/DTA7300, make: EXSTAR. The TGA traces of pure KBO<sub>5</sub> and 3M-LVPPB are shown in figure 3.7 and 3.8.



Figure 3.7 TGA spectrum of KBO<sub>5</sub> crystal



Figure 3.8 TGA spectrum of 3M-LVPPB

# IV. CONCLUSION

By slow evaporation method, Valine mixed potassium pentaborate crystals were grown. The functional groups present in the material were identified using FT-IR spectral analysis. The optical transparency of grown crystals was analyzed with the help of UV-Vis spectrum. The emission of green light from the crystal for the input of Nd: YAG laser confirms the SHG efficiency of the crystal. From the TGA-DTA analysis carried out for the crystal, the thermal stability of the crystal is found to be 162°C.

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