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Growth and Characterization of Pure and Methionine Doped Potassium Pentaborate (KBO_5) Single Crystals

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Abstract: ***Potassium pentaborate (KB_5O_8 . 4 H_2O or $K(H_4B_5O_{10}).2H_2O$ or KBO_5) single crystals were grown by slow evaporation solution growth method at room temperature and the properties of the grown crystals were investigated. L-Methionine doped Potassium pentaborate (LMPPB) single crystals were grown by slow evaporation solution growth method and the characterization for the obtained materials were done using powder x-ray diffraction, FT-IR, UV-Vis and thermal analysis methods. Powder X-ray diffraction studies were carried out to determine the crystal structure and lattice parameters of the grown crystals by using powder-x software.. The optical transmittance of the crystal was determined by recording UV-Vis spectrum. pure KBO_5 and L-Methionine potassium pentaborate (LMPPB) single crystals were synthesized from its aqueous solution at ambient temperature. XRD confirmed the orthorhombic nature of the crystals. The UV-Vis spectroscopy highlighted the transparent nature of the LMPPB crystal. The various functionalities slot in the synthesized compound were analysed by FT-IR technique. The TGA analyses showed the thermal stability and the melting point of LMPPB.***
Keywords: Single crystal, UV-Vis study, FT-IR-study, Thermal stability, Thermo Gravimetric Analysis (TGA)

I. INTRODUCTION

Borate crystals are often employed for high- power UV generation because of their relatively high tolerance to laser-induced damage, large optical nonlinear coefficients, and greater transparency in the UV region [1]. The first borate crystal described for UV generation was $KB_5O_8.4H_2O$ [2]. Potassium pentaborate (KBO_5) is an important inorganic NLO crystal and it is successfully used for UV conversion of laser radiation to the UV and vacuum UV wavelength region [3, 4].The lattice parameters are a = 11.065 A^0, b = 11.171 A^0 and c = 9.054 A^0. A number of research articles have been published in the literature discussing the growth and NLO properties of KBO_5 crystals [5-8]. Our research group had already reported the growth and characterization of KBO_5[9, 10]. In present work, single crystals of pure potassium pentaborate and amino acid doped potassium pentaborate (1M-LMPPB) are synthesized successfully. The x-ray diffraction study confirms the orthorhombic nature of the crystal. The transparent nature in the UV-visible and Infrared region confirms the non linear property of the crystal and the optoelectronic device fabrication. The FTIR study proves the amino group and the borate groups. The TGA analyses explain the thermal stability of the grown crystal. Synthesis and characterization of grown pure Potassium pentaborate and grown L-Methionine doped Potassium pentaborate (1M-LMPPB) single crystals were studies.

II. EXPERIMENTAL

A. Crystal Growth

Potassium carbonate (K_2CO_3) and boric acid (H_3BO_3) were dissolved in double distilled water at room temperature. Then the resultant solution was stirred well to obtain clear solution. The solution was filtered using Whatmann filter paper no. 1 and taken in a beaker. Beaker containing the solution was closed with polythene sheet containing small pinholes and it was kept in a dust free atmosphere. Solvent evaporation at room temperature yielded transparent single crystals of pure KBO_5 of $15 \times 9 \times 8$ mm^3 size in a growth period of 70 to 90 days (Fig.2.1). The same way L-Methionine (AR grade) amino acid is doped into grown pure KBO_5 in 1:1 ratio. The amino acid doped KBO_5 single crystals were prepared by doping 1 mol% amino acid (AR grade: L-Methionine) into pure KBO_5 in double distilled water at room temperature and stirring thoroughly for three hours. The impurities were by successive recrystallization. The supersaturated solutions were filtered using Whatmann filter paper no. 1 and were kept in dust free atmosphere. To get single crystals of high quality, recrystallization was carried out for more than three times. The supersaturated solution was prepared from the recrystallized 1M-LMPPB. The saturated solution was allocated to dry at room temperature by the

slow evaporation technique. Solvent evaporation at room temperature produced transparent single crystals of 1M-LMPPB of 18 × 12 × 9 mm^3 size in a growth period of 70 to 90 days (Fig.2.2). The salt was synthesized according to the relation

$K_2CO_3 + 10H_3BO_3 \rightarrow 2[K\ (H_4B_5O_{10})]\ .\ 2H_2O + 7H_2O + CO_2\uparrow$ (2.1)

Growth and characterisation of potassium pentaborate single crystals have been reported elsewhere by the author et al [10, 11].

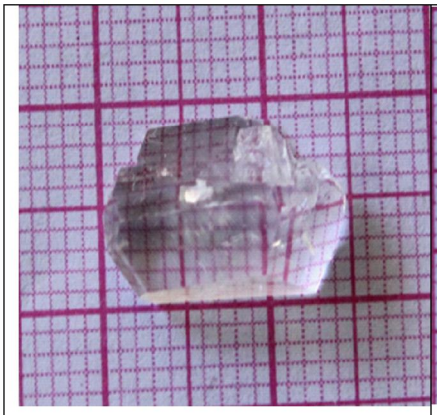

Figure 2.1: Photograph of grown pure KBO_5 crystal

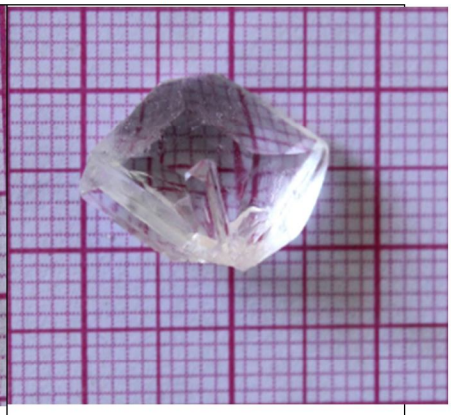

Figure 2.2: Photograph of KBO_5 crystal doped with 1 mol% L-Methionine (1M-LMPPB)

III. CHARACTERIZATION STUDIES

Using powder-x software, the lattice parameters were calculated. Powder X-ray diffraction pattern was recorded for pure KBO_5 and KBO_5 crystal doped with 1 mol% L-Methionine (1M-LMPPB) using a Philips X-ray diffractometer with CuKα (λ = 1.5418Å) radiation. The amino acid and borate groups in the 1M-LMPPB were confirmed by using the Shimadzu FT-IR 8400 spectrometer. The wavelength range used in the spectrometer was 400-4000 cm^{-1} method adopted was KBr pellet method. The UV-Vis of pure KBO_5 and 1M-LMPPB 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) was carried out on the pure KBO_5 and 1M-LMPPB crystals between 30 ^{0}C (RT) and 900 ^{0}C in the nitrogen atmosphere. The rate of heating used was 20 ^{0}C per minute.

IV. RESULTS AND DISCUSSION

A. X – ray Diffraction Analysis

The unit cell parameters of pure KBO_5 and 1M-LMPPB crystals were obtained from the powder X-ray diffraction analysis which suggest that crystals have orthorhombic crystal structure with unit cell parameters a = 11.065, b = 11.171, c = 9.054 and a = 11.072, b = 11.170, c = 9.045 lattice parameter is $\alpha = \beta = \gamma = 90^0$ which is matched with reported value.

[12, 13, 14]. Powder XRD plot of pure KBO5 and 1M-LMPPB are shown in figure 4.1 and 4.2 . Experimental and calculated values of d (A°) for pure KBO5 and 1M-LMPPB are matched.

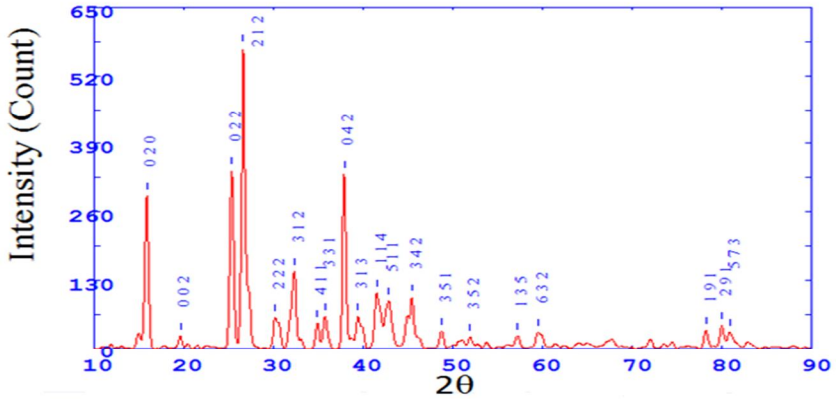

Figure 4.1 Powder X-ray diffraction pattern of Pure KBO_5

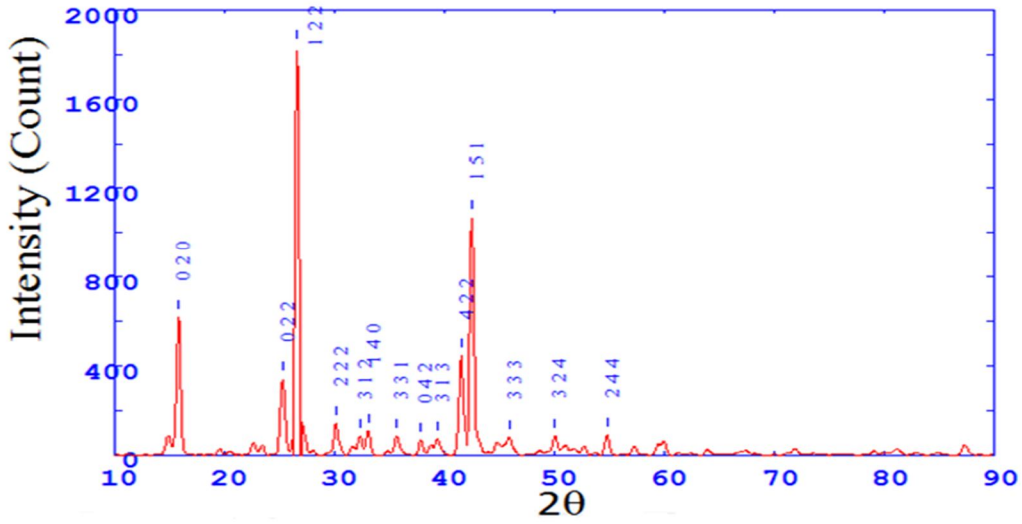

Figure 4.2 Powder X-ray diffraction pattern of 1M-LMPPB

B. FT-IR spectral Analysis

The FT-IR spectrum was recorded on SHIMADZU spectrophotometer in the regions 4000–400 cm^{-1} 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 associated with each other due to the intermolecular forces between the hydrogen bonding. This hydrogen bonding which the molecules to be held jointly due to the hydroxyl groups of pentaborate anion and water. For pure KBO_5 the OH stretch of water is a doublet due to unequal H bonds for 2H of H_2O and ring O_2 of pentaborate which occurs at 3368.49 cm^{-1}. The OH stretch of $B_5O_6(OH)_4$ due to strong H bonding interaction with ring O_2 occurs at 3052 cm^{-1}. The broad envelope between 1246.66 cm^{-1} and 1330.35 cm^{-1} is assigned to B-O stretching vibrations and the corresponding bending modes are assigned at 1022.45 cm^{-1}. The O-B-O ring vibrations occur between 914.22 cm^{-1} to 453.89 cm^{-1}. For 3M-LVPPB the OH stretch of water is a doublet due to unequal H bonds for 2H of H_2O and ring O_2 of pentaborate which occurs at 2924.09 cm^{-1}. The OH stretch of $B_5O_6(OH)_4$ due to strong H bonding interaction with ring O_2 occurs at 2856.68 cm^{-1}. The broad envelope between 1246.02 cm^{-1} & 1330.88 cm^{-1} is assigned to B-O stretching vibrations and the corresponding bending modes are assigned at 1028.06 cm^{-1} in both. The O-B-O ring vibrations occur between 918.12 cm^{-1} to 561.29 cm^{-1}. The spectra of the pure KBO_5 and 1M-LMPPB crystals are shown in figure 4.3 and 4.4.

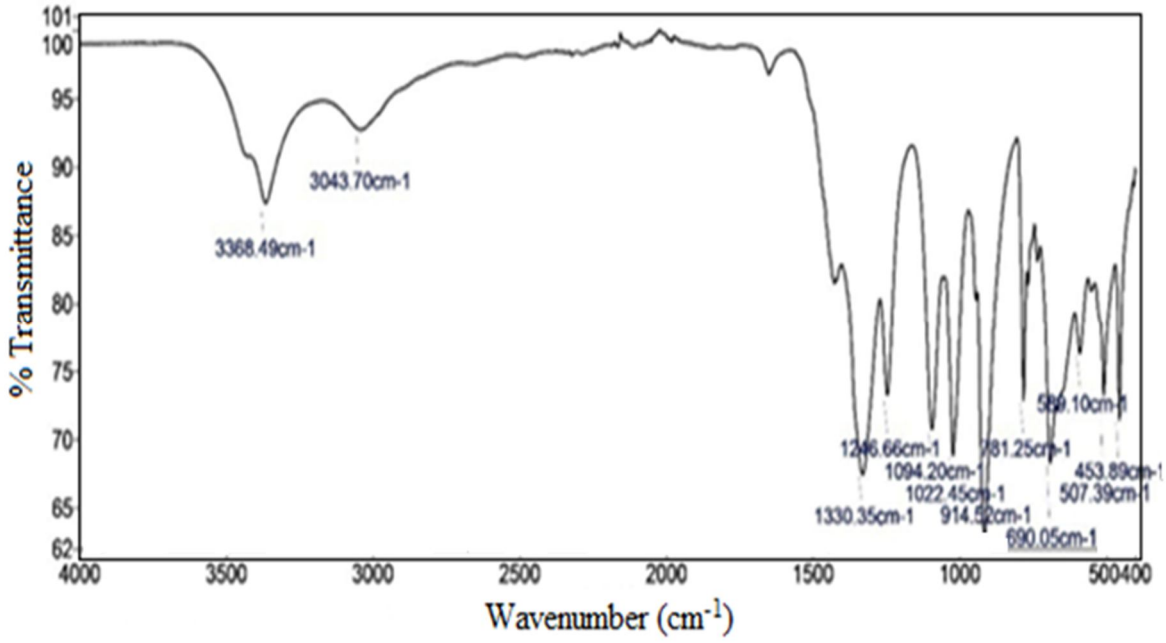

Figure 4.3 FT-IR of pure KBO_5

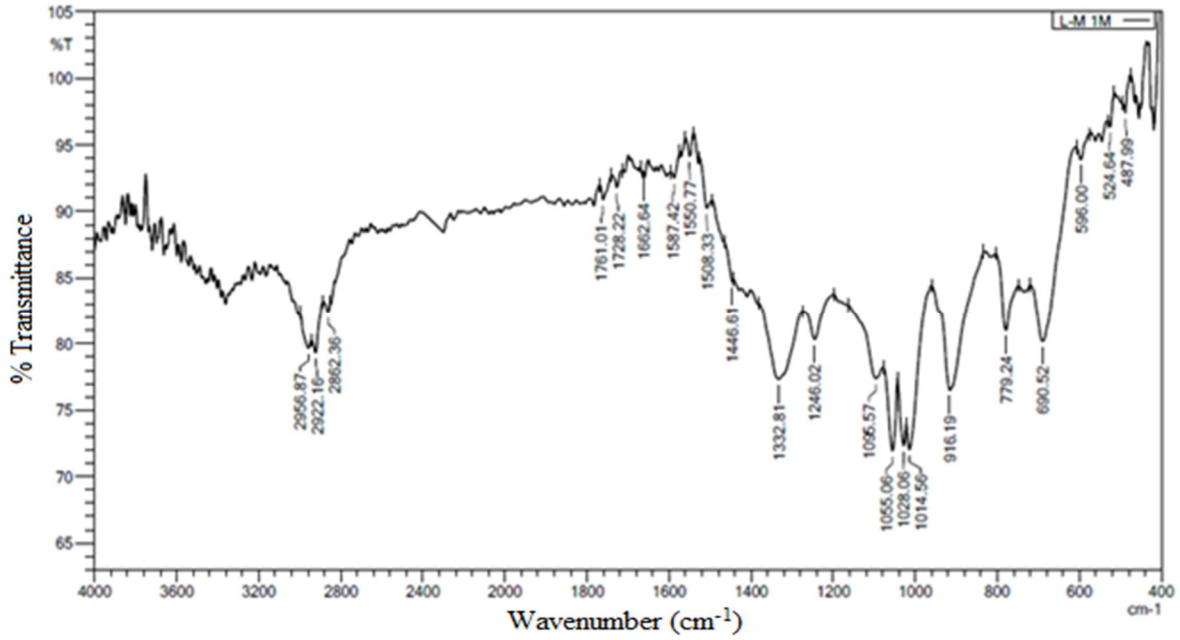

Figure 4.4 FT-IR of 1M-LMPPB

C. UV-Vis Spectral Studies

UV-Vis optical transmission spectrum was recorded in the range of 200 - 900 nm. There is no absorption between 286 nm to 800 nm in pure KBO_5 and 286.5 nm to 800 nm in 1M-LMPPB respectively. The good transmission property of the crystal in the entire visible region suggests its suitability for NLO applications. The transmittance spectrum of pure KBO_5 and 1M-LMPPB are presented in Fig. 4.5 and 4.6.

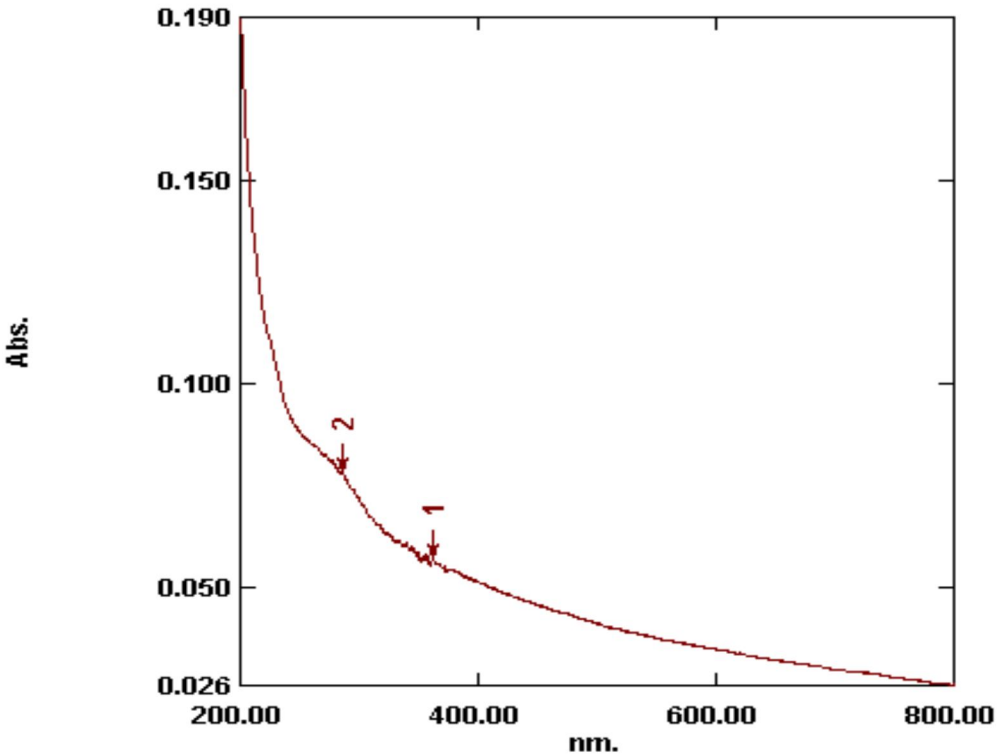

Figure 4.5 UV-Vis. spectrum of KBO_5 crystal

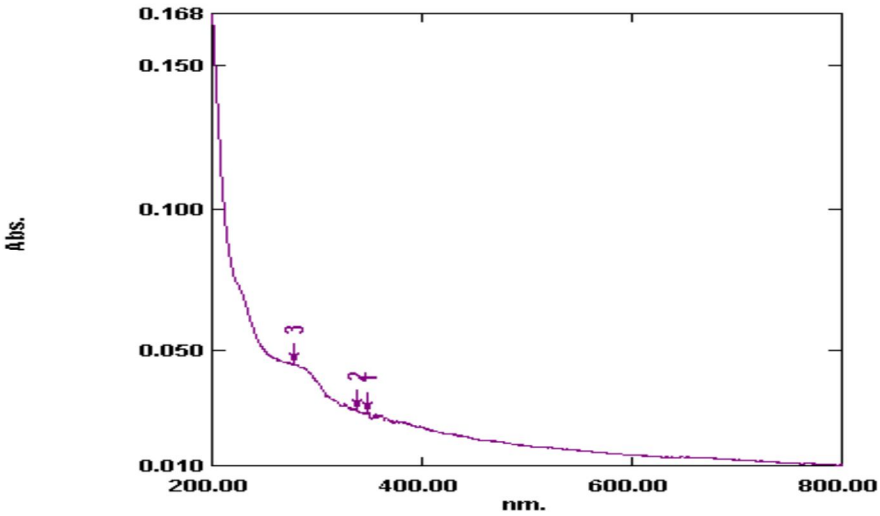

Figure 4.6 UV-Vis. spectrum of 1M-LMPPB

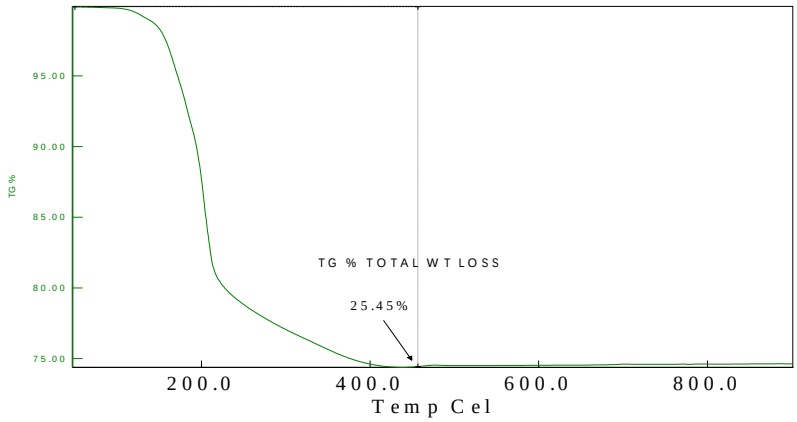

Figure 4.7 TGA spectrum of KBO_5 crystal

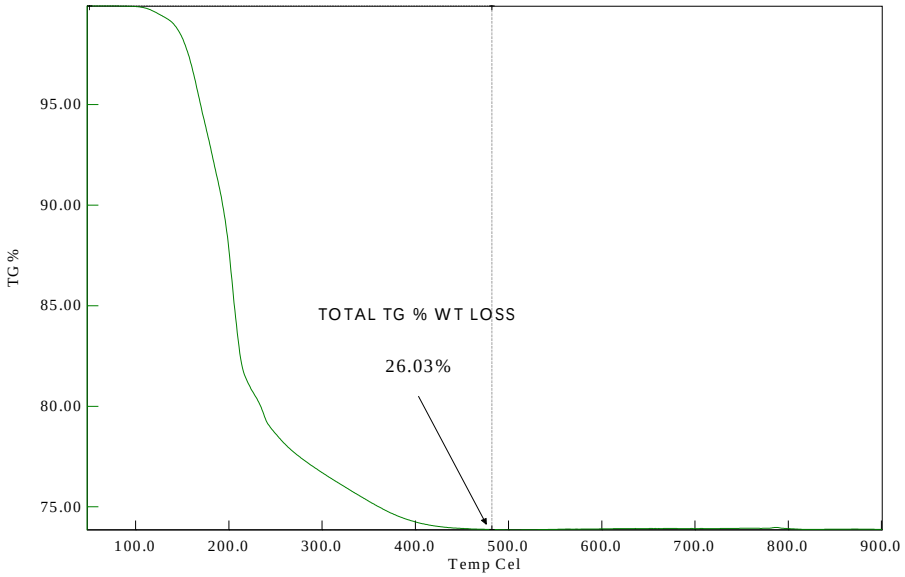

Figure 4.8 TGA spectrum of 1M-LMPPB

A. Thermo gravimetric Analysis

The thermo gravimetric analysis of KBO_5 was carried out between 30°C (Room Temperature) to 900°C in nitrogen atmosphere and alumina reference at a heating rate of 20 ^{0}C/min. It is observed that the dopant slightly increases the decomposition temperature and alter the weight loss of KBO_5 crystals. The weight loss of pure KBO_5 is 25.45% and 1M-LMPPB is 26.03%. No further decomposition was observed in the temperature range 442- 900 °C The difference in decomposition temperature of pure and 1M-LMPPB crystals confirms the incorporation of dopant in the crystal lattice. From the TGA curve it is observed that there is no weight loss up to 162 ^{0}C in pure KBO_5 and 160 ^{0}C in 1M-LMPPB which indicates the good thermal stability of the crystal. Above 210°C, there is no weight loss in both pure KBO_5 and 3M-LVPPB. The TGA are carried out by using Model: TG/DTA7300, make: EXSTAR. The TGA traces of doped KBO_5 and 1M-LMPPB are shown in figure 4.7 and 4.8.

V. CONCLUSION

Single crystals of pure potassium pentaborate and L-Methionine potassium pentaborate (1M-LMPPB) are synthesized successfully. The single crystal study confirms the orthorhombic nature of the crystal. The FT-IR study shows the amino group and the borate groups. The transparent nature in the UV-visible and Infrared region confirms the non linear property of the crystal and also the optoelectronic device fabrication. The TGA analyses explain the thermal stability and the melting point of 1M-LMPPB is equivalent to 253˚C.

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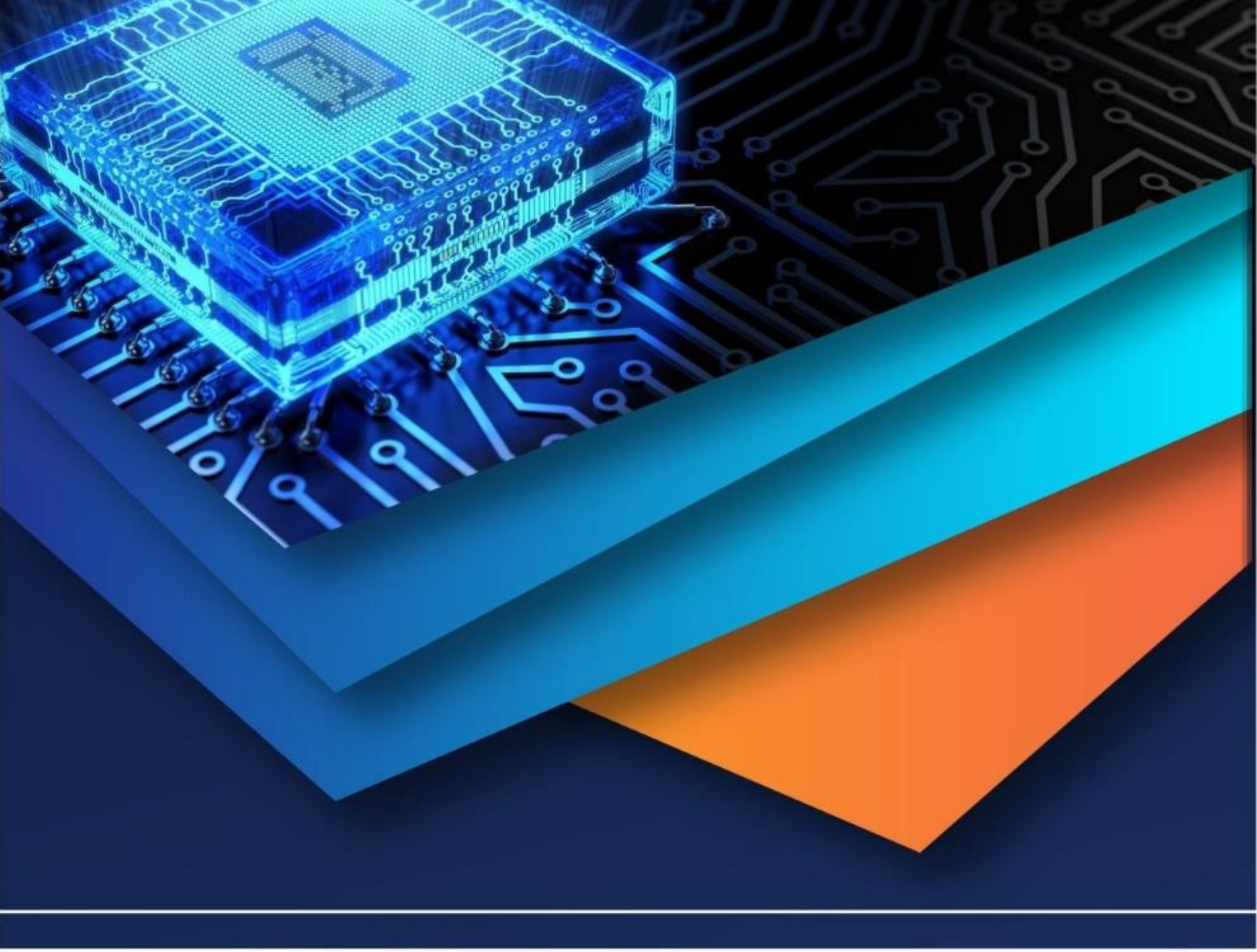

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