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Synthesis, Optical, Thermal and Microhardness Studies of L-Alanine Potassium Penta Borate Octa Hydrate (LAPPB)

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Abstract: The aim of the work is to synthesise a new semiorganic crystal of L-Alanine Potassium Penta Borate Octa Hydrate [LAPPB]. The LAPPB crystals were synthesised by adopting slow evaporation method. The Size of the materials obtained are $a = 9.04 \text{ \AA}$, $b = 11.5 \text{ \AA}$, $c = 11.03 \text{ \AA}$. The characterization for the obtained materials were done using UV-VIS, FTIR, Microhardness, SHG and Thermal analysis methods. The structure of the synthesised crystals were found to be orthorhombic. The FTIR spectrum of the synthesized compound reveals the functional groups present in it. The UV-VIS Spectrum shows the optical transmission of the crystal. The SHG of APPB was tested by Kurtz-Perry technique using Nd:YAG laser. TG/DTA analysis shows the thermal stability of the crystal. The mechanical strength was analysed by measuring microhardness of the grown LAPPB crystals.

Key words: Alanine potassium penta borate, single crystal, FTIR, microhardness, TG-DTA

INTRODUCTION

Nowadays, various applications are attributed in the field of NLO crystals and plays an important role in Science and Technology (Muncheryan, 1991; Shen, 1984; Meystrey and Sargent, 1991; Sakai, 1992; Raghavan and Ramasamy, 1999; Ragavan and Ramasamy, 2002). Nowadays, semiorganic nonlinear materials have attracted great attention due to their importance in areas such as efficient signal processing, optical data storage, frequency shifting, optical switching and modulation (Janarthanam *et al.*, 2009). Semiorganic materials combines the important characteristics such as efficient SHG, better mechanical and thermal stability etc., L-Alanine is one of the chiral amino acid. The growth and studies of L-alanine have also been reported (Razzetti *et al.*, 2002; Misoguti *et al.*, 1996; Vijayan *et al.*, 2006). Similarly, the characterizations of L-Alanine Maleate (Balasubramanian *et al.*, 2009; Urit, 2010), Lalanine Tetra Fluro Borate (Rajan Babu *et al.*, 2002), LalanineAcetate (Kumar *et al.*, 2005), L-Alanine sodium chloride (Prabha and Palaniswamy, 2010) Alanine Barium Chloride (Chitra and Palaniswamy, 2010), Alaninium Oxalate (Devaprasad and Madhavan, 2010) have also been investigated. Research articles shows that borate compounds have better non linear response. In this series, potassium penta borate octa hydrate is successfully used for frequency conversion process (Ambujam *et al.*, 2006; Vimalan *et al.*, 2007). The growth and the characterization of potassium penta borate were

reported in the literature (Justin Raj *et al.*, 2009; Raja *et al.*, 1993; Rajasekar *et al.*, 2003; Thamizharasan *et al.*, 2000). In this work, the characterization of L-Alanine Potassium Penta Borate Octa Hydrate is discussed.

SYNTHESIS

The LAPPB was prepared by mixing L-Alanine and potassium penta borate octa hydrate in 2:1 ratio. Using de-ionized water and stirred continuously about two hours. After the complete dissolution of the materials, the solution was filtered and poured in a Petri dish and it was covered by multi holed paper so as to enhance slow evaporation. After 6-7 days the crystals of LAPPB were grown. The obtained crystals were subjected to the following methods.

RESULTS AND DISCUSSION

Single crystal study: Using ENRAF NONIUS X-Ray Diffractometer, the structure of the crystals were calculated. The obtained crystal belongs to Orthorhombic System. The three lattice parameters are $a = 9.04 \text{ \AA}$, $b = 11.5 \text{ \AA}$, $c = 11.03 \text{ \AA}$ and $V = 1111 \text{ \AA}^3$. The axial angles are $\alpha = \beta = \gamma = 90^\circ$.

Ftir analysis: IFS BRUKKER 66V Spectrophotometer was used to determine the functional groups present in the compound by KBr pellet technique. The strong band at 3416 cm^{-1} is due to OH stretching vibration. Symmetric

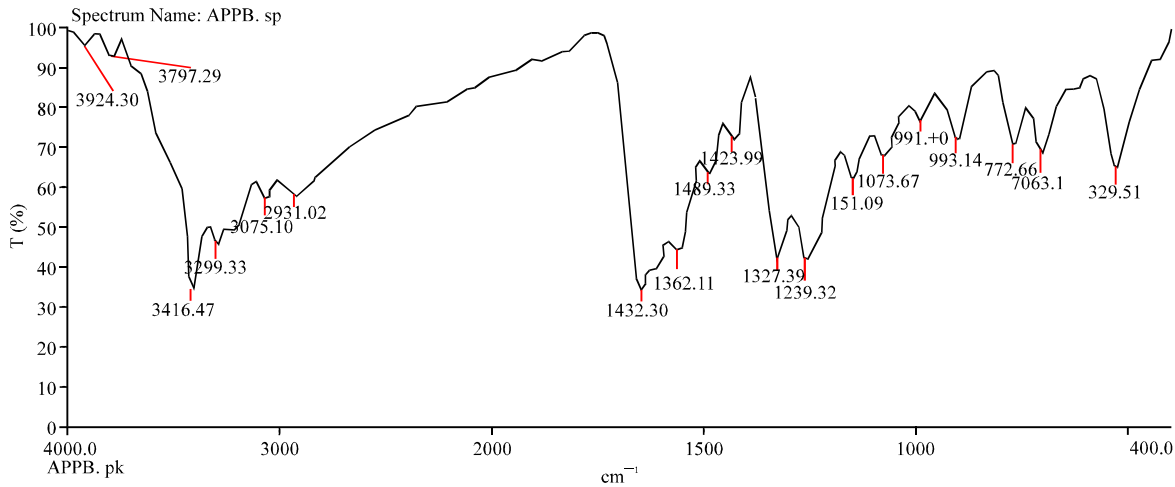


Fig. 1: FTIR-spectrum of LAPPB

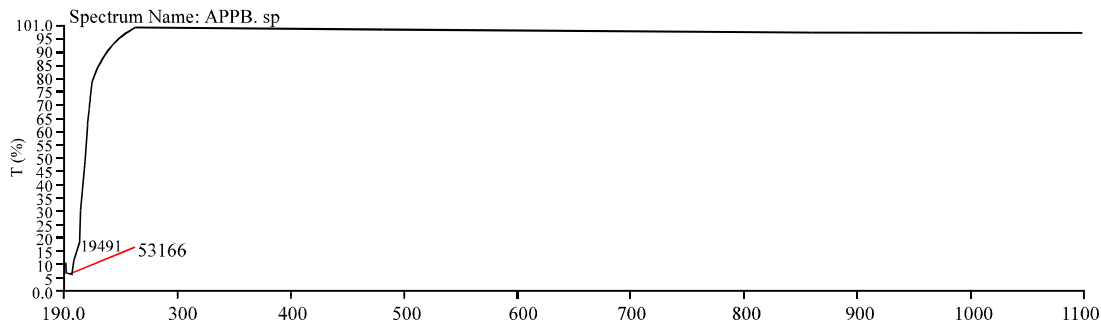


Fig. 2: UV-Vis spectrum of LAPPB

Table 1: FTIR spectral band assignments of LAPPB

Wave No. (cm ⁻¹)	Band assignments
3416	OH stretching vibration
3075	Symmetric and asymmetric stretching vibrations of N-H
2931	Symmetric and asymmetric stretching vibrations of N-H
1562	CH ₂ bending
1489	O-H symmetric stretching
1372	CH ₃ bending
1259	O-H bending of COOH
1151	C-O stretching
905	B-O symmetric stretching
772	B-O symmetric stretching
529	COO ⁻ rocking

and asymmetric stretching vibrations of N-H were assigned at 3075 and 2931 cm⁻¹. The B-O vibrations of Borate group is identified by the absorption bands at 772 and 1428 cm⁻¹ (Justin Raj *et al.*, 2009). The Spectral band assignments of LAPPB is shown in Table 1. The FTIR-spectrum of LAPPB is shown in Fig. 1.

UV-Visible spectrometer analysis: The Optical Transmission of LAPPB crystal was taken in the range

190-1100 nm and it is shown in Fig. 2. The transparency is around 95% within the range of 230-1100 nm. This is the main requirement for the materials possessing NLO property. This high transmission of the LAPPB is due to the electronic transitions associated within the sample. The synthesised crystal shows equal transmission as that of the conventional grown crystals (Justin Raj *et al.*, 2009). The UV-Vis spectrum of LAPPB is shown in Fig. 2.

Microhardness study: LAPPB crystal was subjected to the load variation from 25 to 100 g (Ambujam *et al.*, 2006) in the Vickers Micro hardness test. The Micro hardness number was calculated using the relation:

$$Hv = 1.8544 P/d^2 \text{ kg mm}^{-2}$$

Vickers micro hardness number vs applied load graph is illustrated in the Fig. 3.

It shows that the hardness of the crystal increases with the increase of load. The work hardening coefficient

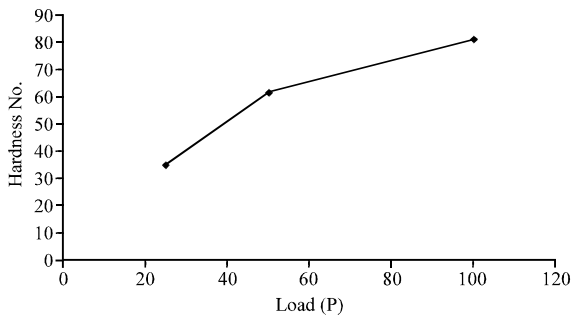


Fig. 3: Micro hardness No. versus load

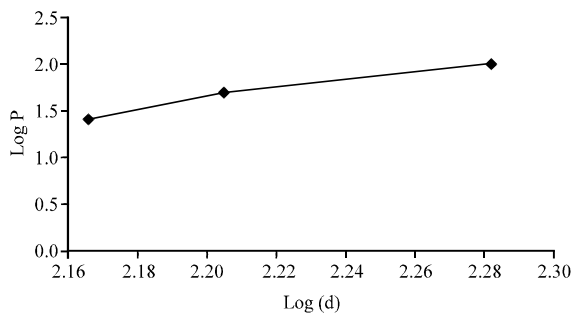


Fig. 4: Microhardening coefficient log P versus log d

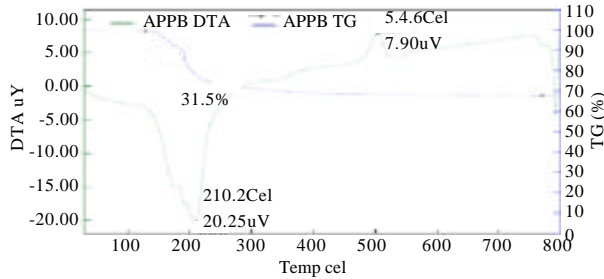


Fig. 5: Thermal studies

n is determined from the plot of log P Vs log d. It is shown in Fig. 4. It is observed that the Vickers hardness profile of the crystal increases with the increase of load (Vimalan *et al.*, 2007). The work hardening coefficient is found to be 5.7. This shows that the synthesized material is a soft material (Lucia Rose *et al.*, 2011).

Second harmonic generation testing (SHG)

Using the Nd: YAG laser beam at 1064 nm, the SHG conversion efficiency of the crystal was carried by Kurtz powder method. An input of 4.1 mJ was used. The SHG was confirmed by the emission of green signal of wavelength at 532 nm. The efficiency is confirmed with KDP and it is 0.27 times that of KDP.

Thermal studies: From the TG/DTA studies, we can find out the thermal stability of the sample. The TG/DTA Spectrum of LAPPB was taken using the instrument "SII EXSTAR 6000". Sample initially weighing 7.387 mg was taken in an alumina crucible and it was heated at a rate of $20^{\circ}\text{C min}^{-1}$ from 30 to 800°C . The TGA curve shows that there is no weight loss upto 156°C and there is weight loss of about 31.6% of the initial mass after 156°C . From the DTA curve, it is observed that there is one endothermic peak at 210.2°C . From this it is concluded that the crystal is decomposed at 210.2°C . This is shown in Fig. 5.

CONCLUSION

Single crystals of L-Alanine Potassium Penta Borate are successfully grown by slow evaporation technique. The above crystal belongs to orthorhombic system that was characterized by single crystal XRD. The UV-VIS spectrum confirmed the device fabrication property of the crystal. The FTIR spectrum confirmed the presence of both Alanine and Potassium penta borate molecules for LAPPB. The NLO test using the Nd-YAG laser proved the NLO property of the crystal. The Vickers Micro hardness study revealed the soft category of the grown crystal. The TG/DTA Analyses explained that the stability of the crystal was upto 153°C and it started melting at 210.2°C .

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