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

K. Kamatchi and T. Radha Krishnan
Department of Physics, SRC, SASTRA University, 612 001, Kumbakonam, India

Abstract: The aim of the work is to synthesise a new semiorganic crystal of L-Alanine Potassium Penta Borate Octa Hydrate [LAPBP]. The LAPBP crystals were synthesised by adopting slow evaporation method. The Size of the materials obtained are $a = 9.04\,\text{Å}$, $b = 11.5\,\text{Å}$, $c = 11.03\,\text{Å}$. The characterization for the obtained materials were done using UV-VIS, FTIR, Microhardness, SHG and Thermal analysis methods. The structure of the synthesised crystals was 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 LAPBP 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 LAPBP 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 combine 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; Uriri, 2010), Lalanine Tetra Fluro Borate (Rajan Babu et al., 2002), Lalamineacetate (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 LAPBP 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 LAPBP 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 $a = 9.04\,\text{Å}$, $b = 11.5\,\text{Å}$, $c = 11.03\,\text{Å}$ and $V = 1111\,\text{Å}^3$ The axial angles are $\alpha = \beta = \gamma = 90^\circ$.

FTIR analysis: IFS BRUKER 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

<table>
<thead>
<tr>
<th>Wave No. (cm⁻¹)</th>
<th>Band assignments</th>
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<tbody>
<tr>
<td>3416</td>
<td>OH stretching vibration</td>
</tr>
<tr>
<td>3075</td>
<td>Symmetric and asymmetric stretching vibrations of N-H</td>
</tr>
<tr>
<td>2931</td>
<td>Symmetric and asymmetric stretching vibrations of N-H</td>
</tr>
<tr>
<td>1562</td>
<td>CH bending</td>
</tr>
<tr>
<td>1489</td>
<td>O-H symmetric stretching</td>
</tr>
<tr>
<td>1372</td>
<td>CH₂ bending</td>
</tr>
<tr>
<td>1259</td>
<td>O-H bending of COOH</td>
</tr>
<tr>
<td>1151</td>
<td>C-O stretching</td>
</tr>
<tr>
<td>905</td>
<td>B-O symmetric stretching</td>
</tr>
<tr>
<td>772</td>
<td>B-O symmetric stretching</td>
</tr>
<tr>
<td>529</td>
<td>COO⁻ rocking</td>
</tr>
</tbody>
</table>

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:

\[ H_v = 1.8544 P/d^2 \text{ kg mm}^{-2} \]

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
**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°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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**REFERENCES**


