Synthesis, Growth and Optical Studies of L-Valine Potassium Nitrate Single Crystal

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Abstract - The Nonlinear optical single crystal of L-Valine Potassium Nitrate (VKN) crystal was grown by slow evaporation technique at room temperature. The powder X-ray diffraction confirms the presence of metal nitrate and the formation of new crystal. Further, it is supported by the presence of COO group vibrations of carboxylic acid and NO₂ vibrations which identified from FT-IR spectra. The UV-Visible spectra show split transmittance curve, which is useful for band pass filter applications. The dielectric studies prove that the sample has low dielectric constant and dielectric loss at higher frequencies. The SHG efficiency of the VKN crystal is found to be 0.9 times that of pure KDP.

Key Words: Semi organic Crystal, L – Valine, Metal nitrate, Band pass filter NLO

1. INTRODUCTION

The design of optoelectronics and photon devices relies heavily in the development of nonlinear optical materials with higher efficiency. The materials possessing large second order nonlinear susceptibility with favorable in thermal and mechanical stability are intensively used in many device applications (1). The development of semi organic materials, where the organic ligand is ionically bonded with inorganic host refined the search of new materials with high optical nonlinearities which is an important area due to their optical applications such as optical communication, optical computing, optical information processing, optical disk data storage, laser fusion reaction, laser remote sensing, color display, medical diagnostics, etc. (2). Amino acid of L-valine has been exploited for the formation of salts with inorganic acids. As a result, good NLO material such as L-valine hydrobromide, L-valinum succinate, and L-valine hydrochloride, N-Glycyl-L-Valine, L-valine cadmium chloride, L-valine nickel (II) Chloride were already reported (3-5). In the present paper the report consist of synthesis of L-valine Potassium nitrate crystal by slow evaporation method and the characterization by powder XRD, FTIR, optical transmission, dielectric analysis, and powder SHG test.

2. Experimental Procedures

The compound L-Valine Potassium nitrate (VKN) was synthesized by dissolving equimolar quantities of L-Valine and Potassium nitrate (VKN) in double distilled water. The resulting solution was stirred well for two hours and a white colored precipitate was obtained. The filtered precipitate and then dried well at room temperature. A saturated solution of L-Valine Potassium nitrate precipitate was prepared in water solvent (10ml) and the resulting solution was stirred well about two hours. Then the solution was filtered using Whatmann filter paper to eliminate if any suspended impurities present in the solution. The clear filtered solution was taken in beaker and placed at room temperature. After 25 days, a good optical and colorless single crystal of L-Valine Potassium nitrate single crystal of (6mm X 3mm X 1mm) were obtained by slow evaporation solution growth technique. The grown L-Valine Potassium nitrate (VKN) single crystal is shown in Fig.1.

Fig. 1. Photograph of L-Valine Potassium Nitrate Crystal

3. RESULT AND DISCUSSION

3.1. Powder X-ray Diffraction

The powder XRD pattern was recorded using Rigaku XRD with CuKα radiation (wavelength 1.5406 Å) and it is shown in Fig. 2.
It reveals that, all the necessary functional groups are present in the compound and are identified in the spectrum. The $NH_2^+$ asymmetric stretching vibration for L-Valine was observed at 3435 cm$^{-1}$ and 1502 cm$^{-1}$. The asymmetric stretching vibration at 684 cm$^{-1}$ is assigned to carboxylic (COO$^-$) group of the amino acid. The stretching vibrations at 1036 cm$^{-1}$ is due to NO$_3^-$ functional group of Metal nitrate (4). From the above vibrational assignments of infrared spectrum it is confirmed that synthesized compound is L-Valine Potassium nitrate (VKN) and the corresponding vibrational assignment are presented in Table 1.

**Table 1 FTIR Vibrational frequency Assignment**

<table>
<thead>
<tr>
<th>Wavenumber(cm$^{-1}$)</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>3423</td>
<td>$NH_2^+$asymmetric stretching</td>
</tr>
<tr>
<td>3173</td>
<td>CH stretching</td>
</tr>
<tr>
<td>2960</td>
<td>CH$_3$ Symmetric stretching</td>
</tr>
<tr>
<td>2580</td>
<td>O-H stretching</td>
</tr>
<tr>
<td>2141</td>
<td>$NH_4^+$symmetric stretching</td>
</tr>
<tr>
<td>1606</td>
<td>N=O stretching</td>
</tr>
<tr>
<td>1502</td>
<td>$NH_2^+$Asymmetric stretching</td>
</tr>
<tr>
<td>1424</td>
<td>CH$_3$ Symmetric stretching</td>
</tr>
<tr>
<td>1323</td>
<td>C-O Stretching</td>
</tr>
<tr>
<td>1178</td>
<td>C-C Stretching</td>
</tr>
<tr>
<td>1036</td>
<td>NO$_3^-$ symmetric stretching</td>
</tr>
<tr>
<td>825</td>
<td>N-O stretching</td>
</tr>
<tr>
<td>773</td>
<td>CH out of plane deformation</td>
</tr>
<tr>
<td>684</td>
<td>carboxylic group of the amino acids</td>
</tr>
<tr>
<td>535</td>
<td>COO$^-$ rocking</td>
</tr>
</tbody>
</table>

3.2 FTIR Analysis

The FTIR spectrum of VKN sample is recorded in the region of 400–4000 cm$^{-1}$ using KBr pellet technique is shown in Fig. 3.

**Fig. 3.** FTIR spectra of L-Valine Potassium nitrate Crystal

The grown crystals show relatively sharp XRD reflection peaks which are due to the high crystalline nature of the grown crystal. However, different variations in the intensity and 2θ values were observed when compared with pure L-Valine (JCPDS NO.22-1930) and Potassium nitrate (JCPDS NO.78-0955). The presence of new peaks in the PXRD pattern of VKN crystal reveals the addition of metal nitrate into L-Valine and the formation new crystal structure.

**Fig. 2.** Powder XRD pattern of L-Valine Potassium nitrate Crystal
3.3 UV-Visible transmission spectral analysis

The optical transmission spectrum of the grown crystal is recorded in the range from 200 nm to 800 nm (Figure 4). UV-Vis spectra of the sample revealed the information about electron excited from the valence band to conduction band (5).

Fig 4. UV-Visible Spectra of L-Valine Potassium nitrate Crystal

The UV-Visible spectra show split transmittance with the cut of wavelengths of 207 nm, 334 nm, and 646 nm, which is useful for band pass filter application. Also the transmission spectra show good transparency therefore wide transmission window exists in visible region. This wide transparency play a vital role in optoelectronic applications and device fabrication.

3.4 Dielectric study

The electrical behavior of the grown crystal was studied over a range of frequency (1 Hz - 50 MHz) at temperature (30°C) using an impedance analyzer PSM 1735 LCR Meter. The variation of dielectric constant with frequency is shown in Fig 5. Materials with low dielectric constant at higher frequencies are important for the fabrication of ferroelectric and electro-optic device (7).

Moreover, the lower value of dielectric constant at higher frequency is a suitable parameter for the enhancement of second harmonic generation efficiency.

3.5 Non Linear Optical Study

Nonlinear optical property of VKN crystal is confirmed by Kurtz-Perry powder SHG test (8) using 1064 nm as a fundamental wavelength. The powdered VKN sample was filled in the micro capillary tube and it was used to analyze the SHG efficiency. A second harmonic signal of 36 mV/pulse was obtained, while the standard potassium dihydrogen phosphate crystal gave a SHG signal of 40 mV/pulse for the same input energy. It is found that, VKN crystal possesses 0.9 times that of KDP crystal.

CONCLUSIONS

L-Valine Potassium Nitrate (VKN) single crystals were synthesized by slow evaporation technique. The powder X-ray diffraction confirms the presence of metal nitrate and the formation of new crystal structures. The functional groups were identified from FTIR spectrum. The presence of COO- group vibrations of carboxylic acid and NO$_2$ vibrations are also evident for the existence of new structure. The UV-Visible spectra shows split transmittance curve, which is useful for band pass filter application. The lower cut of wavelength of the VKN crystal is found to 207 nm and the band gap energy is 5.98 eV. The dielectric studies prove that the sample has low dielectric constant at higher frequencies. The SHG efficiency of the VKN crystal is found to be 0.9 times that of standard KDP. Hence, the splitted transmittance and...
other optical properties of VKN makes it as a potential candidate for optoelectronic applications.

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References


