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Growth and Characterization of L-Threonine doped Potassium Sulphate:A new NLO Semi-Organic Crystal

[1]T. Manimaran, [2]P.Paramasivam, [3]S.Bhuvaneswari, [4]R.S. Abina Shiny, [5]B.Ravindran & [6]M.Mariappan

[1,3&4] Research Scholar, PG and Research Department of Physics, Thiru.Vi.Ka. Govt. Arts College, Thiruvarur 610 003 (India)
[2] Assistant Professor, PG and Research Department of Physics, Rajah Serfoji Arts College(Autonomous)Thanjavur 613 005. (India)
[5]Assistant Professor,PG and Research Department of Physics, Thiru.Vi.Ka.Govt.Arts College,Tiruvarur 610 003. (India)
[6] Assistant Professor,PG and Research Department of Chemistry, Thiru.Vi.Ka.Govt.Arts College,Tiruvarur 610 003. (India)

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***Corresponding Author**
Email: ravitvk74[at]gmail.com

ABSTRACT

L-Threonine doped potassium sulphate crystals have been successfully grown by a slow evaporation technique. Characterization studies such as UV-Vis-NIR, FTIR, XRD, and TGA-DSC were used to analyze the optical and structural properties. Functional groups were confirmed by FTIR, Thermal stability of the grown crystal was observed by TG-DSC spectrum. Occurrence of change in structure of the grown new material was studied using powder XRD pattern. By the Kurtz Perry Powder technique, the Second Harmonic Generation behavior of the grown crystals was studied.

1. Introduction

Semi-organic NLO crystal growth is a fascinating field of research nowadays because of the highly pronounced SHG or THG. L-Threonine is an important organic NLO material with attractive SHG efficiency among the several organic materials. Due to the presence of polar molecules, the SHG efficiency seems to be more on compared with other amino acids (1). The UV-Vis-NIR, FTIR, XRD, TGA-DSC, and SHG efficiency studies have been carried out for the grown crystal. Most of the research works have been carried by current researchers on pure and metal ions doped crystals of amino acid (2-5). Optoelectronics device fabrications and photonic applications are in a need of optical materials with special NLO behavior. In this study, the L-Threonine doped potassium sulphate crystals were grown by slow evaporation solution growth technique from the aqueous solution. The pronounced SHG efficiency was compared with the frequently used commercial NLO material such as KDP.

2. Experimental Procedure

Using deionised water Potassium sulphate has synthesised successfully doping with L-Threonine. An optically transparent NLO crystal has grown in a aqueous solution adopting solution growth slow evaporation technique at room temperature. The dissolved solution was taken in a clean beaker and stirred well using a magnetic stirrer for about 3 hours. Then the prepared solution was filtered by Wattmann filterpaper and collected in a clean beaker. The top of the beaker is closed with a perforarted plastic cover and kept in an undisturbed position. Under periodical observation over a period of month a single crystal has been collected under a dimension of 45mm length as shown in the figure 1.By repeated crystallization process the size and quality were improved.

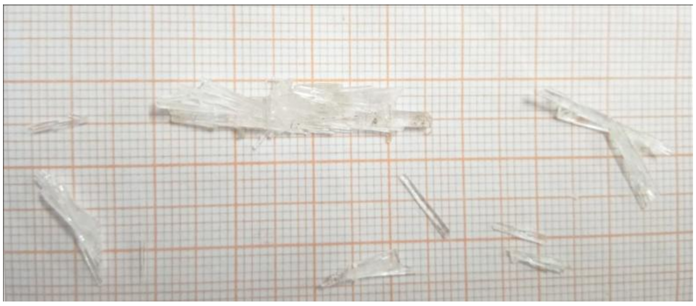

Fig.1. Morphology of LTPS

3. Results and Discussion

3.1. UV-Vis-NIR Analysis

Excitation of electrons from its ground level to higher energy state occurs when a molecule is exposed by ultraviolet of visible light. The spectrometer of Lambda 35 is used to predict the spectra of optical transmittance of the grown crystal. Because of the wide transmission window, the UV-Vis-NIR transmission spectrum plays a vital role in determining the quality of an optical material. From the recorded spectrum the high transparency was confirmed on absorption that no peaks were found within a range 235-1100nm. Here we came to note about the advantage of doping L-Threonine on potassium sulphate resulting in wide transparency due to the absence of strongly conjugated bands. The suitability of the grown crystals for optoelectronic applications is justified by its good transparency due to its lower cutoff wavelength found around 235 nm attracts to concentrate on the enhancement of optical behavior(6).

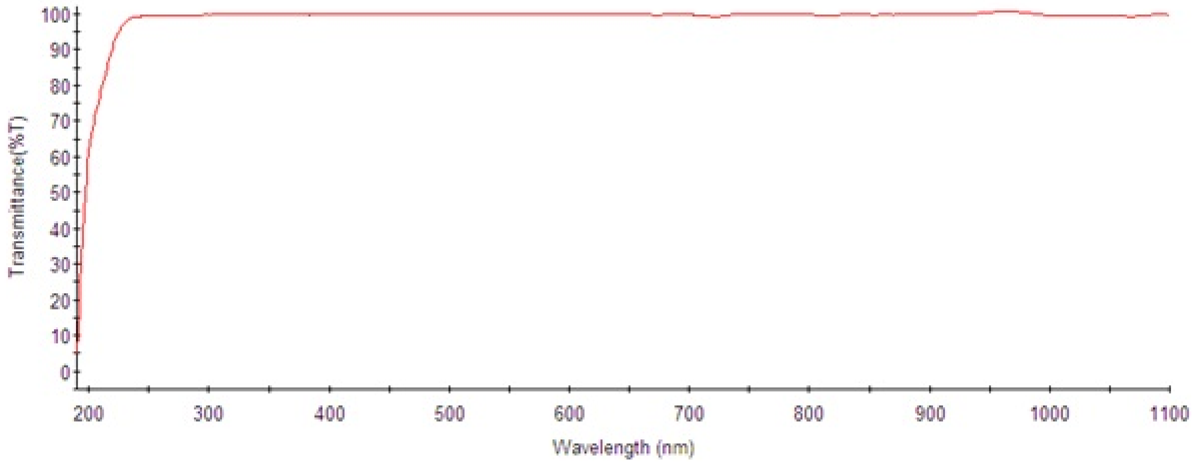

Fig.2. UV-Visible spectrum of LTPS

3.2. FT-IR Analysis

Ten different inorganic sulphates have been examined by Wilkins and Miller. Hunt et al. reported two other sulphates. A very strong band is shown in all the above said cases found in the range 1130-1080 cm^{-1} followed by a weaker band considerably in most of the cases lies within a range 680-610cm^{-1}. Miller et al. confirmed those findings. A strong band centered due to potassium sulphate was found at 1117cm^{-1} and at 618cm^{-1}, a bending vibration mode has occurred. Inclusion of L-Threonine took the responsibilities for the following peaks; one of them is the NH_2 asymmetric deformation occurs at 1627cm^{-1}. And at 2976cm^{-1} a small peak was observed due to the assignment of –CH stretching vibration which confirms the inclusion of the dopant.

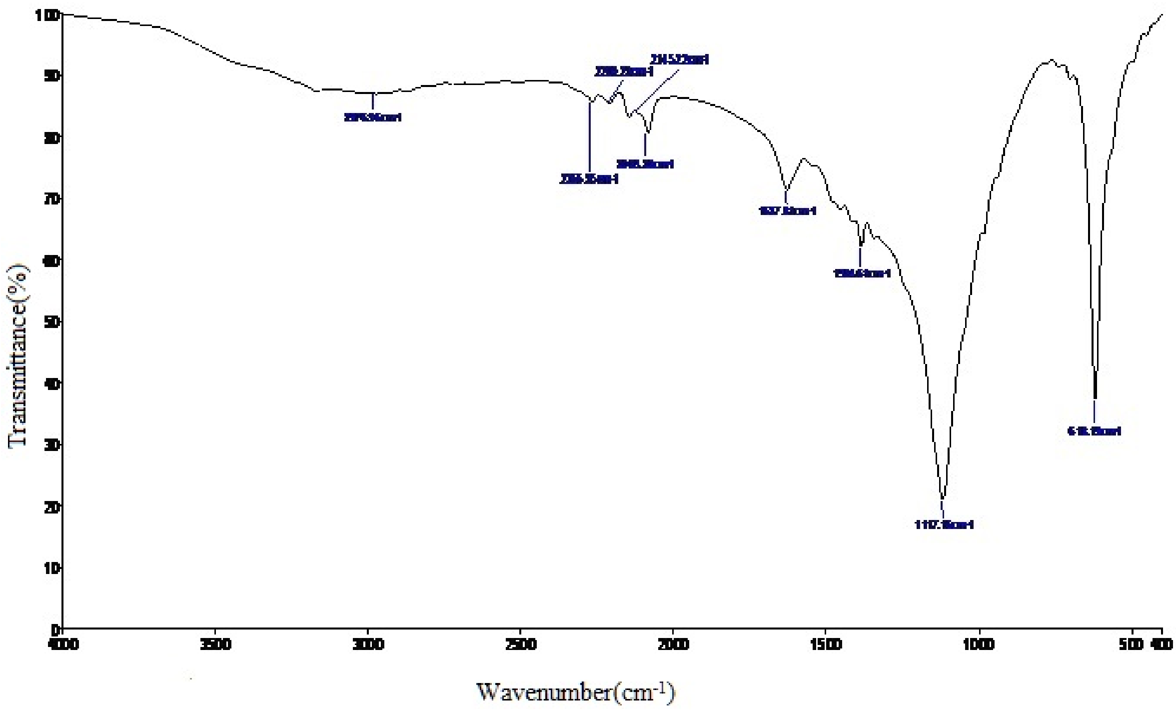

Fig.3.FT-IR spectrum of LTPS

3.3. TGA-DSC Analysis

In nitrogen atmosphere the Differential Scanning Calorimetry and Thermogravimetric analysis were carried out using Thermal analyzer. Fig.4. shows the TGA-DSC curve of LTPS. It was observed on heating compound in the temperature range 0-1000°C. From 0-368 °C, Co_2 and NH_3 molecules present in the dopant L-Threonine on potassium sulphate lost on heating the compound. Between the temperatures range of 237.5°C to 287.5°C occurrence of lower weight loss was observed. The inclusion of L-Threonine was confirmed on the first stage of decomposition. At a temperature rang 368°C the remaining portion of potassium sulphate decomposed steeply. Presence of donor and parent compound was indicated by the low and high weight-loss percentage. It was observed from the TGA spectrum that the 28.5% weight loss occurred due to L-Threonine and the 55.35% weight loss was for potassium sulphate.

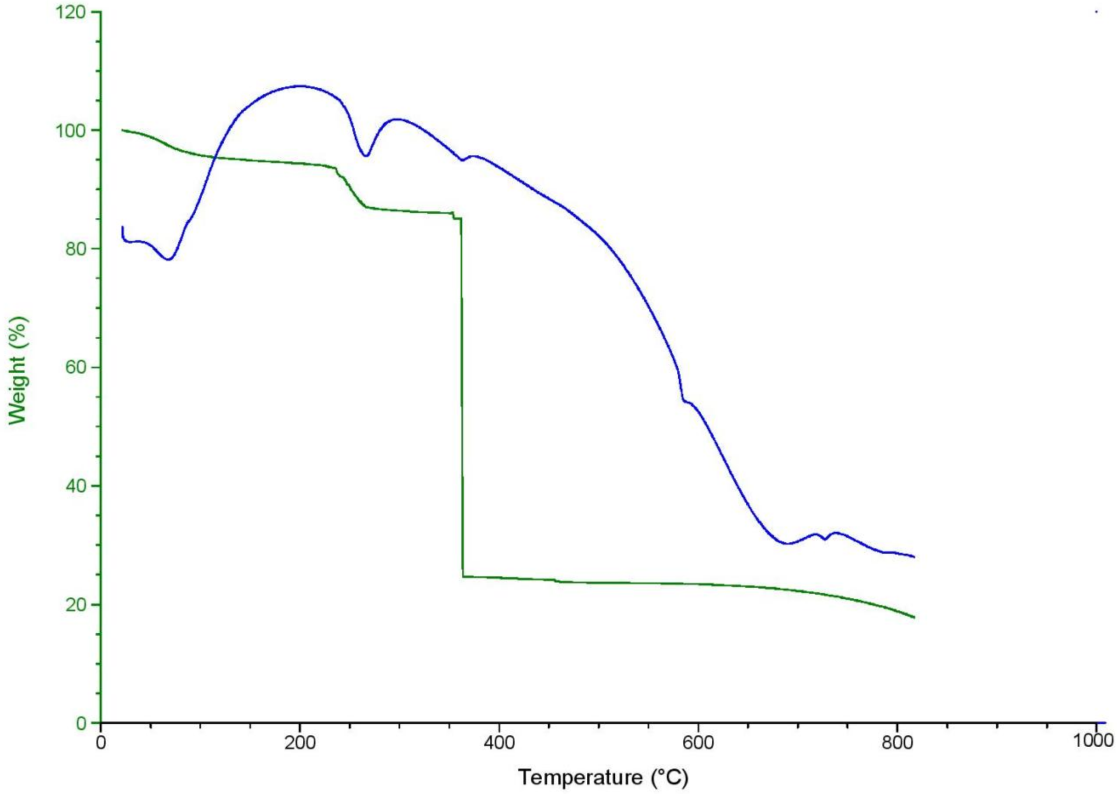

Fig.4. TG-DSC spectrum of LTPS

3.4. X-Ray Diffraction studies

Crushed fine powder with uniform particle size of the grown LTPS crystals are subjected to diffraction studies with X-ray using advance AXSD8 powder diffractometer with copper(λ=1.5406A°). Scanning of the specimen was done in the mode of reflection in the 2θ range 20-80°. The diffraction pattern observed are shown in table 2 which results in the nature of purity and good crystalline nature (7).

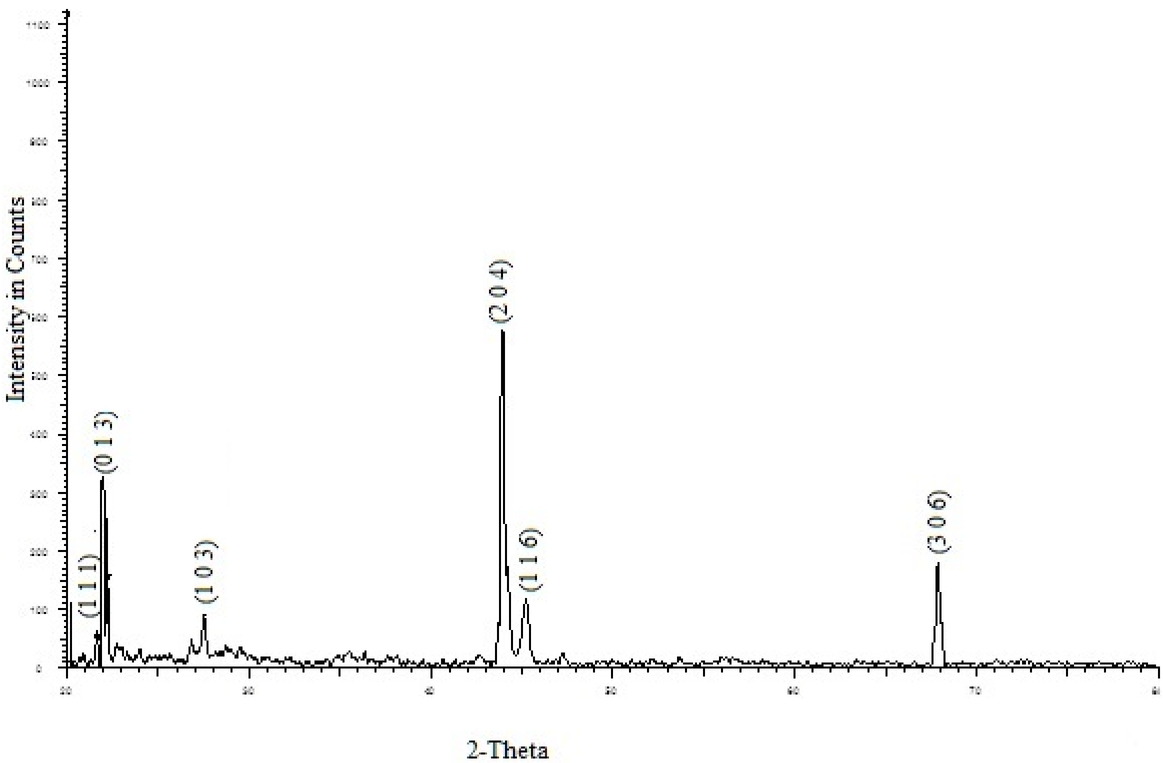

Fig.5. XRD spectrum of LTPS

Table 1.Powder XRD of LTPS

θ	hkl
21.586	110
21.894	111
22.155	013
27.453	103
43.884	204
45.183	116
67.901	306

3.5. NLO Studies

The grown LTPS crystal was illuminated by an input pulse of energy 6.2mj in powdered form which was rigidly packed in capillary tube with bore uniformity fused on one side. The SHG efficiency of the grown crystal was detected and recorded using Kurtz Perry Powder technique (8-9). The green light emerged out from the sample placed in holder confirms the NLO behavior of the grown crystal. Comparison of obtained result of grown crystal with KDP was done for knowing the effect of doping.

4. Conclusion

Grown L-Threonine doped potassium sulphate crystals by solution growth slow evaporation technique at room temperature were subjected to various characterizations and the dopant incorporation were confirmed. The transmission range was calculated from the U-Vis-NIR spectrum. Availability of amino and functional groups was studied using the trace of FTIR analysis. At about 90% of transparency was studied along with the lower cutoff wavelength235nm.Thermal stability of grown crystals absorbed at 368°C shows that the LTPS crystals may be used for device fabrication as 368°C as its working temperature. SHG efficiency of the grown crystal was carried out and output is compared with KDP shows the suitability of grown crystals for NLO device fabrications.

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