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Synthesis and Characterization of 0.5 mol % KHP doped L-Asparagine Monohydrate

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ABSTRACT

0.5 mol% KHP doped L-asparagine monohydrate (LAM) single crystal was synthesized by slow evaporation technique. The lattice parameters are observed by single crystal X-ray diffraction method and the lattice parameters are a= 5.592 Å b=9.825 Å c=11.811 Å. The wide transparency range of the doped crystal in the visible region is identified by UV-Vis-NIR spectrum. The modes of vibration of different functional groups were confirmed by FTIR spectrum. The thermal property of the crystal was reported from TGA/DTA analysis Nonlinear optical property of the crystal was confirmed by Kurtz and Perry powder method.

Keywords: Solution growth; Optical analysis; Thermal analysis; Second harmonic generation

1. INTRODUCTION

Nowadays the materials exhibiting SHG have a great demand because of its technological importance in various fields such as optical communication, optical parametric oscillation, frequency mixing and information storage. According to crystal engineering, SHG occurs mostly in non centrosymmetric crystal due to nonzero hyperpolarizability. Organic materials with nonlinear optical activity generally consist of a π – electron conjugated structure. The conjugated π -electron moiety provides a pathway for the entire length of conjugation under the perturbation of an external electric field. The presence of π bond system with appropriate electron donor and acceptor group can increase the asymmetric electronic distribution in either or both the ground and excited state, thus leading to an increased optical nonlinearity. L- asparagine is one of the fundamental natural amino acid present in proteins and it plays an important role in the formation of the secondary structures in proteins due to the fact that the side chain can form efficient hydrogen bond with the peptide backbone. It crystallizes in structures exhibiting a complex network of hydrogen bonds among asparagine and water molecules which makes the material very interesting to investigate. Many L-asparagine complexes have been synthesized recently and its characterization studies have been reported. In the present work KHP doped L-asparagine monohydrate crystals were synthesized and it is subjected to characterization studies.

Experimental Details: Highly pure L-asparagine monohydrate was dissolved in double distilled water and purified by the repeated recrystallization process and the recrystallized material was used to prepare the saturated solution. 0.5 mol% of KHP was added to the saturated solution. The solution is filtered and kept in undisturbed position. After a period of 25 days colorless, transparent crystals were harvested and the crystal is shown in fig. 1.

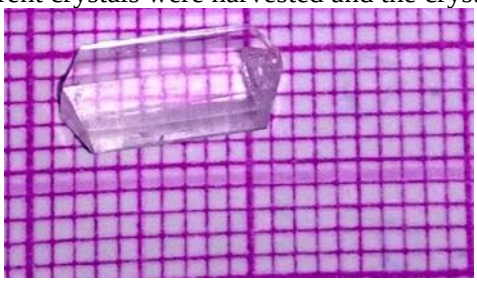

Figure.1. 0.5 mol% KHP doped L-asparagine monohydrate

Characterization Studies:

Single crystal X- Ray diffraction studies: The single crystal XRD analysis was carried out using ENRAF Nonius CAD4 single crystal x-ray diffractometer. It is used to confirm the crystalline nature of the grown crystal. The lattice parameters are a= 5.592 Å b=9.825 Å c=11.811 Å and α=β=γ= 90°. The crystal crystallizes in orthorhombic system with a space group of $P2_12_12_1$.

Optical studies: The UV-Vis spectrum explains valuable information about the atomic structure of the molecules because the absorption of UV and visible light involves the promotion of σ and π orbital electrons from the ground state to higher energy state. The absorption spectrum is recorded using Shimadzu 1601 UV – Vis spectrophotometer in the range of 200nm-900nm. From Fig.2 the optical absorption spectrum indicates the lower cut-off wavelength is 245 nm. From the absorption spectrum, it can be observed that there is less absorbance in the visible and near infrared region.

FTIR analysis: The Fourier transform infrared spectrum of KHP doped L-asparagine monohydrate was recorded using Perkin-Elmer FTIR spectrometer using KBr pellet technique in the range 4000 - 400 cm^{-1} (fig 3) . The characteristic vibrations corresponding to the identity of the functional groups present in the compound are represented in Table.1.

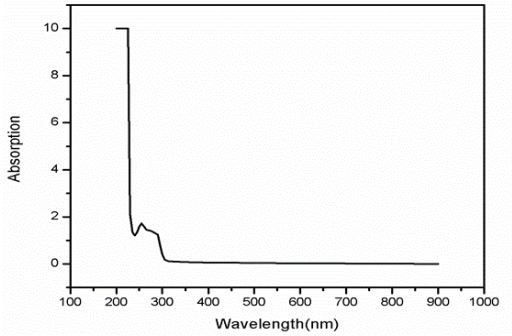

Fig.2.UV-Vis Spectrum of KHP doped L-asparagine crystal

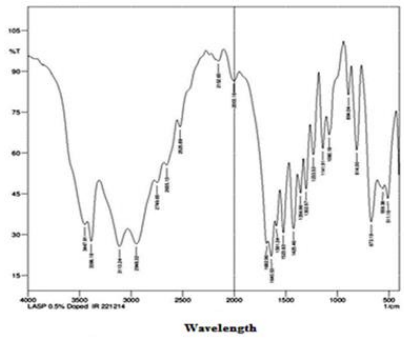

Figure.3.FTIR Spectrum of KHP doped L-asparagine crystal

Table.1.FTIR Spectral assignments of KHP doped L- asparagine monohydrate

Wave number (cm^{-1})	Vibrational Assignments
3447.91	O-H stretching
3386.18	NH_2 stretching
3113.24	NH_3^+ stretching
2948.32	C-H stretching
2003.16	N-H stretching
1640.53	NH_3^+ deformation
1523.83	NH_2 bending
1425.46	COO^- symmetric stretching
1356.98	CH bending
1302.97	CH_2 wagging
1233.53	NH_2 rocking
1141.91	NH_3^+ rocking
1080.80	C-N stretching
894.04	CH_2 rocking
814	C-C stretching
673.19	H_2Orocking
511.16	C-N torsion

Thermal analysis: The TG/ DTA analysis give information about the phase transition and different stages of decomposition takes place in a crystal system. The analysis was carried out in nitrogen atmosphere at a heating rate of 20°C/min from 20°C – 800° C and it is shown in fig.4. From TGA it is observed that the weight loss occurs in three stages. In first stage the weight loss starts at 106°C which may be due to the presence water molecules. The second and third stage of decomposition occurs at 215°C and 254°C which is due to the decomposition of major compounds. In DTA, two endothermic peaks are observed at 114.9°C and 253.7°C which is assigned to decomposition of the compound.

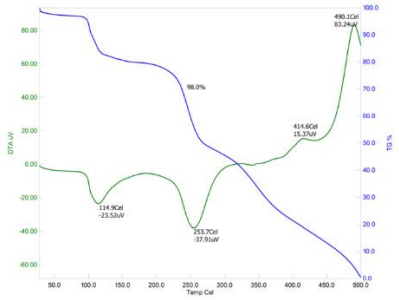

Figure.4.TG/ DTA curve of KHP doped L-asparagine crystal

SHG studies: The SHG efficiency of KHP doped L- asparagine monohydrate was determined by Kurtz and Perry method. The KHP doped L-asparagine crystal was finely powdered and it was packed densely between two transparent glass slides. A high intensity Nd:YAG laser beam of wavelength 1064 nm was used with a pulse duration

of 10 ns. The second harmonic signal generated in the crystal was confirmed by the emission of green radiation of wavelength 532 nm. The SHG efficiency of the KHP doped L- asparagine monohydrate crystal is 0.8 times greater than KDP crystal.

2. CONCLUSIONS

KHP doped L-asparagine monohydrate single crystals were successfully grown by slow evaporation method. The cell parameter was measured by single crystal XRD and the crystal belongs to orthorhombic system. The functional groups of the compound have been determined by FT-IR spectrum. From UV-Vis studies the absence of absorption peak in the entire visible region making it suitable for optoelectronic device fabrication. From thermal studies it is observed that the crystal is stable up to 106°C. The NLO efficiency of the material was observed by Kurtz and Perry method and it is good for NLO applications.

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