International Journal of Researches In Biosciences, Agriculture & Technology | Feb. 2015, Special Issue (2) | ISSN No. (Online) 2347-517X

Growth Structural and Spectral Studies on L- Leucine Doped Ammonium Dihydrogen Phosphate Single Crystals

B. A. Shingade#, N. M.Gahane*, N. S. Meshram*, P. S. Sawadh$, K. G. Rewatkar*

#Department of Physics, Bhawabhuti Mahavidyalaya, Amgaon, India
$Department of Physics, Bapurao Deshmukh College of Engineering Sewagram, Wardha,India
*Department of Physics, Dr. Ambedkar College,Deekshabhoomi, Nagpur-440010,India
***E-mail**:*bashingade@gmail.com*

Abstract

Single crystals of pure and L-Leucine doped Ammonium Dihydrogen Phosphate (ADP) were grown from aqueous solutions, employing slow evaporation technique at room temperature. The grown crystals were subjected characterized by powder Xray diffraction to analyze their structural parameters. Fourier transform infrared (FTIR) spectral analysis was performed to identify the presence of various functional groups in the crystals. The UV-Visible-NIR spectral analysis was carried out to confirm the improvement in the transparency of the ADP crystal on the addition of L- Leucine. The studies performed have revealed the incorporation of L- Leucine into the lattice of ADP crystal.

Key Words: ADP; L-leucine; Crystal growth; X-ray diffraction;; Single crystal etc.

1. INTRODUCTION

Ammonium Dihydrogen Phosphate (ADP) is a representative of hydrogen bonded materials that possesses excellent dielectric, piezoelectric, anti-ferroelectric, electro-optic and nonlinear optical properties. Growth and studies of ammonium dihydrogen phosphate is a centre of attention to researchers because of its unique properties and wide applications. Single crystals of ADP are used for frequency doubling and frequency tripling of laser systems, optical switches in inertial confinement fusion and acousto-optical Devices [1]. ADP crystallizes in a body centered tetragonal structure with the space group I 4 2d and has tetra molecular unit cell [2] with unit cell parameters a = b = 7.6264 Å and c = 7.7151 Å. ADP has been the subject of a wide variety of investigations over the past decades. Reasonable studies have been done on the growth and properties of pure ADP [3-4]. In recent years, efforts have been taken to improve the quality, growth rate and properties of ADP, by employing new growth techniques, and also by the addition of organic, inorganic and semi organic impurities [5, 6]. Organic nonlinear optical materials have large optical susceptibilities, inherent ultrafast response times, and high optical thresholds for laser power as compared with inorganic materials. Amino acids are interesting materials for NLO applications as they contain a proton donor carboxyl acid (-COOH) group and proton acceptor amino (-NH2) group in them [7]. Amino acids, when added as impurities, have improved material properties [8]. Amino acid, L-leucine has formed several complexes, which are promising materials for second harmonic generation [9, 10]. In the light of research work being done on ADP crystals, to improve the properties, it was thought interesting and worthwhile to investigate the effect of L-leucine on ADP. In this work, the structural spectral and nonlinear optical behaviour of single crystals of L-leucine added ADP against pure ADP has been studied and reported.

2. EXPERIMENTAL

Ammonium dihydrogen phosphate and L-leucine (Merck-Germany) along with de-ionised water were used for the growth of single crystals. ADP was mixed with L-leucine in the ratio 1:0.09 to prepare 100 ml of saturated solution at 30°C The solution was stirred for four hours using magnetic stirrer and filtered using Whatman filter paper. The filtered solution was transferred to borosil glass beaker. It was porously sealed and placed in a dust free atmosphere for slow evaporate n. 100 ml of saturated solution of pure ADP was also prepared with de-ionised waterat 30°C.. The solution was stirred for four hours using magnetic stirrer. It was then filtered using Whatmann filter paper, transferred to borosil glass beaker, porously sealed and kept in a dust free atmosphere for slow evaporation. The grown crystals were harvested after a period of 30 days. Pure and 0.9 mol% L-leucine added ADP crystals.

3.RESULT AND DISSCUSION

Powder X-ray diffraction (XRD) studies: Powder X-ray diffraction studies was carried out for the pure and 0.9 mol% L-leucine added ADP crystals using XRERT PRO Diffractometer with copper (K- Alpha 1) radiation (λ= 1.54056 Å) operating at a voltage of 40 kV and a current of 20 mA. The scanning rate was maintained at 1.6° / min over a 2θ range of 10 - 70° employing the reflection mode for scanning. All the reflections of powder XRD pattern have been indexed using the TREOR software package following the procedure of Lipson and Steeple [11]. The indexed X-ray powder diffraction patterns of pure and 1 mol% L- added ADP crystals are shown in figure 2 and figure 3 respectively. The sharp peaks

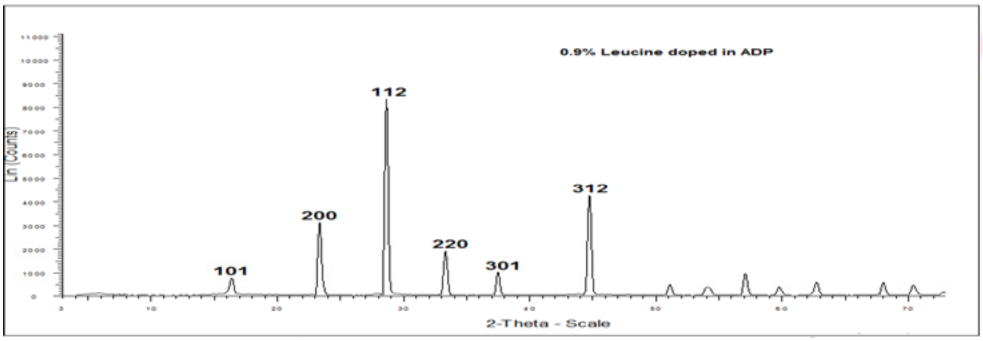


Fig. 1: XRD of Leucine doped ADP

indicate the crystallinity of the grown crystals. There is slight shift in the diffracted peaks in the XRD pattern of L- leucine added ADP crystals when compared to that of ADP. The QAobserved prominent peaks are (101), (200), (112), (220), (301) and (312).

International Journal of Researches In Biosciences, Agriculture & Technology
Feb. 2015, Special Issue (2)
ISSN No. (Online) 2347-517X

The intensity of the diffracted peak (112) is found to vary in the XRD pattern of L-leucine added ADP crystal. The above mentioned changes are due to the presence of the additive L-leucine into the lattice of ADP crystal. However, there are no other phases emerging besides the tetragonal system. The observed results are in good agreement with the reported values [12]. The unit cell parameters of pure and L-leucine added ADP crystals were calculated using "UNIT CELL" software package as a=b=7.4972 Å, c=7.5438 Å, volume=424.0246 $(Å)^3$ for pure ADP and a=b=7.4917 Å, c= 7.5429 Å, volume=423.3511 $(Å)^3$ for L-leucine added ADP respectively.

Fourier Transform Infrared Spectroscopy

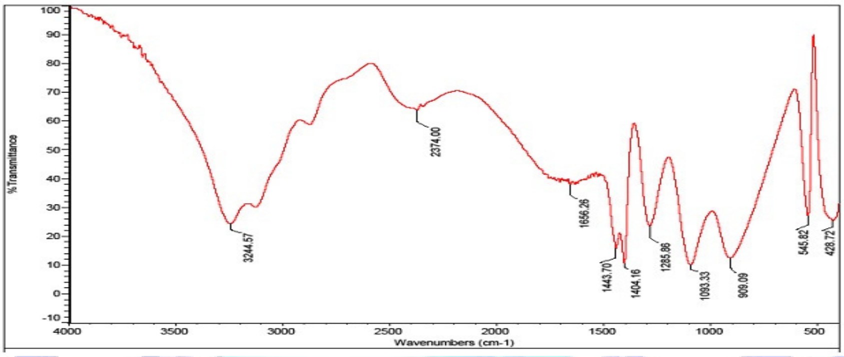
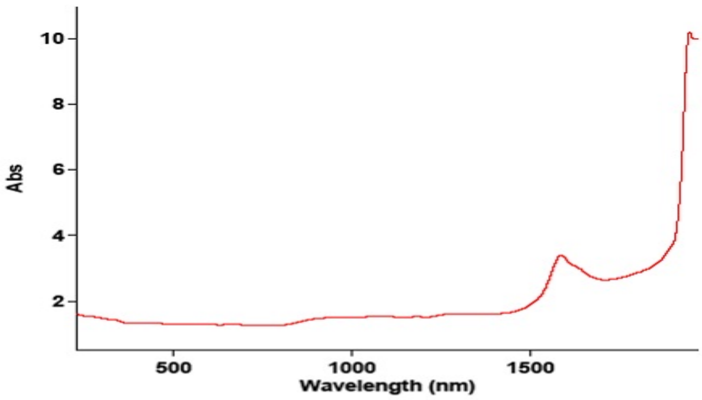


Fig. 2: FTIR of Leucine doped ADP

The FTIR spectra were taken from the Perkin Elmer RXI.FTIR spectrometer in the range of 450 – 4000 cm-1. The spectrographs are shown in fig. 1. and fig.2. By comparing the peaks obtained from the spectrum with standard values, it is clear that 1637.47, 1100.47, 1584.98, 1086.55 correspond to the peaks of ADP [13, 14]. It also closely matches with literature values. Therefore it confirms the presence of ADP.

UV –VIS Spectral studies



ig. 3: UV-VIS of Leucine doped ADP

The UV-VIS spectrum of the crystal was recorded on the region 190-2000 nm using Perkin Elmer Mode - Lambda Spectrometer. Pure ADP have more absorbance in the entire spectral range in comparison with the doped specimen as shown in fig. for doped ADP , better lower cut- off wavelength is noted. The transparency of doped crystal is better thereby enabling to be a good candidate for electro –optic application and also SHG efficient.

4. CONCLUSION

Optical quality, colourless and transparent single crystals of pure and 0.9 mol% L-leucine added in ADP were grown employing slow evaporation solution growth technique. The single crystal and powder XRD studies reveal that the tetragonal structure of ADP is preserved and that the lattice of ADP crystal is slightly distorted due to the addition of L-leucine. The FTIR spectra confirm the presence of all the functional groups and the presence of L-leucine in the grown crystals. The optical transmission spectrum shows good transmission in the entire visible and NIR region for both the crystals with higher transmission for the L-leucine added crystal.

References

1. N. Zaitseva, L. Carman, Prog. Crystal Growth Charact. **43** (2001) 1.
2. L. Tenzer, B.C. Frazer, R. Pepinsky, Acta Cryst. 11 (1958) 505.
3. H.V. Alexandru, J. Cryst. Growth 10 (1971) 151.
4. W.J.P. Van Enckevort, R. Janssen-van Rosmalen, W.H. Van der Linden, J. Cryst. Growth 49 (1980) 502.
5. P. Rajesh, P. Ramasamy, Materials Letters 63 (2009) 2260.
6. P. Rajesh, P. Ramasamy, Physica B 404 (2009) 1611.
7. P.Selvarajan, J.Glorium Arul Raj, S.Perumal, J. Crystal Growth 311 (2009) 3835.
8. P. Kumaresan, S. Moorthy Babu, P.M. Anbarasan, Optical Materials 30 (2008) 1361.
9. S.A. Martin Britto Dhas, G. Bhagavannarayana, S. Natarajan, J. Cryst. Growth 310 (2008) 3535.
10. G. Anantha Babu, P. Ramasamy, Mater. Chem. Phys. 113 (2009) 727.
11. H. Lipson, H. Steeple, Interpretation of X-ray Powder Diffraction Patterns, fifth edi. Macmillan, New York, 1970.
12. Dongli Xu, Dongfeng Xue, J. Cryst. Growth 286 (2006) 108.
13. Yoshiharu Murata, Kaoru Wada and Masakuni Matsuoka, Tokyo University of Agriculture and Technology, Koganei, Tokyo 184, Japan Received 25 November 1982; accepted 22 February 1983.
14. Russel S Drago, "Physical Method in Inorganic Chemistry", East-West Press Pvt. Ltd, New Delhi (1968).