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Influence of L-Histidine on Growth, Structural and Optical Properties of Ammonium di-hydrogen phosphate (ADP) single crystal by Rotation Method

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Abstract:

Pure Ammonium Di-hydrogen Phosphate (ADP) and Ammonium di-hydrogen Phosphate doped with L-Histidine have been synthesized by *rotation crystallization method* at constant temperature 35^0 C. The required seed crystals for *rotation crystallization method* have been grown by slow evaporation at room temperature in a dust free atmosphere. The lattice parameters were determined by powder XRD pattern to know the structure. The well-developed pure ADP and L-Histidine doped ADP crystal have been analyzed by FT-IR to identify the various functional groups. SHG, EDAX, measurements are also carried out to judge the structure and nature of optical crystal.

Keywords: ADP, Crystal growth, L-Histidine, PXRD, FT-IR, EDAX, SHG.

1. Introduction:

Crystals are the pillars of modern technology, without them; there would be no electronics, photonic industry, no fiber optic communication, very little modern optical equipment and some very important crystals in conventional production engineering [1-4]. Among these crystals Ammonium di-hydrogen Phosphate (ADP) are one of the important single crystal which has a beauty of gem and has emerged as a promising inorganic material with application in the area of nonlinear optics (NLO), electro-optics, telecommunications, holography, optical communication devices, color displays, optical modulation Q switch, quantum electronics, frequency converter etc. L-Histidine (Amino Acids) doped ADP crystals act as sharp tool in nonlinear optics [5-8]. In many complexes L-Histidine enhances the properties of ADP crystal [9-11].

This research paper present the comparative study of the grown pure ADP and L-Histidine doped ADP crystals by *Rotation Crystallization Method.* The grown crystals have been characterized by Powder XRD, FT-IR, EDAX, SHG etc. The results obtained for pure ADP and doped with different concentrations in terms of mole percentage of L-Histidine doped ADP crystals are given below.

2. Experimental:

Crystal growth: Solution of Ammonium di-hydrogen Phosphate (ADP) of analytical reagent (AR) grade prepared with triple distilled water and kept it in dust free atmosphere at room temperature. Within 25 to 30 days seed crystal are formed. These seed crystals are used for preparing L-Histidine doped ADP crystals by *rotation crystallization method* with 0.4 mole%, 0.8 mole%, 1.2 mole%, concentration of L-Histidine.

All these crystals are grown at constant temperature at 35^0 C, which was maintained at constant temperature bath using temperature controller. The good quality transparent crystals of suitable size (nearly 1.4 cm) grown in 14-15 days. The grown crystals of pure ADP and ADP doped with various concentration of L-Histidine of good quality are as shown in fig. 1.

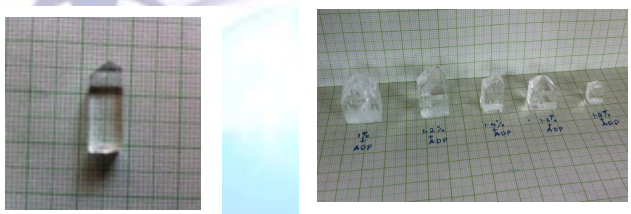


Fig. 1(a) photograph of pure ADP (b) photograph of L- histidine doped ADP

3. Result and Discussions:

3.1 Powder X-Ray diffraction (PXRD) Analysis

Powder of grown pure ADP and L-Histidine doped crystals were analyzed by XRD studies. The powder sample were loaded into X-Ray diffractometer with radiation (λ=1.5406 Å) with an operating voltage 40kV and current 35mA. Scanning rate was maintained at 32.8 s over a 2θ range of $10\text{-}80^0$. From XRD measurement we found the lattice parameters as a=b= 7.510 Å and c= 7.654 Å for pure ADP and lattice parameters of L-Histidine doped crystals are well matched with the result reported [12], having symmetry space group $I\bar{4}2d$ and result shows that L-Histidine entered into ADP lattice. No additional peaks are present in the XRD spectra of doped ADP crystal, showing the absence of any additional phases besides the tetragonal system, due to doping.

The observed prominent peaks of all L-Histidine doped crystals are (101), (200), (112), (202), (301), (213), (114), (204), (323), are shown in fig. (2).The variation in intensity of diffracted peaks are found. The differences in the peak amplitude can be ascribed to the different sizes and orientation of the powered grains. The degree of sharpness of peaks

indicates the crystallinity of the grown crystals. There is small variation in lattice parameters with concentration.

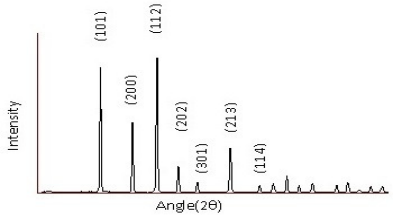


Fig2 (1) PXRD of pure ADP

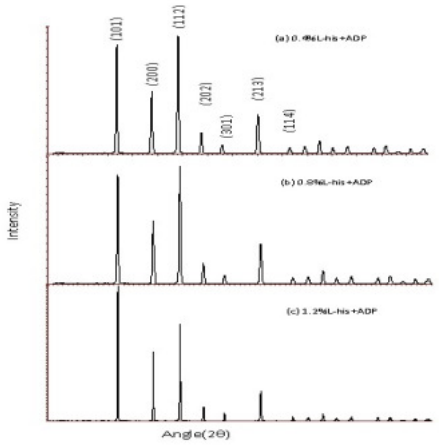


Fig. 2(a,b,c) PXRD of L-Histidine doped ADP

3.2 Fourier Transforms Infrared(FT-IR) analysis:

The powdered samples of L-Histidine doped ADP were also attempted to Fourier Transform Infrared (FT-IR) investigation. The spectrum was observed from VARIAN resolution pro FTIR spectrometer in the range 400- 4000 cm $^{-1}$ by KBR pallet technique. The prominent peaks in the FT-IR pattern for different concentration of L-Histidine doped ADP crystals are shown in the fig. (3).The FT-IR spectra of pure ADP and L-Histidine doped ADP shows that band in the high energy region is due to free O-H stetching of water, P-O-H group of pure and L-Histidine doped ADP[13]. Graphs of pure ADP and L-Histidine doped ADP have high similarities which indicate pure ADP peaks are predominant over L-Histidine peaks due to very small doping of L-Histidine. From FT-IR spectrum of pure and L-Histidine doped ADP it is been observed that all major peaks have shifted towards the higher wave number region, which indicates that dopant L-Histidine has brought about this changes.

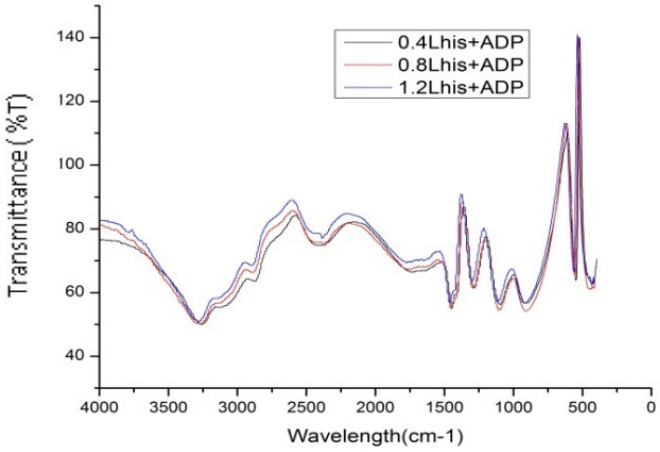


Fig. (3) FT-IR of L-Histidine doped ADP

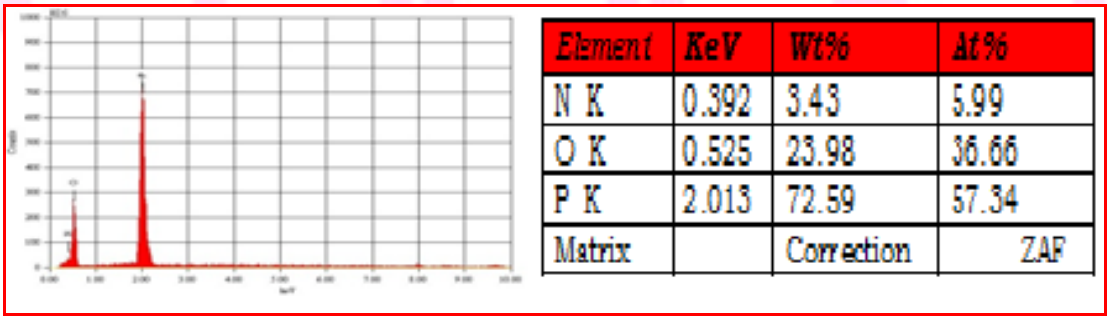
The characteristics absorption frequencies of various functional groups are given in the following table1.

Table:-1

Sr. No.	Frequency Range	0.4 mole%+ADP	0.8 mole%+ADP	1.2mole% +ADP	Bond Assignments
1	3700-3100	3258.35	3259.84	3259.86	O-H Stretching
2	2800-2400	2364.35	2409.45	2364.46	Vibration of combination bond
3	1450-1200	1442.28	1443079	1731.30	Bending vibration of NH_2
4	1100-900	1095.03	1097.76	1283.70	P-O-H vibration
5	550-430	544	545.56	545.03	PO_4 Vibration

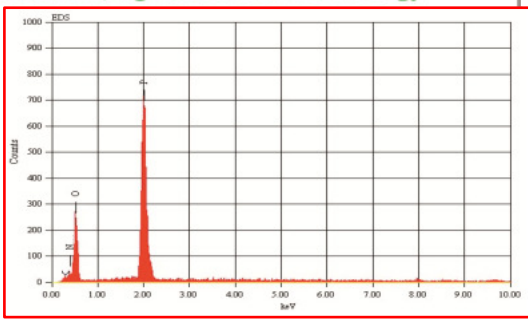
3.3 Energy Dispersive X-ray diffraction (EDAX)

The presence of L-Histidine was confirmed by EDAX analysis. In the present study, the chemical composition of the crystal was analyzed by INCA 200 energy dispersive X-ray micro analyzer equipped with LEO Steroscan 440 Scanning electron microscope. The EDAX spectra for pure and L-Histidine doped ADP crystal recorded are shown in fig (4).



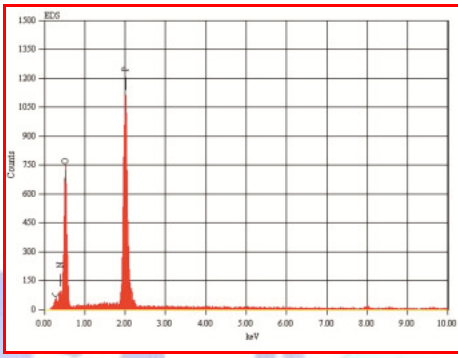
Element	KeV	Wt%	At%
N K	0.392	3.43	5.99
O K	0.525	23.98	36.66
P K	2.013	72.59	57.34
Matrix		Correction	ZAF

Fig. (a) EDAX of pure ADP



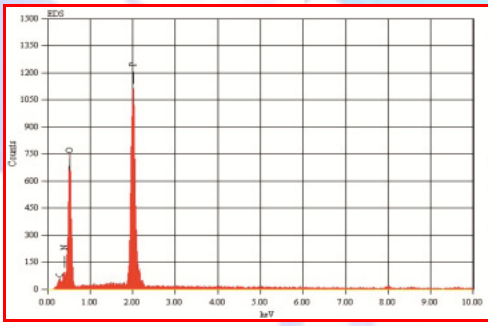
Element	(keV)	Wt %	At%
C K	0.277	0.39	0.19
N K	0.392	3.43	5.99
O K	0.525	23.98	36.66
P K	2.013	72.2	57.16
Matrix		Correction	ZAF

Fig. (b) EDAX of 0.4 mole %L-Histidine ADP



Element	(keV)	Wt %	At%
C K	0.277	0.78	0.47
N K	0.392	3.6	6.21
O K	0.525	25.39	38.37
P K	2.013	70.23	54.95
Matrix		Correction	ZAF

Fig. (c) EDAX of 0.8 mole %L-Histidine ADP



Element	(keV)	Wt %	At%
C K	0.277	1.16	0.61
N K	0.392	4.32	6.97
O K	0.525	34.17	48.21
P K	2.013	60.35	44.21
Matrix		Correction	ZAF

Fig. (d) EDAX of 1.2mole %L-Histidine ADP

3.4 Second harmonic generation (SHG) test:

The relative second harmonic generation ratio (SHG) was examined by Kurtz powder technique. A Q-switched Nd:YAG laser operating at the fundamental wavelength of 1064 nm, generating about 10.3 mJ and pulse width of 10 ns was used for the present experimental study. The input laser beam was passed through an IR reflector and then incident on the fine powder form of the ADP specimen, which was packed in a glass capillary tube. A photodiode detector integrated with oscilloscope assembly recorded the output energy. The second harmonic generation was confirmed by the green emission of wavelength 532 nm from the samples, when the laser beam was passed through L-Histidine doped ADP specimen. The second harmonic generation ratio was measured with

respect to ADP. From SHG measurement we found that the relative SHG ratio of L-Histidine doped ADP slightly changes that of standard ammonium dihydrogen phosphate. SHG of pure ADP is found to be 34mV. There is increase in SHG of ADP with the addition of L-Histidine which is due to the fact that L-Histidine has NH_4^+ and COO^- groups. The optically active amino group may get added in the ADP structure and increase its non-centro symmetry there by increasing its SHG ratio. SHG ratio results of various concentration of L-Histidine doped ADP are summarized in the table 2.

Table:-2 SHG of various concentration of L-Histidine doped ADP

Sr. No.	L-Histidine doped% in ADP	SHG in mV	SHG Efficiency(Ratio with pure ADP)
1	0.0	34mV	1.00
2	0.4	36mV	1.05
3	0.8	39mV	1.14
4	1.2	40mV	1.17

4. CONCLUSIONS:

Optical quality, colorless and pure and 0.4 mole%, 0.8 mole%,1.2 mole%, L- histidine doped ADP crystals were grown by *rotation crystallization technique*.

The powder X-ray diffraction studies of pure and L-Histidine doped ADP showed that crystal possess tetragonal structure having $I\bar{4}2d$ symmetry space group, with lattice parameter in good agreement with JCPDS data card no. 850815.

Even after doping crystal system remains unchanged. Intensity peaks of L-Histidine doped ADP crystal resembles with diffraction angle of pure ADP crystal with negligible small variation, while intensity variation observed.

The FT-IR spectrum confirms the presence of all functional group of ADP and L-Histidine. As concentration of dopant increases the peaks shifted towards higher wave number side. The EDAX analysis confirmed the presence of dopant (L-Histidine).The second harmonic generation test revealed that the increasing concentration of dopant (i.e. L-Histidine) enhances the SHG efficiency of ADP crystals.

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