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Growth and characterization of L-Threonine doped ADP Crystal.

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***Abstract*---**Synthesis and nonlinear optical characterization of L-Threonine Doped ADP have been studied. Pure ADP and Threonine doped ADP have been synthesized using low temperature crystal growth method.X-ray diffraction analysis show the presence of antase phase of crystalline ADP.The particle size was found to be 2x0.5x0.5cm (Lxbxh) which is in close agreement with the particle size calculated.The Four probe method study shows as temperature increases then the conductivity also increases with respect to the Pure ADP crystal. The Dielectric furnance is use to study the dielectric constsnt of the material and it shows the incriment in the Dielectric value with respect to the voltage The detail results are presented in this artical.

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1.*Introduction*

Amino acid family crystals are the famous organic materials playing an important role in the field of non-linear optics due to the fact that almost all the amino acids have chiral symmetry and crystallize in non-centrosymmetric space groups such as L-histidine nitrate, L-arginine triflurocetate. An amino acid consists of a free NH2 (amino) and a free COOH (carboxyl) group. Both are attached to the same carbon atom. Recently several new complexes incorporating the amino acids have been crystallized and their structural, optical and thermal properties have been investigatedAmmonium 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. Amino acids can be used as dopants and it was observed that there is enhancement in the material properties such as NLO and ferroelectric properties. We have grown pure and doped ADP crystals by solution growth technique and subjected to different types of characterizations Fourier Transform Infrared Spectroscopy (FTIR), UV-Vis Spectroscopy, X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDAX), Fluorescence Spectroscopy, Nonlinear optic measurement (NLO), Microhardness, Thermo gravimetric and differential thermal analysis (TGA-DTA).

Structure of ADP

Ammonium dihydrogen phosphate (ADP), also and better known as monoammonium phosphate in order not to confuse it with adenosine diphosphate (ADP), with formula $NH_4H_2PO_4$, is formed when a solution of phosphoric acid is added to ammonia until the solution is distinctly acidic. It crystallizes in tetragonal prisms.

2.*Material and method.*

2.1 *Material preparation*

2.1.1 *Pure ADP Crystal Solution.*

A pure ADP was grown by 4 mole solution of Adp with 100 Distilled water. The solution is then filtered with Filter paper and prepared solution is pour in beaker and Placed in a constant temperature water Bath.The temperature is made fixed on 36 ^{0}C. The Pure Adp Crystal then obtained within5-6 Days.

2.1.2 *L-threonine Doped ADP Crystal solution.*

L-threonine ADP was grown by 8gm with 100 saturated solution of ADP Distilled. The solution is then filtered with Filter paper and prepared solution is pour in beaker and Placed in a constant temperature water Bath.The pure ADP crystal was hung with thread insie the beaker.The temperature is made fixed on 36 ^{0}C. The Pure Adp Crystal then obtained within5-6 Days.

2.2Method of crystal growth

2.2.2 LOW TEMPERATURE SOLUTION GROWTH

The method of crystal growth from low temperature solutions is extremely popular in the production of crystals for many basic and technological applications. The method of growing crystals from solutions may be used for substances fairly soluble in a solvent and not reactive with it. Despite this limitation of usability, this method is in constant use because of the following reasons:

- The method and growth apparatus are relatively simple and
  cheap

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- Low growth temperature introduces small thermal stresses in
  the crystals obtained
- It leads to good quality crystals
- It lends itself to continuous operation
- The crystals obtained usually have well developed faces (growth habit), which enables to investigate crystal growth processes including in situ observations and capture of impurities
- In addition, growth from aqueous solution can be visually analyzed and has been extensively used for studying the growth parameters by means of photography..

EXPERIMENTAL SETUP

CRYSTAL GROWTH APPARATUS

The schematic diagram of the growth apparatus used for the low temperature solution growth technique. It consists of a large tank (constant temperature bath) heated at the base using an infrared lamp. The IR lamps are energized through a relay switch. The control is effected by a jumo contact thermometer coupled to an on-off controller, which has a controlling accuracy of ± 0.01°C. The temperature of the constant temperature bath is converted into a signal by a suitable sensor. The controller is contacted with an on-off switch. It gets activated when the process variable (bath temperature) crosses the set point. There are only two stable states in an on-off controller. "On" state is enabled when the temperature is below the set point. As the desired set point is arrived, the controller goes to the "off" state. To get change in the state, the temperature must cross the set point. Set point variations, which occur due to electrical noise interference and process disturbances, seriously affect the practical applications of the controller. Contrary to this, a proportional controller continuously manipulates the process variable so that the heat input is in balance with the heat demand. The controller consists of a power supply, processor, booster and proportional controllers. In the present investigation, the growth instrument was modified by replacing the infrared lamp using a programmable controller heater. The constant temperature bath used in the present study is shown in Figure

.

Fig: Constant temperature Water bath apparatus

3.Result and discusion

3.1 Characterization

3.1.1 XRD Study:

MiniFlex 300/600 machine X-ray software was used for analysis of powder XRD patterns. Well defined Bragg peaks are obtained at specific 2θ angles indicating that crystals are ordered. The 'd' spacing and hkl values for prominent peaks in the spectrum were identified and compared with ICDD (International Centre for Diffraction Data). Using tetragonal crystallographic equation, lattice parameter values are calculated and listed in Table This suggests that the crystals retain almost the single phase structure and exhibit very slight variation in the unit cell parameters on doping of L-histidine.

Measurement conditions

X-Ray	40 kV , 15 mA	Scan speed / Duration time	10.0000 deg/min
Goniometer	MiniFlex 300/600	Step width	0.0200 deg
Attachment	Standard	Scan axis	Theta/2-Theta
Filter	None	Scan range	20.0000 - 80.0000 deg
CBO selection slit	-	Incident slit	1.250deg
Diffrected beam mono.	None	Length limiting slit	10.0mm
Detector	SC-70	Receiving slit #1	13.0mm
Scan mode	CONTINUOUS	Receiving slit #2	13.0mm

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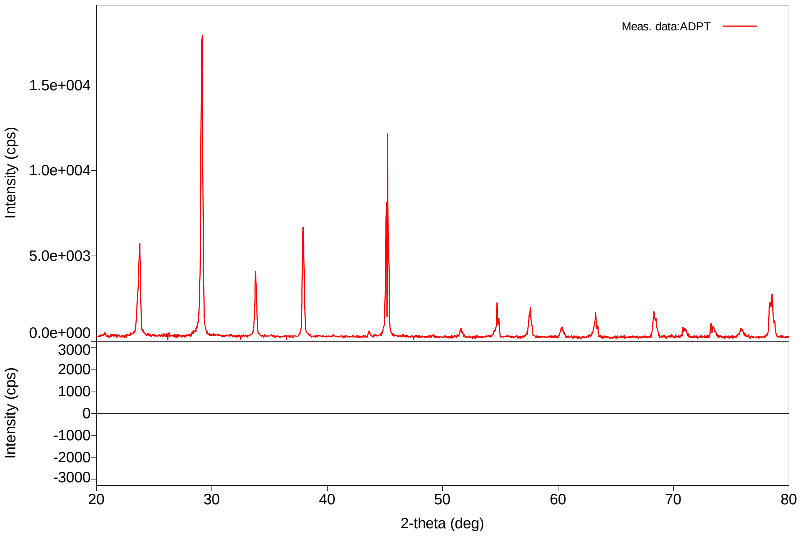
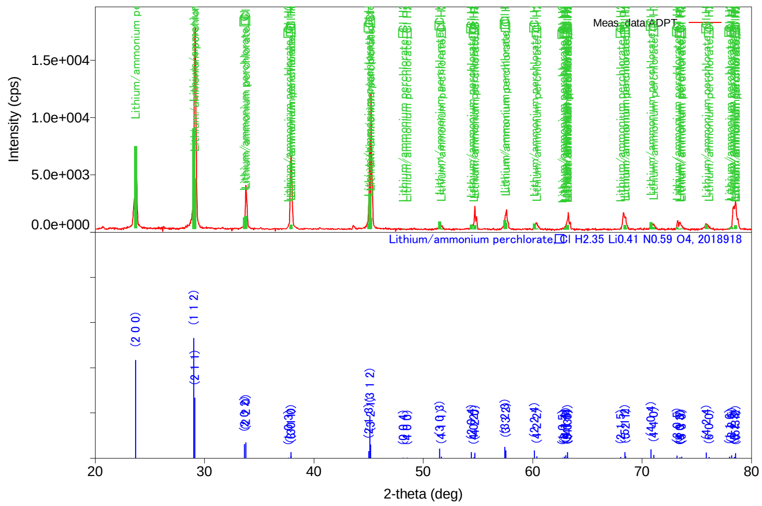


Peak to Peak Analysis of ideal and doped ADP Crysl using XRD is shown in fig below.



Peak list

No.	2-theta(deg)	d(ang.)	Height(cps)	FWHM(deg)	Int. I(cps deg)	Int. W(deg)	Size(ang.)
1	23.772(2)	3.7399(3)	3481(170)	0.249(6)	1058(18)	0.30(2)	341(8)
2	26.26(6)	3.391(8)	168(37)	0.07(6)	13(10)	0.08(8)	1271(1193)
3	29.150(4)	3.0610(4)	13566(336)	0.162(3)	2916(21)	0.215(7)	530(9)
4	33.797(3)	2.6500(2)	3283(165)	0.108(4)	553(8)	0.168(11)	802(32)
5	37.913(4)	2.3712(2)	5641(217)	0.129(4)	950(11)	0.168(8)	679(20)
6	43.614(13)	2.0736(6)	326(52)	0.106(14)	41(4)	0.13(3)	845(112)
7	45.1659(18)	2.00587(8)	7471(250)	0.100(7)	963(34)	0.129(9)	903(61)
8	45.2346(14)	2.00298(6)	8251(262)	0.047(4)	434(37)	0.053(6)	1919(164)
9	45.308(16)	1.9999(7)	3218(164)	0.061(8)	229(32)	0.071(13)	1481(198)
10	45.57(3)	1.9888(10)	254(46)	0.10(5)	32(11)	0.13(6)	867(448)
11	51.62(2)	1.7693(7)	383(57)	0.17(3)	93(8)	0.24(6)	540(83)
12	54.741(9)	1.6755(2)	1787(122)	0.115(10)	318(12)	0.178(19)	813(74)
13	57.637(10)	1.5980(2)	1257(102)	0.228(9)	323(10)	0.26(3)	416(16)
14	60.35(3)	1.5324(6)	440(61)	0.26(3)	137(7)	0.31(6)	367(37)

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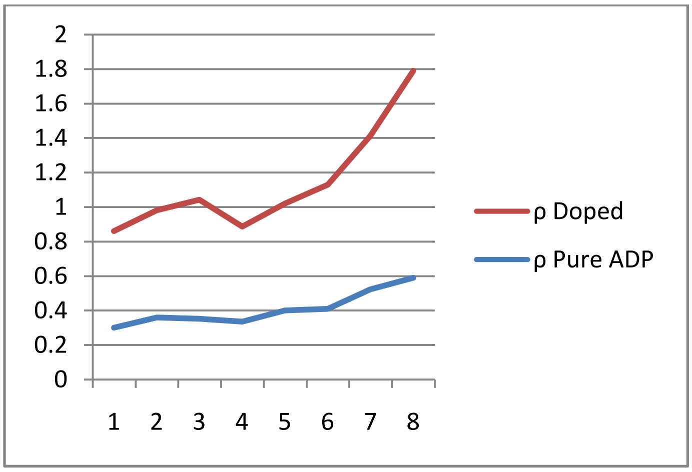
15	63.278(4)	1.46845(8)	1413(109)	0.111(8)	297(8)	0.21(2)	877(67)
16	68.316(9)	1.37192(17)	1357(106)	0.212(10)	344(10)	0.25(3)	473(23)
17	70.878(10)	1.32847(16)	437(60)	0.29(3)	145(10)	0.33(7)	352(35)
18	73.278(10)	1.29078(16)	809(82)	0.144(15)	195(9)	0.24(4)	719(74)
19	78.352(5)	1.21940(7)	2016(130)	0.289(8)	649(13)	0.32(3)	370(10)

Table: Shows the values of Peak to Peak XRD

3.1.2 Conductivity:

The conductivity of original sample and Histidine doped ADP crystal was studied by using four probe method. The following graph shows the variation of pure and doped ADP. Table 2 shows the experimental readings of the crystal.



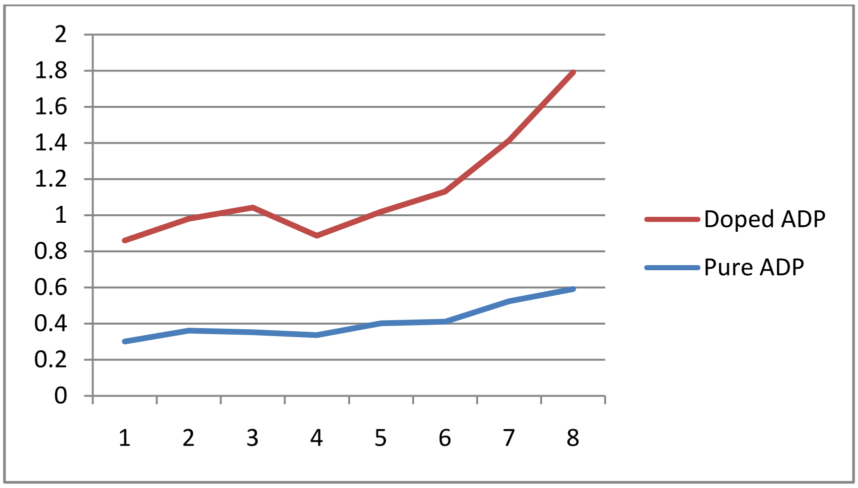
Graph 1: Experimental variation of conductivity of pure Vs Doped ADP.

Temperature (^{0}C)	Voltage (mv)	ρ Pure ADP (Ωm)	ρ doped (Ωm)
30	9.6	6.2	7.21
35	11.5	8.4	9.6
40	18.8	12.36	10.3
45	32.5	18.65	16.2
50	48.6	28.33	29.6
55	57	35.6	32.3
60	75.98	49.3	53.9
65	85.85	63.68	62.8

Table 1: For experimental Reading of conductivity of pure Vs Doped ADP crystal.

3.1.3 Dielectric Constant :

The Dielectric constant of pure ADP and Histidine doped ADP crystal was studied by using Dielectric furnance. The following graph in fig 3 shows the variation of pure and doped ADP. Table 3 shows the experimental readings of the crystal.

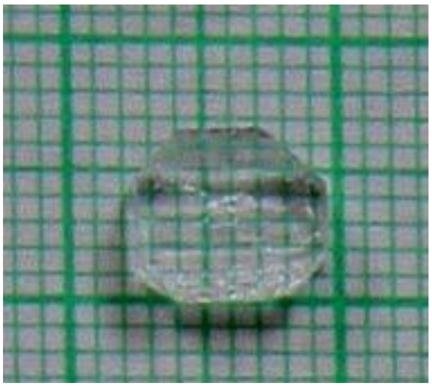


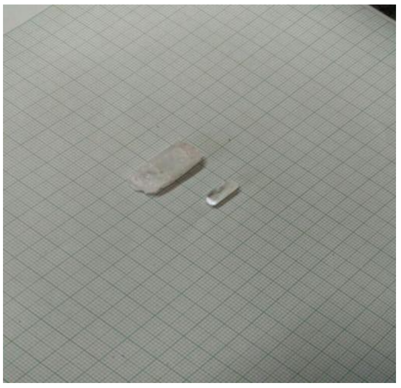
Graph 2: Experimental variation of Dielectric constant of pure Vs Doped ADP.

Voltage	Temperature	Dielectric connstant Pure ADP	Dielectric constant of Doped ADP
0	36	0.3	0.56
20	36	0.36	0.62
40	36	0.352	0.69
60	36	0.336	0.55
70-	36	0.4	0.62
80	36	0.41	0.72

Table 2: For experimental Reading of Dielectric constant of pure Vs Doped ADP .

3.1.4 Obtained crystals





Photograph of Grown and Pure ADP crystal.

The pure and 6gm Threonine doped ADP crystals are also grown by the same process.The resultant sample of Pure and Threonine Doped ADP was compared using various characterization processes.The XRD study compares the various peaks of pure and Doped ADP which shows the great resemblense in there properties.Also the Four probe method study shows as temperature increases then the conductivity also increases with respect to the Pure ADP crystal. The Dielectric furnance is use to study the dielectric constsnt of the material and it shows the incriment in the Dielectric value with respect to the voltage.From this we conclude the material obtained by doping l-histidine to the ADP improves its chemical ,optical, thermal,conductivtity etc properties of the Pure ADP. Amonium dihydrogen phosphate (ADP) is a model system for nonlinear optical device application, it continues to be an interesting material both academically and industrially and is extensively studied from various aspects. Its excellent qualities such as high nonlinear conversion efficiency, wide optical transmission range with low cutoff wavelength and high laser damage threshold has drawn the attention of severalcrystal growers. Many research works have been resulted in the modification

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of ADP properties and growth rates by varying the growth conditions and by adding suitable impurities.

4.References

[1] Ram Kripal, Santwana Shukla, Prashant Dwivedi, Physica B 407,656(2012).
[2] P. Rajesh, K. Boopathi, P. Ramasamy, J. Cryst. Growth 318, 751 (2011).
[3] K. Srinivasan, A. Cantoni, G. Bocelli, Cryst. Res. Technol. 45, 737(2010).
[4] R. Ananda Kumari, Ind. J. Pure & Appl. Phys. 47, 369(2009).
[5] Y. Asakuma, Q. Li, H. M. Ang, M. O. Tade, K. Maeda, K. Fukui, Appl. Surf. Sci. 254, 4524(2008).
[6] Xiue Ren, Dongli Xu, DongfengXue, J. Cryst. Growth 310, 2005(2008).
[7] S. S. Hussaini, N. R. Dhumane,V. G. Dongre, P. Ghughare,M. D. Shirsat, J. Optoelectron. Adv. Mater. - Rapid Commun. 1, 707(2007).
[8] S. S. Hussaini, N. R. Dhumane, V. G. Dongre,M. D. Shirsat, J. Optoelectron and Adv. Mater Rapid Commun. 2, 470(2008).
[9] N. R. Dhumane, S. S. Hussaini, V. V. Navarkhele,M. D. Shirsat, Cryst. Res. Technol. 41, 897(2006).
[10] M. D. Shirsat, S. S. Hussaini, N. R. Dhumane,V. G. Dongre, Cryst. Res. Technol. 43, 756(2008).
[11] N. R. Dhumane,S. S. Hussaini ,V. G. Dongre ,M. D. Shirsat, Opt. Mater. 31, 328(2008).
[12] J. D. Bernal, Z. Kristallogr, “The Crystal Structure of the Natural Amino Acids and Related Compounds”, 78, 363(1931). 93
[13] H. J. Simpson, R. E. Marsh, Acta Crystallographica 20, 550 (1966).
[14] R. Destro, R. E. Marsh, R. Bianchi, Journal of Physical Chemistry 92, 966(1988).
[15] L. Misoguti,A. T. Varela,F. D. Nunes,V. S. Bagnato,F. E. A. Melo,J. MendesFilho, S. C. Zilio, Optical Materials.