

Growth and characterization of pure and l-ornithine monohydrochloride doped ADP crystals

K.V. Vadhel^{1*}, V.J. Pandya², M.J. Joshi³, H.O. Jethva⁴

^{1,2,3,4}Department of Physics, Saurashtra University, Rajkot, Gujarat, India

*Corresponding Author: kiritvadhel1@gmail.com

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Abstract— In the present study, growth and various characterizations of pure and different wt% l-ornithine monohydrochloride (LOMHCl) doped ammonium dihydrogen phosphate (ADP) crystals are reported. The slow solvent evaporation method at room temperature is used to grow these crystals. After nearly 25 days, good quality, transparent and rectangular crystals are obtained. The grown crystals are analyzed by CHN, EDAX, FTIR, thermal and PL characterizations. EDAX and CHN analysis confirms the presence of dopant atoms. The EDAX analysis shows gradual increase in the weight% of carbon (C) and chlorine (Cl) in LOMHCl doped ADP crystals. The presence of all constitutive functional groups of ADP in pure and LOMHCl doped ADP crystals is confirmed by FT-IR spectra. The doping of LOMHCl in ADP gives rise to small shift in some of the characteristic vibrational frequencies. The thermo-gram of pure and different wt% LOMHCl doped ADP suggests slight increase in thermal stability of doped ADP crystals as well as shows reduction in the weight loss % of pure ADP at the end of first and second decomposition stages. It is observed from the thermal analysis that the presence of dopant LOMHCl prevents pure ADP to decompose into gaseous products. The PL study shows L-defect and D-defect. The doping of LOMHCl shows reduction in PL emission spectra and increase in Stokes shift value.

Keywords— ADP, EDAX, FTIR, thermal analysis, PL

I. INTRODUCTION

Technologically, ADP is a very important crystal and useful as 2nd, 3rd and 4th harmonic generator for Nd:YAG laser [1,2]. The small amount of metallic impurities as well as amino acids can affect the growth rate, shape and transparency of grown crystals. Recently, Joshi et al. [3,4,5] have studied the effect of doping of l-phenylalanine and picric acid on the PL property and FTIR spectroscopy of ADP, l-serine on the EDAX, FTIR spectroscopy and PL property of ADP [6,7], l-threonine on FTIR spectroscopy, CHN and PL property of ADP [8], l-methionine on PL property of ADP [9] and bivalent metal ion strontium on FTIR, EDAX and thermal property of ADP [10]. The effect of various dopants on the PL property of pure ADP is such that PL emission peak intensity either increase or decrease for a particular concentration of dopant. The increase in peak intensity suggests that the sample's quality has improved. Furthermore, the difference in wavelength (energy) between the spectrum maximum of the absorption and emission spectra in the PL emission and absorption spectra gives Stokes shift [11]. For a given dopant concentration, more or less Stokes shift might occur, with less Stokes shift indicating less lattice distortion. Such behavior indicates that the grown crystal quality is improved. From thermal property, based on the weight loss in the doped crystals compared with the pure ADP, it may

be determined if the doped crystals are used for NLO device manufacturing or not. Hence, an investigation on doped ADP crystals is significant in order to enhance the grown crystals quality and use them in the production of NLO devices. With this aim, the present authors decide to study the effect of l-ornithine monohydrochloride doping by varying its weight% on the EDAX spectroscopy, FTIR spectroscopy, thermal property and PL property of pure ADP crystals.

II. EXPERIMENTAL TECHNIQUE

At room temperature, the slow solvent evaporation method is utilized to grow pure and LOMHCl doped ADP crystals. Prepare a pure ADP's saturated solution in 0.4L distilled water, and then stir it to ensure homogeneity. The solution is filtered by filter paper and divided into four 100 mL beakers. One beaker is used for grown pure ADP crystal, while, 0.2gm, 0.4gm, and 0.6gm l-ornithine monohydrochloride dissolved into the remaining 3 beakers (ADP solution) with proper stirring, for grow LOMHCl doped ADP crystals. For controlled evaporation, all beakers are stored in a dust-free, disturbance-free environment and coated by filter paper. Good quality, translucent, and rectangular crystals are carefully extracted from the beakers after roughly 25 days. Pure and LOMHCl doped ADP crystals are shown in Figure 1a-d.

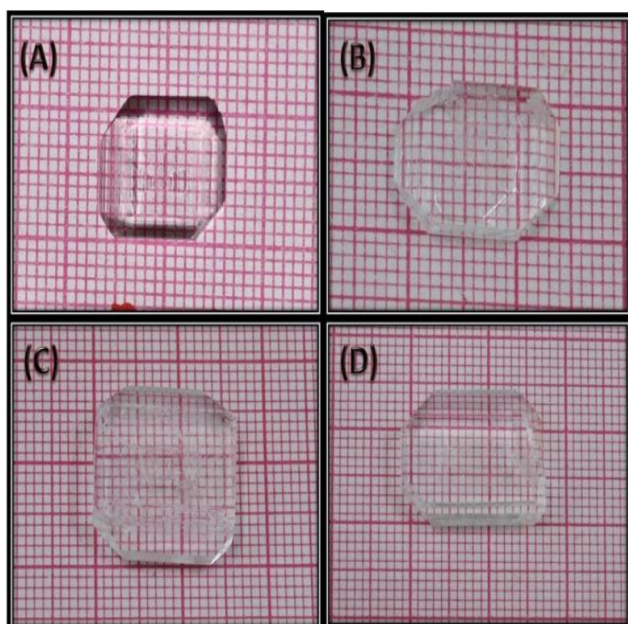


Figure 1. (A). Grown Pure ADP crystal, Figure 1 (B-D). Grown (0.2, 0.4 and 0.6)wt% LOMHCl doped ADP crystals

III. RESULTS AND DISCUSSION

CHN analysis:

The presence elements of dopant are find in used crystals, CHN analysis and EDAX analysis is carried out by using CHNS/O Analyzer, model unicube, make Elementar and Philips XI-30, respectively. Table 1 shows the results of CHN study of pure and different wt% LOMHCl doped ADP crystals.

Table 1. CHN analysis data of pure and different wt% LOMHCl doped ADP crystals

Name of Samples	C%	H%	N%
1. Pure ADP	-	4.59	12.06
2. 0.2wt% LOMHCl doped ADP	0.14	5.02	12.10
3. 0.4wt% LOMHCl doped ADP	0.23	5.08	12.14
4. 0.6wt% LOMHCl doped ADP	0.53	5.11	12.16

The chemical formula of ammonium dihydrogen phosphate (ADP) is $(\text{NH}_4)\text{H}_2\text{PO}_4$. The formula suggests that ADP contains both nitrogen (N) and hydrogen (H). Hence, the presence of both nitrogen (N) and hydrogen (H) is revealed by a CHN analysis of pure ADP. The chemical formula of dopant l-ornithine monohydrochloride (LOMHCl) is $\text{C}_5\text{H}_{13}\text{ClN}_2\text{O}_2$. As a result, when LOMHCl is doped into pure ADP, the CHN analysis reveals the presence of carbon (C), confirming the existence of a dopant in the ADP crystal. Furthermore, as the wt% of LOMHCl in ADP increases, the wt % of carbon (C) in LOMHCl doped ADP crystal increases. This indicates that LOMHCl is successfully doped in ADP crystals. The amount of percentage carbon (C) in ADP crystals increases as the doping concentration of wt % of LOMHCl increases from 0.2wt % to 0.6wt %, but the concentration of (C) in ADP crystals cannot be obtained exactly due to the addition of LOMHCl in very small amounts. Further, the difference in

solubility as well as crystal growth rate of ADP and LOMHCl may result into the limited quantity of LOMHCl entering in ADP's lattice sites.

EDAX Spectroscopy Study:

The EDAX analysis is done to determine the elemental composition of used crystals. Table 2 shows the EDAX result.

According to table (3.2), pure ADP contains phosphorous (P), oxygen (O), and nitrogen (N), whereas LOMHCl doped ADP crystal has carbon (C), chlorine (Cl), along with presence of elements of pure ADP.

Table 2. EDAX Result

Sample Name	C Wt%	N Wt%	O Wt%	P Wt%	Cl Wt%
Pure ADP	-	12.12	52.51	35.37	-
0.2 Wt% LOMHCl doped ADP	2.12	11.77	56.90	29.18	0.03
0.4 Wt% LOMHCl doped ADP	2.53	10.39	53.86	32.48	0.06
0.6 Wt% LOMHCl doped ADP	3.20	11.01	53.91	32.36	0.19

In addition, as the doping concentration of LOMHCl in ADP increases, the wt % of carbon (C) and chlorine (Cl) increases. This indicates that the dopant LOMHCl has been doped into the crystalline lattice of pure ADP, indicating that LOMHCl doping in ADP has been successful.

FTIR Spectroscopy Study:

Table 3 lists the observed absorption frequencies and their assignments in regard to their characteristic vibrational modes, while Figure 2 shows the FTIR spectra of pure and different wt% LOMHCl doped ADP.

The FTIR spectra of pure as well as LOMHCl doped ADP possess the characteristic vibrations of ammonium ion as well as phosphate ion. The absorption bands observed at 3221 cm^{-1} and 3023 cm^{-1} in pure ADP due to O-H stretching of P-OH [12]. When LOMHCl is doped, the same are observed to shift slightly towards higher wavenumber and appeared at 3222 cm^{-1} and 3025 cm^{-1} for 0.2wt%, 3223 cm^{-1} and 3028 cm^{-1} for 0.4wt% and 3223 cm^{-1} and 3027 cm^{-1} for 0.6wt% LOMHCl doped ADP. The absorption band appeared at 2848 cm^{-1} for pure ADP because of N-H stretching vibration of ammonium ion [8]. When LOMHCl is doped the same is shifted towards higher wavenumber and appeared at 2862 cm^{-1} for all the three LOMHCl doped ADP crystals. The band at 2322 cm^{-1} for all the samples is assigned to hydrogen bond [13].

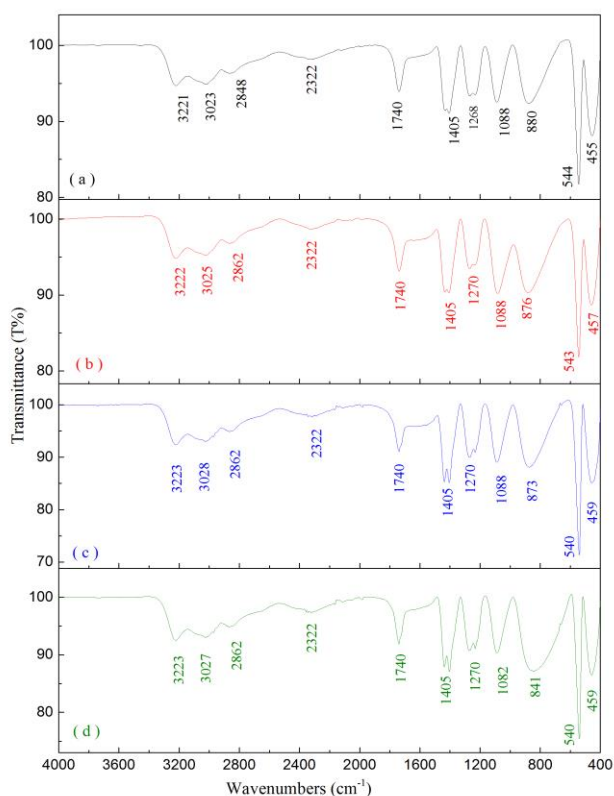


Figure 2. FT-IR spectra of (a) Pure ADP (b-d) (0.2, 0.4 and 0.6) wt% LOMHCl doped ADP

The broadness of the band is due to the hydrogen bonding interaction with adjacent molecules. The absorption observed within $1710\text{--}1745\text{ cm}^{-1}$, i.e., All of the samples can be attributed to a stretching vibration of $\text{P}=\text{O}$ at wavenumber 1740 cm^{-1} . The N-H bending vibrations of the ammonium ion are responsible for the narrow absorption band within group frequency $1430\text{--}1390\text{ cm}^{-1}$ [12, 14]. The splitting of this narrow band is observed as the concentration of dopant was increased. The absorption bands near 1270 cm^{-1} and 1088 cm^{-1} in pure as well as LOMHCl doped samples is due to the asymmetrical stretching vibrations P-O of phosphate ion [8, 12,15,16]. The symmetric stretching vibrations P-O of the phosphate ion are responsible for the absorption band observed at 880 cm^{-1} for pure ADP [15]. When LOMHCl is doped at different wt %, the wavenumber shifts to lower values, appearing 876 cm^{-1} at 0.2wt %, 873 cm^{-1} at 0.4wt %, and 841 cm^{-1} at 0.6wt % LOMHCl doped ADP crystals. Out of plane bending vibration of phosphate is appeared at 544 cm^{-1} [15,17] in pure ADP and the same is observed to shift slightly towards lower wavenumber and appeared at 543 cm^{-1} in 0.2wt% and at 540 cm^{-1} in 0.4wt% and 0.6wt% LOMHCl doped ADP. A symmetric bending mode of phosphate is observed at 455 cm^{-1} [15] in pure ADP and the same is observed to shift towards higher wavenumber and appeared at 457 cm^{-1} in 0.2wt% and at 459 cm^{-1} in 0.4wt% and 0.6wt% LOMHCl doped ADP. According to table 3, it is revealed that the doping of LOMHCl in different wt% in the ADP crystal matrix give rise to small shift in some of the characteristic vibrational frequencies.

Table 3. Assignments of functional groups

Assignments	Wave numbers (cm^{-1})			
	Pure ADP Sample 1	0.2wt% LOMHCl doped ADP Sample 2	0.4wt% LOMHCl doped ADP Sample 3	0.6wt% LOMHCl doped ADP Sample 4
O – H stretching of P-OH	3221, 3023	3222, 3025	3223, 3028	3223, 3027
N – H stretching of ammonium	2848	2862	2862	2862
Band due to hydrogen bond	2322	2322	2322	2322
P = O stretching	1740	1740	1740	1740
N – H bending of ammonium	1405	1405	1405	1405
P – O asymmetric stretching	1268, 1088	1270, 1088	1270, 1088	1270, 1082
P – O symmetric stretching	880	876	873	841
P – O out of plane bending	544	543	540	540
P – O symmetric bending	455	457	459	459

Thermal Analysis:

The analysis of thermogravimetric and heat value curves provide useful information regarding phase transition, water of crystallization associated with the crystals because of use of water as a solvent for crystallization, different stages of decomposition, melting point and thermal stability of the crystal system. The analysis in this work is done in an air atmosphere at temperatures ranging from ambient temperature to 900°C .

The TGA/DSC plots of pure and different wt% LOMHCl doped ADP crystals are shown in Figure 3, and the TGA result is listed in Table 4. The TG curves show that pure ADP and 0.2wt% LOMHCl doped ADP remain stable up to temperature 190°C , while 0.4wt% and 0.6wt% LOMHCl doped ADP remain stable up to 200°C with negligible amount of weight loss. This implies there's almost no chance of water getting into the crystal lattice that was utilized as a solvent throughout crystallization process. The increased wt% of dopant LOMHCl, i.e., 0.4wt% and 0.6wt% increase the thermal stability of pure ADP from 190°C to 200°C .

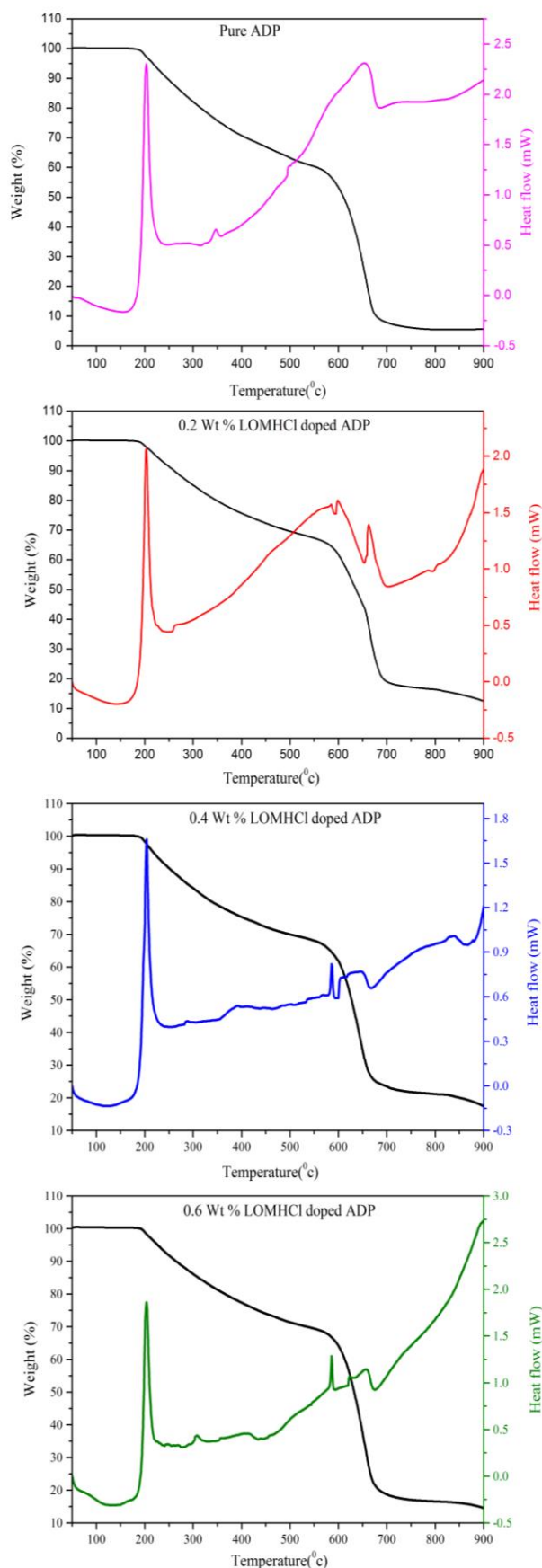


Figure 3. TGA/DSC curves of pure and 0.2wt%, 0.4wt% and 0.6wt% LOMHCl doped ADP crystal

Table 4. Thermogram result

Sample	Temperature range (°C)	Remaining weight (%)
Pure ADP	From RT to 190	100
	From 190 to 580	58
	From 580 to 690	9
	From 690 to 900	9
0.2 wt% LOMHCl doped ADP	From RT to 190	100
	From 190 to 580	65
	From 580 to 690	21
	From 690 to 900	21
0.4 wt% LOMHCl doped ADP	From RT to 200	100
	From 200 to 580	65
	From 580 to 680	21
	From 680 to 900	21
0.6 wt% LOMHCl doped ADP	From RT to 200	100
	From 200 to 580	68
	From 580 to 680	25
	From 680 to 900	21

After this stable region, pure ADP and 0.2wt% LOMHCl doped ADP show gradual weight loss of 42% and 35%, respectively within temperature range 190°C to 580°C and 0.4wt% and 0.6wt% LOMHCl doped ADP show gradual weight loss of 34.5% and 32%, respectively within temperature range 200°C to 580°C. The presence of dopant LOMHCl decreases weight loss at the end of the first decomposition stage of ADP, according to this result.

Within temperature range 580 to 690°C, pure ADP and 0.2wt% LOMHCl doped ADP show rapid decomposition following weight loss of 91% and 79%, respectively. While, within temperature range 580 to 680°C, 0.4wt% and 0.6wt% LOMHCl doped ADP show rapid decomposition and weight loss of 79% and 75%, respectively. After this stage, Up to the temperature's highest limit, the all samples stay constant, i.e., 900°C. The observed that the addition of the dopant LOMHCl reduces the weight loss of pure ADP at the end of the second decomposition stage significantly.

The TG curves of pure ADP show two major weight loss stages or two stages of decomposition, which corresponds with the studies by Pardo et. al. [18]. When LOMHCl is doped in different weight%, the doped ADP also retains the same decomposition nature. The weight loss observed during the decomposition process can be related to the decomposition of ADP into orthophosphoric acid (H₃PO₄) with the liberation of ammonia (NH₃), then conversion of orthophosphoric acid (H₃PO₄) into pyrophosphoric acid

(H₄P₂O₇) and then to metaphosphoric acid (HPO₃) as suggested by Abdel-Kader et. al. [19] and supported by Pardo et. al. [18]. The TG thermogram of pure ADP also shows that the remaining weight is only 9% at 690°C. This indicates that the majority of the pure sample decomposes into gaseous products [18]. When LOMHCl is doped, the weight loss reduces and remaining weight increases from 9% to 21% for 0.2wt% and 0.4wt% LOMHCl doped ADP and 25% for 0.6wt% LOMHCl doped ADP. This indicates that the presence of dopant LOMHCl may be responsible for the prevention of pure ADP into decomposition into gaseous products.

The DSC curve of pure ADP clearly shows an endothermic peak having onset and peak temperatures of 190.99°C and 202.93°C, respectively. This result is consistent with that reported by Chen et.al. [20], but does not coincide with the report of Pardo et. al. [18]. The variation in the results can be attributable to the two tests using various heat rates and weights of sample. When LOMHCl doped in different weight%, the onset and peak temperatures are observed to raise nearly 1°C. The sharp endothermic peak indicates changes in crystallinity [21]. The good sharpness suggests of the used crystals good crystallinity. Further, as the weight % of dopant LOMHCl is increased, the area of the peak reduces which indicates increase in the sharpness of the peak and hence the crystallinity of LOMHCl doped ADP crystals.

At temperatures of 654°C and 662°C, respectively, the DSC curves of pure and 0.2wt % LOMHCl doped ADP reveal another endothermic peak. Whereas DSC curves of 0.4wt% and 0.6wt% LOMHCl doped ADP show exothermic peak at temperature 666°C and 674°C, respectively. Endothermic curves are generated by physical changes, whereas exothermic curves are generated by chemical reactions. The study of curves enables the identification of chemical or physical changes whether or not they are accompanied by a weight change[21]. Both endothermic and exothermic peaks are within the temperature region of second decomposition process. The presence of endothermic peak in pure and 0.2wt% LOMHCl doped ADP and exothermic peak in 0.4wt% and 0.6wt% LOMHCl doped ADP during the stage of second decomposition process can be attributed to the physical changes in pure and 0.2wt% LOMHCl doped ADP and chemical reactions in 0.4wt% and 0.6wt% LOMHCl doped ADP.

Photoluminescence (PL) study:

The analysis of PL emission at a certain wavelength is a well-established method for examining the quality of used crystals, observational determination of impurity incorporation, and optical processes such as electron transition recombination [22].

Figures 4 and 5 show the PL emission and absorption spectra of pure and different wt% LOMHCl doped ADP crystals, respectively. The emission spectra are measured in the range of 350 to 600 nm wavelength, while the absorption spectra are measured in the range of 220 to 300

nm wavelength. The emission wavelength of the PL is 280 nm, whereas the excitation wavelength is 254 nm.

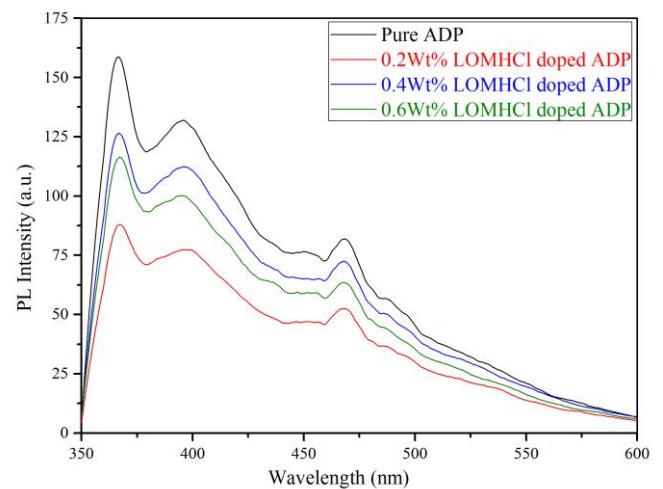


Figure 4. PL spectra of emission

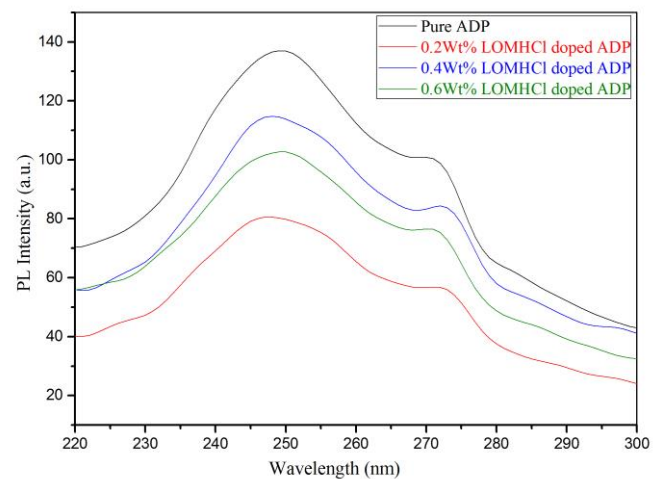


Figure 5. PL spectra of absorption

Figure 4 shows that the PL emission spectra of grown crystals exhibit two emission peaks at distinct wavelengths, as mentioned in table 5. The PL emission spectra of LOMHCl doped crystals have a lower intensity, indicates the dopant is incorporation with pure ADP. Obtained a result is consistent with that reported by Joshi et. al. [8]. The emission peaks around (366-367 nm) and (468-469 nm) are attributed to electron recombination on the trapped hole centre due to hydrogen vacancy (L-defect) and hole recombination at the electron centre due to double occupied hydrogen vacancy (D-defect). L-defect, i.e., unoccupied hydrogen vacancy and D-defect, i.e., double occupied hydrogen vacancy are the simplest orientational defects in the crystal of pure ADP [7, 23]. Doped ADP has a lower intensity than pure ADP, which can indicate that a destabilization of the orientational L-defect in neighboring impurity atoms for charge compensation. There is an association between the PL emission intensity and the electrical conductivity of material [8], The ac electrical conductivity of LOMHCl doped ADP crystals would be higher than pure ADP crystals due to a decrease in PL emission intensity. As a result, the PL emission study

exhibits that amino acid doping (in this case, LOMHCl) causes a defect in the ADP structure [24].

Table 5. PL emission peaks for pure and LOMHCl doped ADP crystals

Sample	Emission wavelength (nm)	
	Pure ADP	366.44
0.2 wt% LOMHCl doped ADP	367.02	468.72
0.4 wt% LOMHCl doped ADP	366.96	468.04
0.6 wt% LOMHCl doped ADP	366.99	468.07

The PL absorption spectra of grown crystals are shown in Figure 5. The reduction in the peak intensity is observed when LOMHCl is doped in different weight%. The obtained result for the absorption is also consistent with that reported by Joshi et. al. [8]. Stokes shift is caused by the difference in wavelength (energy) between the maximum of the emission and absorption spectra [11]. The Stokes shift occurs when the emitted photon has less energy than the absorbed photon, while the anti-Stokes shift occurs when the emitted photon has more energy than the absorbed photon [25]. Table 6 shows Stokes-shift value of pure and LOMHCl doped ADP crystals.

Table 6. Stokes-shift variation of grown crystals

Sample	Absorption Energy (eV)	Emission Energy (eV)	Stokes shift (eV)
Pure ADP	4.9628	3.3858	1.5770
0.2 wt% LOMHCl doped ADP	4.9827	3.3804	1.6023
0.4 wt% LOMHCl doped ADP	5.0028	3.3810	1.6218
0.6 wt% LOMHCl doped ADP	5.0435	3.3807	1.6628

The Stokes shift increases as the wt% of dopant LOMHCl is increased, as seen in table 6. A dipole is formed by the positive and negative charge of the dopant, which is surrounded by the ADP lattice. Under the effect of electromagnetic radiation, the dopant enters into the excited state and its dipole moment changes. The ADP lattice cannot adjust quickly to these changes. The ADP lattice becomes increasingly difficult to realign as the doping concentration increases. When a result, increase in Stokes shift is observed as weight% of dopant LOMHCl is increased. The obtained result is consistent with what Joshi et.al. reported. [8].

IV. CONCLUSION AND FUTURE SCOPE

Using a slow solvent evaporation technique at room temperature, pure and different wt% LOMHCl doped ADP crystals are grown. The CHN and EDAX analysis reveals that LOMHCl is successfully doped. The various functional groups of pure ADP and their changes are studied by FTIR spectroscopy. The thermogram of pure and different wt%

LOMHCl doped ADP shows the thermal stability of doped ADP crystals is slightly increases as well as reduction in the weight loss % of pure ADP at the end of first and second decomposition stages. The presence of dopant LOMHCl also prevents the decomposition pure ADP into gaseous products. The PL study shows L-defect and D-defect. The doping of LOMHCl shows reduction in PL emission spectra and increase in Stokes shift value.

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