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ORIGINAL ARTICLE

Crystal growth, spectroscopic, second and third order nonlinear optical spectroscopic studies of L-phenylalanine doped ammonium dihydrogen phosphate single crystals

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Abstract The frequency doubling and tripling can be reached for Nd:YAG laser beam using the single crystal of ammonium dihydrogen phosphate (ADP). The growth and characterization of pure and L-phenylalanine doped ADP crystals are considered in the present contribution. The transparent good quality single crystals are grown using low temperature solution growth technique. The functional group alteration is studied using FT-IR spectroscopy. The larger improvement is observed in linear and nonlinear optical properties of ADP crystals on doping by L-phenylalanine. The reduction in charge carrier density is observed in photoconductivity spectra of all the grown crystals.

1. Introduction

The violation of superposition theory of light waves falls under the subject of the nonlinear optics. To study the behaviour of coherent light, the excellent nonlinear optical crystal like ammonium dihydrogen phosphate has been used (Nikogasyan, 2005). To modify the properties of such crystals, the amino acids are considered as a promising item. By nature, the L-phenylalanine it behaves as an electron donor amino acid (Parikh et al., 2007), hence it is quite beneficial and easy to dope L-phenylalanine into the ADP crystal. In the present contribution, the influence of L-phenylalanine on different properties of ADP crystals, including spectroscopic, linear and nonlinear optical and electrical characteristics, is considered.

1.1. Experimental technique

The crystal growth of pure and L-phenylalanine doped ADP crystals are carried out using the slow evaporation, a variant of low temperature solution growth techniques. The detailed description of this experimental technique is given elsewhere (Parikh et al., 2007). Here,

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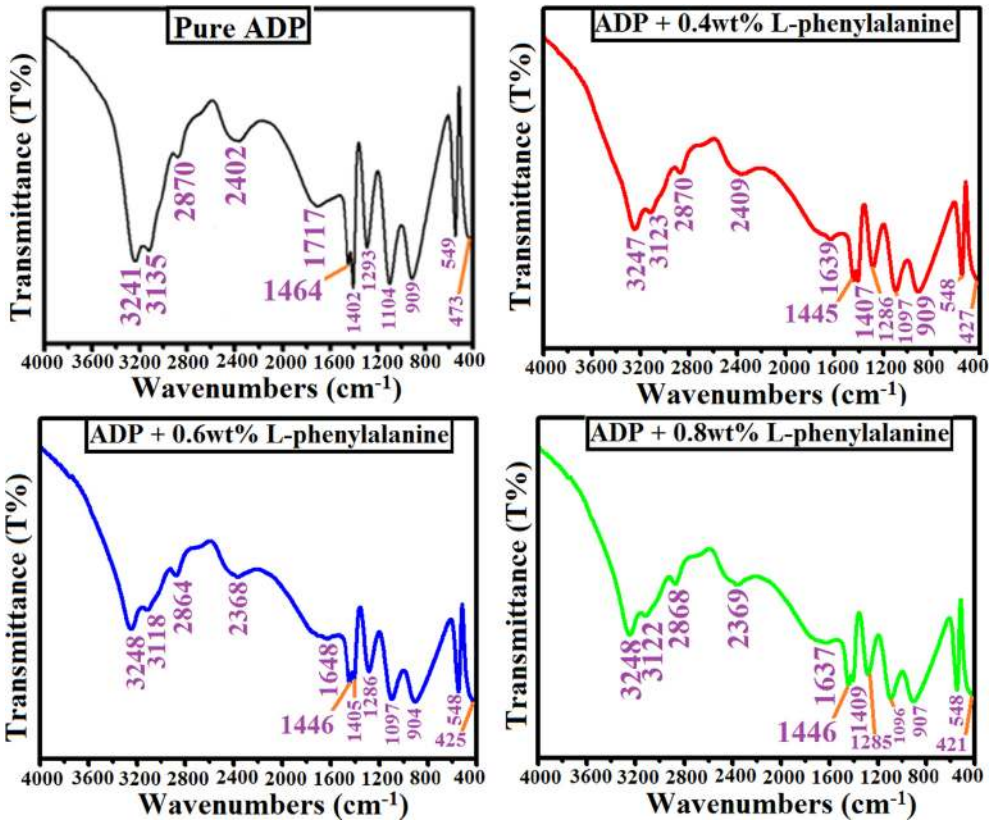

Fig. 1 FT-IR spectra of pure and L-phenylalanine doped ADP crystals.

the abbreviations of Pure, 0.4PHY, 0.6PHY and 0.8PHY are used for pure, 0.4 wt%, 0.6 wt% and 0.8 wt% L-phenylalanine doped ADP crystals, respectively.

1.2. Characterization techniques

The FT-IR spectra were recorded with a THERMO NICOLET 6700 device. The wavenumber range of 400–4000 cm^{-1} is observed using the potassium bromide (KBr) media technique. The linear UV–Visible spectra were recorded over the wavelength range of 200–900 nm with use of a Shimadzu UV – 2450 spectrophotometer. To study the complex admittance spectra of the pelletized sample of crystals in the frequency range from 10 Hz to 10 MHz and the temperature range of 323–373 K, a HIOKI 3532 LCR HITERSTER is used. Here we have grounded our grown crystals into fine powder using the morter pestol and then using the circular pelltizer we have prepare the solid circular pellets for electrical study. To study the photoconductivity, a Keithley 485 pm with the field variation from 5 to 100 V/cm is used. The second harmonic generation efficiency (SHG) of grown crystals is measured using a Q-switched Nd:YAG laser with wavelength of 1064 nm and output energy 0.71 J. The CW He-Ne laser of wavelength 632 nm is used to study the Z-scan. A Nd:YAG laser of 1064 nm wavelength, 10 ns pulse width and 35 cm focal length is applied to estimate the laser damage threshold of the grown crystals.

2. Results and discussion

2.1. FT-IR spectroscopy

The FT-IR spectra of pure and L-phenylalanine doped ADP crystals are shown in Fig. 1. It is seen that the absorption spectrum of pure ADP crystal is changed by L-phenylalanine doping that confirms the presence of L-phenylalanine in grown ADP crystals. The band assignments for pure and L-phenylalanine doped ADP crystals are given in Table 1.

2.2. UV–Visible spectroscopy

The UV–Visible transmittance spectra of pure and doped ADP crystals are shown in Fig. 2. In the present study the transparent crystals of flat surface having thickness of 2 mm is used to study the optical transmittance. The optical transmittance increases as the wavelength and doping level increase in the ADP crystals. The pure ADP crystal shows 67% maximal transmittance in entire visible range, while the doped ADP crystals show 92% (0.4 PHY), 95% (0.6 PHY) and 99% (0.8 PHY) maximal transmittance. Hence, as mentioned earlier, due to molecular chirality and zwitter ionic structure of L-phenylalanine the optical transmittance is improved.

To evaluate the optical energy band gap, refractive index, refractive index dispersion below the energy band gap etc., the following equations are used (Joshi et al., 2020).

The plots used for the determination of optical energy band gaps of pure and doped ADP crystals are shown in Fig. 3. The energy band gap is deduced by the extrapolation onto the X-axis of such plot. One can see that the optical energy band is enhanced on doping L-phenylalanine in ADP, hence the doped crystals can find various applications in the field of optoelectronics (Parthasarathy et al., 2012). For nonlinear optical materials, it is desirable that such crystal has the large energy

Table 1 FT-IR band assignment of pure and L-phenylalanine doped ADP crystals.

Wavenumbers (cm^{-1})				Band Assignment
Pure ADP	0.4 PHY	0.6 PHY	0.8 PHY	
3241, 3135	3247, 3123	3248, 3118	3248, 3112	O—H vibration of P—O—H group and N—H vibration of NH_4
2870	2870	2864	2868	N—H stretching of NH_4 and Symmetric stretching of C—H of L-phenylalanine doped ADP
2402	2409	2368	2369	Hydrogen Bond
1717	1639	1648	1637	O—H bending of P—O—H group, C═O stretching of COOH
1464	1445	1446	1446	N—H vibration of NH_4, C—O stretching
1402	1407	1405	1409	Bending vibration of ammonia
1293	1286	1286	1285	Combination of asymmetric stretching of PO_4 with lattice
1104	1097	1097	1096	P—O—H stretching, C—O stretching
909	909	904	907	P—O—H stretching, C—H stretching
549 473	548, 427	548, 425	548, 421	PO_4 vibration

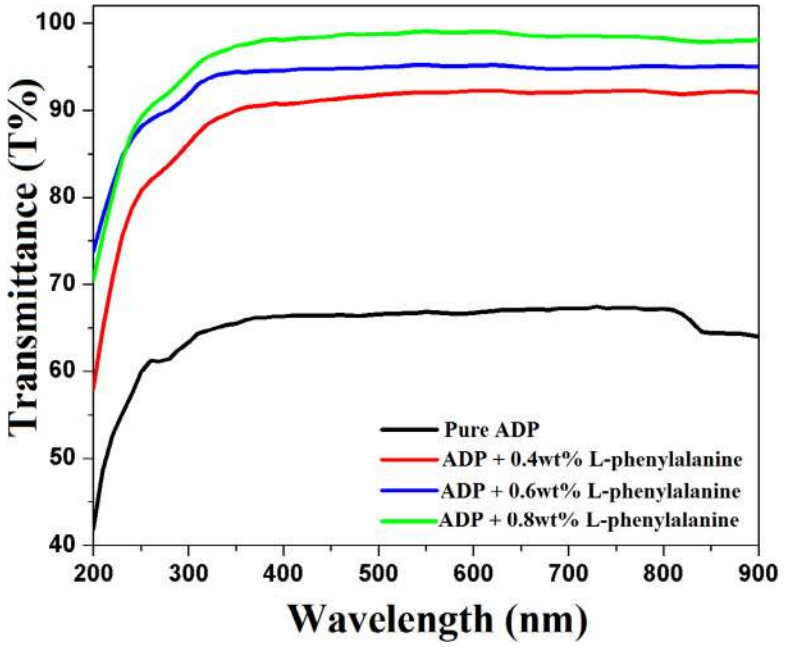

Fig. 2 UV–Visible transmittance spectra of pure and L-phenylalanine doped ADP crystals.

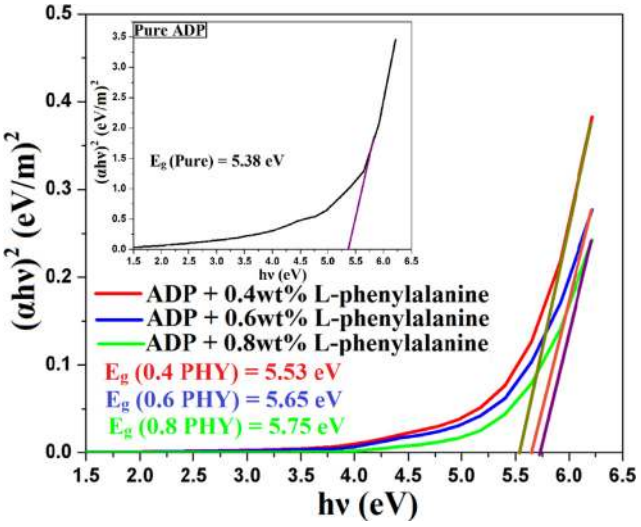

Fig. 3 The tauc plot of pure and L-phyenylalanine doped ADP crystals.

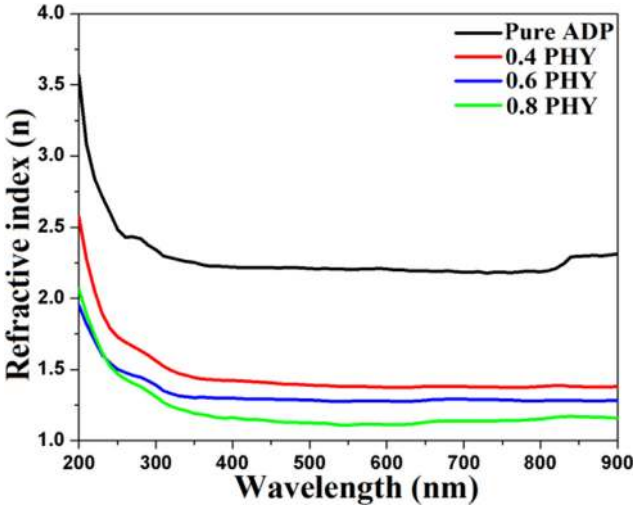

Fig. 4 Refractive index spectra of pure and L-phenylalanine doped ADP crystals.

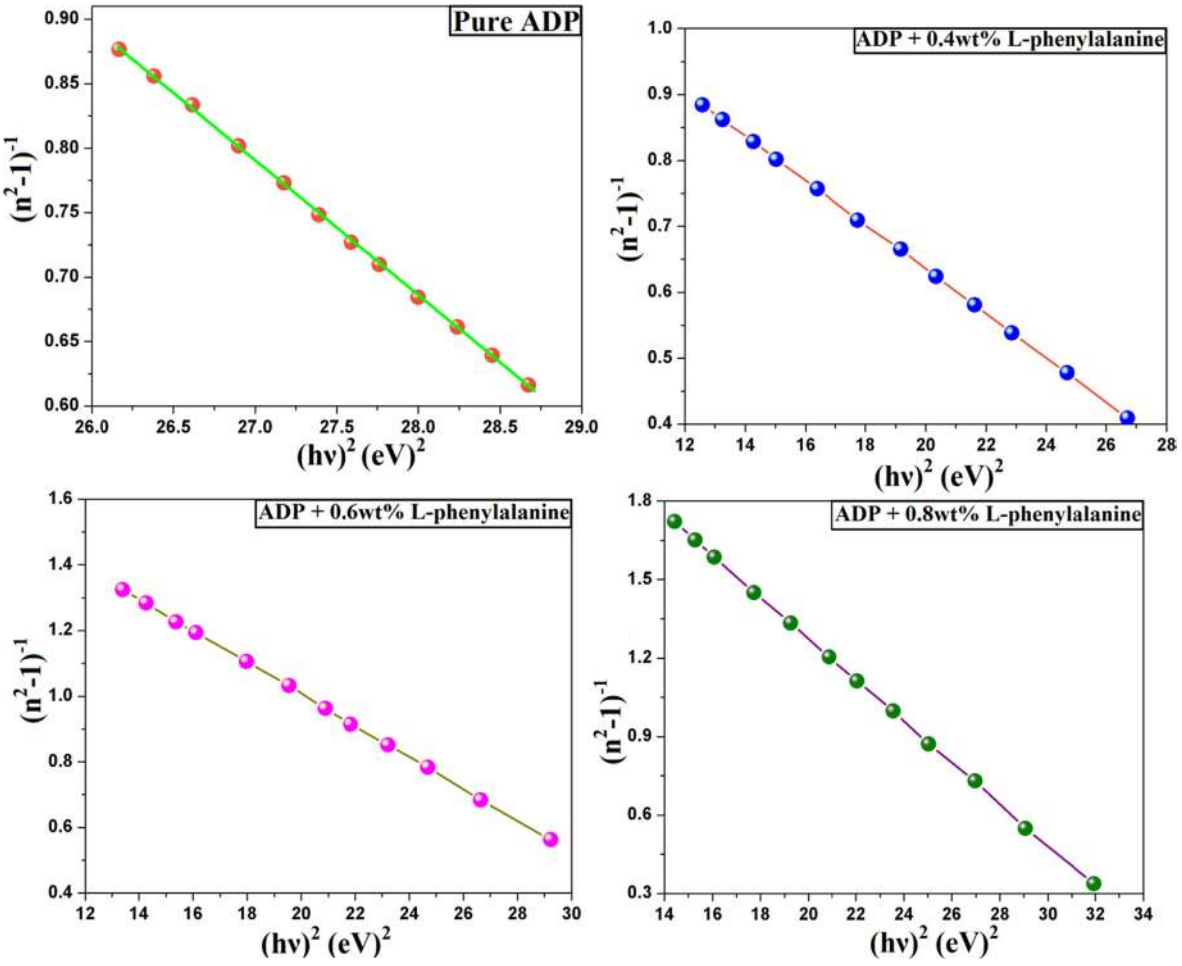

Fig. 5 (a-d). Refractive index dispersion plots for pure and L-phenylalanine doped ADP crystal.

band gap because the value is the measure of polarization for any dielectric material.

The dispersive refractive index of pure and L-phenylalanine doped ADP crystals is shown in Fig. 4. The present refractive index is deduced from the obtained transmitance data. However, our early study shown that the ADP crystal is a negative uniaxial crystal, since the refractive index magnitude of ordinary light is greater than the magnitude of extra ordinary light for ADP crystal (Joshi et al., 2020). From the figure one can see that the trend of refractive index over wavelength range is exactly opposite than that of transmittance for all grown crystals. Hence, the L-phenylalanine doped ADP crystals have lower refractive indices, as compared to pure ADP (Akhtar and Podder, 2011).

2.3. Refractive index dispersion

To study the refractive index dispersion below the energy band gap for pure and L-phenylalanine doped ADP crystals, the Wemple - DiDomenico Single oscillator model is applied (Wemple and DiDomenico, 1971). Such model consists of inter band transition of electron as a single oscillator in brillouin zone, which behaves as an individual oscillator and recognizes that the valence electrons of atoms contribute to one such oscillator.

In Fig. 5, the refractive index dispersion below the energy band gap for pure and L-phenylalanine doped ADP crystals is shown. By fitting of experimental data by straight lines, the single oscillator energy (E_0), the dispersion energy (E_d)

Table 2 Dispersive parameters of pure and L-phenylalanine doped ADP crystals.

Sample	E_d (eV)	E_0 (eV)	M_{-1}	M_{-3}
Pure ADP	1.63	5.58	0.27681	0.00801
0.4 PHY	1.45	4.14	0.34993	0.02038
0.6 PHY	1.80	3.55	0.50727	0.04025
0.8 PHY	1.18	2.85	0.76550	0.09392

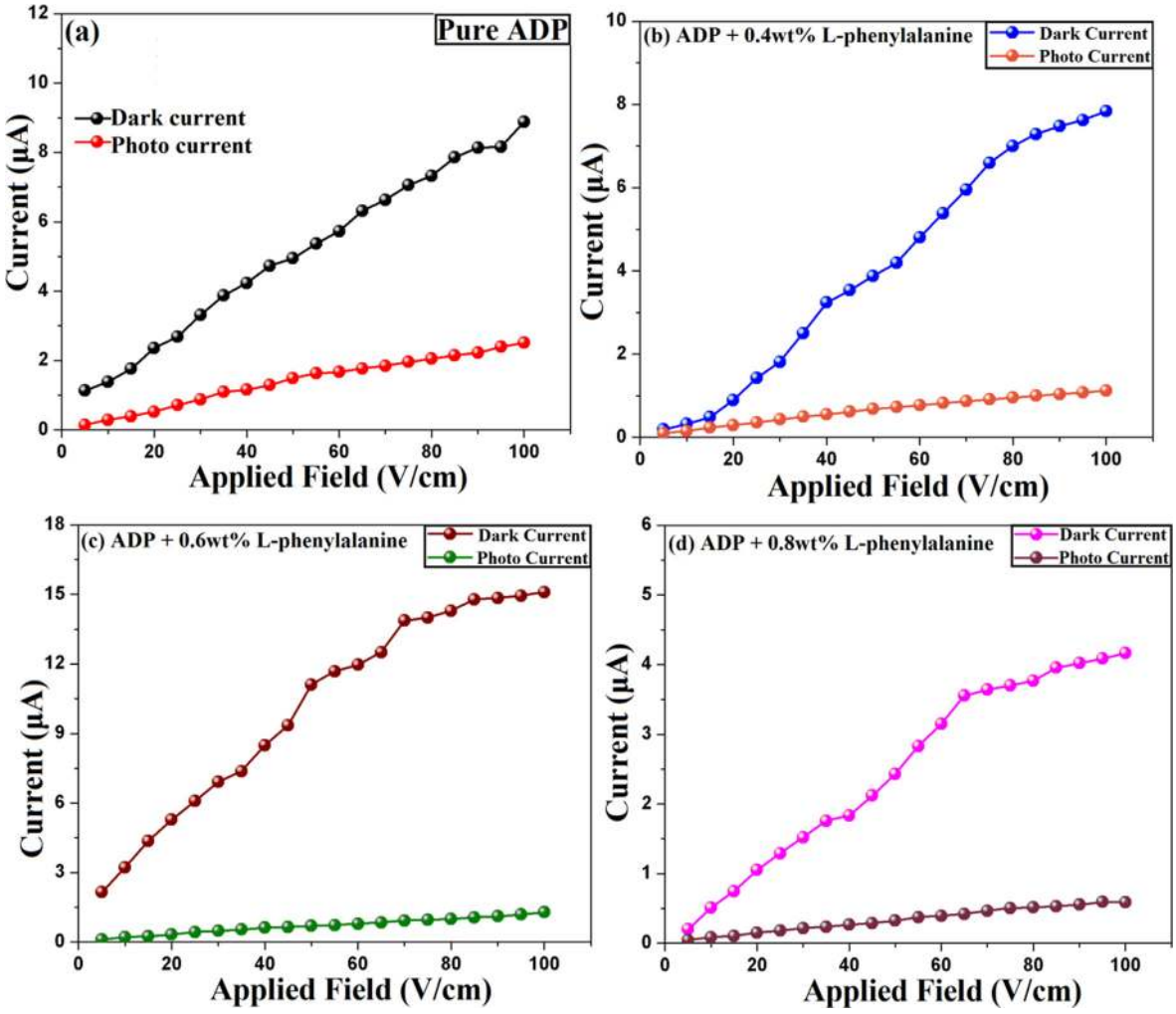

Fig. 6 (a-d). Photoconductivity spectra of pure and L-phenylalanine doped ADP crystal.

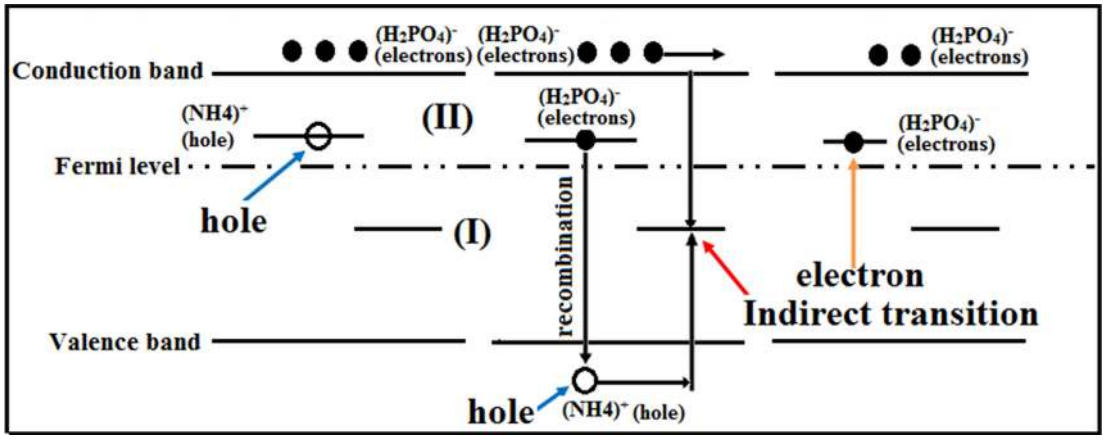

Fig. 7 Illustration of negative photoconductivity.

and the momentum of such oscillator model (M_{-1} and M_{-3}) are calculated. The calculated values are tabulated in Table 2.

From Table 2, it is seen that the single oscillator energy (E_0) decreases on doping the L-phenylalanine in ADP crystal. It is desirable that this parameter is as low as possible because it corresponds to the average bond strength of material. Hence, on doping the L-phenylalanine in ADP, the bond strength of ADP reduces as reflected in decrement in E_0. Therefore, the incorportion of L-phenylalanine in ADP crystal can be expected by mean of decreasing the single oscillator energy. Moreover, the E_0 parameter for 0.8 wt% L-phenylalanine doped ADP crystal is, at least, among those for the other

concentrations, and, hence, that crystal possesses superior nonlinear optical quality.

3. Photoconductivity study

The variation of dark and photo current versus applied field plot for all grown crystals is shown in Fig. 6. The dark current is found to be greater than photo current due to the negative photoconductivity (Bube, 1981). Such behaviour is attributed to the charge carrier concentration reduction and carrier life time in the presence of radiation. The negative photoconductivity of grown crystals is useful for the UV and IR detector applications (Joshi, 1990). The mechanism of negative photoconductivity is illustrated in Fig. 7.

4. Complex admittance spectroscopy

The complex admittance spectroscopy is valuable for the interlayer formation at electro - electrolyte interface and the charge carrier motion. Such spectroscopy consists of capacitance and conductance as a function of temperature or frequency. The equation used for both parameters is given elsewhere (Ertugrul and Tataroglu, 2012).

The temperature variation of capacitance at angular frequency 100 rad s^{-1} for pure and L-phenylalanine doped ADP crystals is shown in Fig. 8. The formation of interlayer at electrode - electrolyte interface and thermally activated charge carrier motion leads to increase of the capacitance of grown crystals as the temperature is increased. By comparing the magnitude of capacitance, it is found that the 0.8 wt% L-phenylalanine doped ADP crystal has highest magnitude among all samples. Such behavior corresponds to fact that the material with highest polarizability should possess the highest magnitude of capacitance.

In Fig. 9, the temperature variation of the conductance for pure and L-phenylalanine doped ADP crystals at angular frequency 100 rad s^{-1} is given. The capacitance and conductance increase with the temperature and it suggests that the thermally activated process occurs within grown crystals. Also, the magnitude of conductance is found to be higher in the case

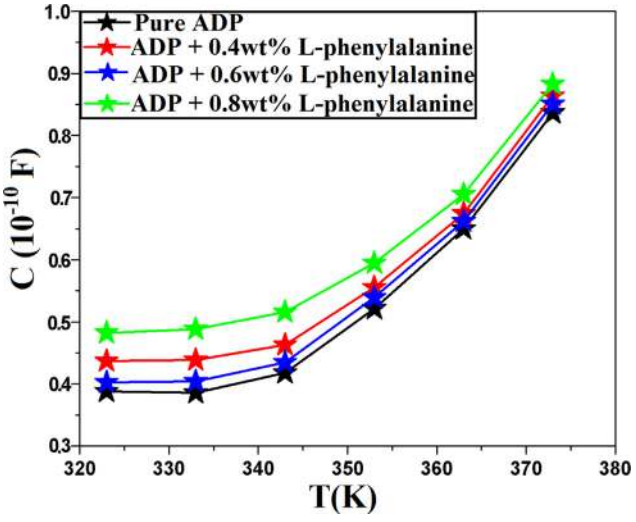

Fig. 8 Capacitance versus temperature of pure and L-phenylalanine doped ADP crystals at ω = 100 rad s^{-1}.

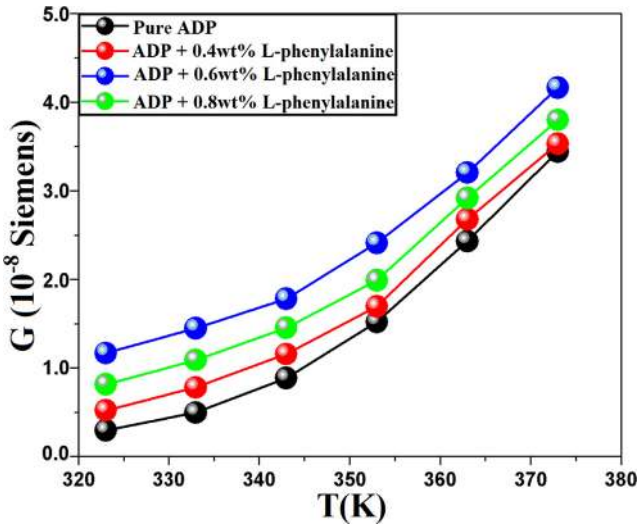

Fig. 9 Conductance versus temperature of pure and L-phenylalanine doped ADP crystals at ω = 100 rad s^{-1}.

of L-phenylalanine doped ADP crystals compare to that pure ADP crystals. Among the three different doping concentrations of L-phenylalanine in ADP crystal, the 0.6 wt% Lphenylalanine doped ADP crystal has the highest magnitude of conductance. Such behavior corresponds to the fact that the dopant causes the defect in terms of vacancy created in hydrogen bonding. Such defect leads to the conductance enhancement of grown crystals (Joshi et al., 2018).

5. Second harmonic generation efficiency (SHG)

The nonlinear optical SHG efficiency of pure and different weight percentage L-phenylalanine doped ADP crystals is measured using the Kurtz and Perry powder NLO technique. The fine powdered sample filled micro glass capillary is illuminated by Q-switched Nd: YAG laser of 1064 nm and the output energy of emitted green light of wavelength 532 nm are measured (Alekseev et al., 2013; Atuchin et al., 2017; Atuchin et al., 2018). For such experiment the output energies of pure KDP, pure ADP and different weight percentage L-phenylalanine doped ADP crystals were measured. By taking the ratio of output energy of different crystals over output enrgy of pure KDP and pure ADP, the SHG efficiency is determined. The SHG efficiencies of pure and L-phenylalanine doped ADP crystals are listed in Table 3.

From Table 3, one can notice that the SHG efficiency of ADP crystal increases on doping by L-phenylalanine. The reason for such increment can be interpreted as the fact that, the

Table 3 SHG efficiency of pure and L-phenylalanine doped ADP crystals.

Sample	Output Energy (mJ)	SHG Efficiency	
		W.R.T. Pure KDP	W.R.T. Pure ADP
Pure KDP	19.5	1	–
Pure ADP	40.3	2.06	1
0.4 PHY	46.9	2.40	1.16
0.6 PHY	49.4	2.53	1.22
0.8 PHY	52.6	2.69	1.31

doping of L-phenylalanine in ADP crystal provides the larger polarizibility by mean of phonon induced structure displacement (Reshak et al., 2010; Reshak et al., 2008; Khyzhun et al., 2009; Atuchin et al., 2012).

6. Z-scan

After the improvement observed in SHG efficiency of grown crystals, we are attempted to determine the magnitude and sign of third order nonlinear optical parameters for the grown crystals such as nonlinear optical absorption coefficient, nonlinear optical refractive index and nonlinear optical third order susceptibility using the Z-scan measurements. The equations used for the calculation of different parameters in Z-scan are given elsewhere (Joshi et al., 2020).

Presently, the Z-scan experiments are done in open and close aperture modes for all the grown crystals. The Z-scan spectra of pure and different weight percentage of L-phenylalanine doped ADP crystals in open and close aperture modes are shown in Figs. 10a–10d. In analysis of the close aperture plots, it is found that such plots of pure and

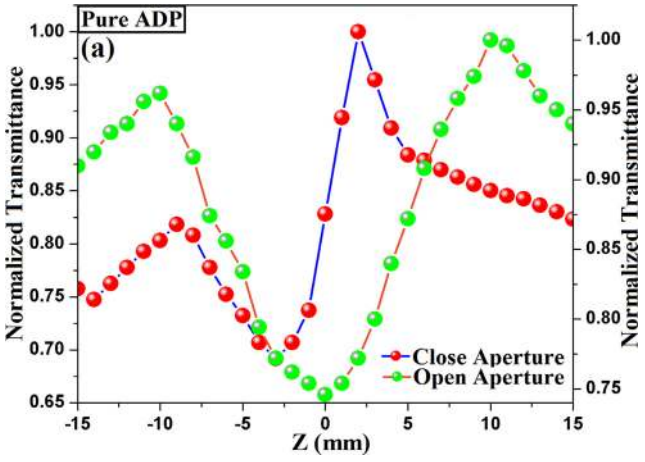

Fig. 10a Z-scan spectra of pure ADP crystal.

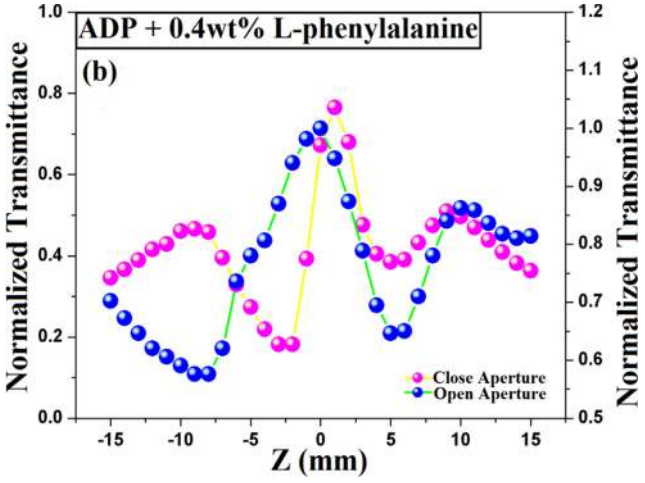

Fig. 10b Z-scan spectra of 0.4 wt% L-phenylalanine doped ADP crystal.

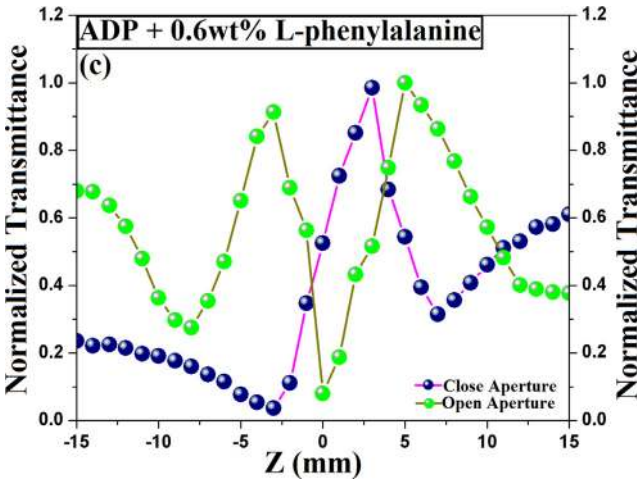

Fig. 10c Z-scan spectra of 0.6 wt% L-phenylalanine doped ADP crystal.

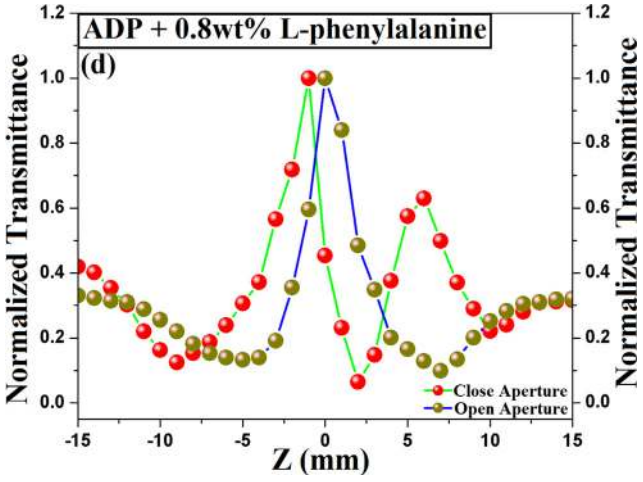

Fig. 10d Z-scan spectra of 0.8 wt% L-phenylalanine doped ADP crystal.

L-phenylalanine doped ADP exhibit the self focusing phenomena as those plots show a valley followed by peak nature. On the basis of such characteristics of grown crystals, they can be utilizing for different nonlinear optical apertures likes optical switches and optical limiters (Ashok Kumar et al., 2010; Natarajan et al., 2010; Dhanaraj and Rajesh, 2011).

Now, on observing the open aperture plots of pure and 0.6 wt% L-phenylalanine doped ADP crystal, it is found that the normalized transmittance of both crystals decrease gradually due to the reverse saturable absorption phenomena. While the similar plots for 0.4 and 0.8 wt% L-phenylalanine doped ADP crystals show gradual increment in normalized transmittance suggested presence of the saturable absorption phenomena.

The obtained third order nonlinear optical parameters such as difference of peak and valley, nonlinear optical refractive index, nonlinear optical absorption coefficient and nonlinear

Table 4 Z-scan parameters of pure and L-phenylalanine doped ADP crystal.

Sample	ΔT_{p-v}	n_2 (cm^2/W)	β (cm/W)	$\chi^{(3)}$ (esu)
Pure ADP	0.00196	6.34×10^{-11}	9.55×10^{-6}	1.73939×10^{-5}
0.4 PHY	0.00589	6.35×10^{-10}	2.46×10^{-5}	4.87864×10^{-5}
0.6 PHY	0.00613	7.79×10^{-9}	4.03×10^{-5}	9.17138×10^{-5}
0.8 PHY	0.00596	1.51×10^{-8}	9.90×10^{-5}	2.15838×10^{-4}

Table 5 Comparative analysis of third order nonlinear optical susceptibilities of KDP family crystals.

Sample	$\chi^{(3)}$ (esu)
Pure ADP [present study]	1.73939×10^{-5}
0.4 PHY [present study]	4.87864×10^{-5}
0.6 PHY [present study]	9.17138×10^{-5}
0.8 PHY [present study]	2.15838×10^{-4}
Formic acid doped KDP (Anis et al., 2014)	3.81×10^{-7}
L-Valine doped ADP (Shaikh et al., 2015)	2.249×10^{-5}
Oxalic acid doped KDP (Anis et al., 2015)	1.90×10^{-7}
Malic acid doped KDP (Anis et al., 2015)	8.99×10^{-8}

Table 6 Power density values of pure and L- phenylalanine doped ADP crystals.

Sample	Power density (P_d) (GW/cm^2)
Pure ADP	3.55
0.4 PHY	10.5
0.6 PHY	12.2
0.8 PHY	20.2

optical third order susceptibility of pure and L-phenylalanine doped ADP crystals are tabulated in Table 4.

Similar to SHG efficiency, from Table 4, one can observe that the third order nonlinear optical susceptibility of L-phenylalanine doped ADP crystals increases compared to pure ADP crystal. Also, from the Table 5 it can be seen that the L-phenylalanine doped ADP crystals retain greater magnitude of the nonlinear optical third order susceptibility compare to other crystals of KDP family. Moreover, alike SHG efficiency, the 0.8 wt% L-phenylalanine doped ADP crystal has consumed highest magnitude of third order nonlinear optical susceptibility among the other samples. Such behaviour may attribute to the improvement in molecular polarizibility due to delocalization of π – electron cloud (Joshi et al., 2018).

7. Laser damage threshold study

For the laser damage threshold study of grown crystals, here we have measured power density (Joshi et al., 2020). The obtained power densities of pure and L-phenylalanine doped ADP crystals are listed in Table 6.

For a nonlinear optical crystal, it is desired to have large optical damage threshold against the intense laser radiation. The crystal with crystalline perfection, higher molecular chirality and ability of polarization may have higher LDT. From Table 6, such characteristic may be attributed to the 0.8 wt % L-phenylalanine doped ADP crystal among the other samples.

8. Conclusion

The successful growth of pure and different weight percentage of L-phenylalanine doped ADP crystals is made. The single phase tetragonal crystals were been grown. The presence and alteration of various functional groups of ADP crystals containing L-phenylalanine are observed by FT-IR spectroscopy. The L-phenylalanine doping in ADP crystals is found to be beneficial for the improvement in optical transmittance, energy band gap, second and third order nonlinear optical efficiencies and the laser damage threshold.

Declaration of Competing Interest

All the authors of present manuscript declare there is no conflict of interest.

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