STM JOURNALS

Research & Reviews: Journal of Physics
ISSN: 2278-2265 (Online), ISSN: 2347-9973 (Print)
Volume 8, Issue 2
www.stmjournals.com

Growth and Studies of Strontium Chloride Doped Ammonium Dihydrogen Phosphate Crystals

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Abstract
Single crystals of undoped ammonium dihydrogen phosphate (ADP) and strontium chloride doped ammonium dihydrogen phosphate were grown by a solution method. It is found that SCADP crystal belongs to the tetragonal crystal system with I-42d space group. It is seen that both undoped and strontium chloride doped ADP crystals crystallize in the same tetragonal system and the incorporation of dopant does not alter the crystal system. This is due to the presence of dopant strontium chloride in the form of metal ions and halogen ions in the interstitial positions of the lattice of the host ADP crystal. Structural studies of the grown crystals were carried out to find the crystal structure and due to doping of strontium chloride, the structure of the doped ADP crystal was not altered. Microhardness studies of the grown crystals of undoped and strontium chloride doped ADP crystals were done to understand the mechanical strength. The second order and third order NLO studies of the samples were carried out to obtain the NLO properties. Spectroscopic studies like FTIR studies, EDAX studies and UV-visible spectral studies were carried out for the grown crystals. Also thermal and LDT studies were performed for the characterization of the prepared samples.

Keywords: *ADP crystal, characterization, doping, hardness, SHG, single crystal, spectroscopy, solution growth Z-scan*

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INTRODUCTION

Nonlinear optical, photonic and optoelectronic technologies need the suitable nonlinear optical materials with high Second Harmonic Generation (SHG) efficiency and high laser damage threshold. It is known that inorganic crystals like potassium dihydrogen phosphate (KDP), lithium triborate (LBO), β-barium borate (βBO), lithium niobate ($LiNbO_3$), potassium niobate ($KNbO_3$), etc have high SHG efficiency and laser damage threshold. Ammonium dihydrogen phosphate (ADP) is an inorganic NLO crystal which crystallizes in tetragonal crystal system like KDP and it is used as the second harmonic and third harmonic generating material. And this crystal is also used in electro-optic and acousto-optic applications [1-3]. Many researchers have done research on the effect of many dopants on the various properties of ADP crystals [4-7]. Solution growth with slow evaporation technique was employed to grow the single crystals of strontium chloride doped ammonium dihydrogen phosphate (SCADP). The solubility of the sample was measured by the gravimetric method at different temperatures. Single crystal X-ray diffraction technique was adopted to identify the crystal structure. Fourier Transform Infrared (FTIR) spectrum of SCADP crystal was recorded and analyzed to find the functional groups present in the sample. Second harmonic generation (SHG) efficiency was measured by Kurtz-Perry technique. Hardness and work hardening coefficient of undoped and strontium chloride doped ADP crystals were evaluated by Vickers microhardness method.

SYNTHESIS AND GROWTH OF CRYSTALS

AR grade chemicals like ammonium dihydrogen phosphate (ADP), strontium chloride were purchased commercially from Merck chemical company, India. The salt of strontium chloride doped ADP was synthesized by an aqueous solution method. 1 mole% of strontium chloride was added into the aqueous solution of pure ADP and solution was prepared and it was heated at 60°C till the solvent evaporates to obtain the doped salt. Single crystals of the synthesized salt of strontium chloride doped ADP were grown from aqueous solution by slow evaporation technique. A saturated aqueous solution of the salt was prepared and the magnetic stirring for two hours was performed to obtain the homogenous solution. The solution was filtered using the Whatman filter papers and then the solution was taken in a growth vessel covered with a porous paper and kept in a dust free environment. Due to slow evaporation, the single crystals of SCADP were harvested after 30 days. Also, here undoped ADP crystals were grown by solution method and the photographs of undoped ADP crystal and strontium chloride doped ammonium dihydrogen phosphate (SCADP) are shown in the Figures 1(a) and (b). The morphology and transparency of ADP crystals are changed when they are doped with strontium chloride.

RESULT AND DISCUSSION

Microhardness Studies

Measurement of hardness is an important characterization of crystalline samples and the hardness is the ratio of the applied load to the surface area generated due to indentation. Usually, it is observed that hardness of mixed and doped crystals always exceeds that of the individual constituent materials. Hardness tests are commonly carried out to determine the mechanical strength of materials and it correlates with other mechanical properties like elastic constants and yield stress. Hardness measurements can also be defined as macro, micro and nano according to the forces applied and displacement obtained. Among the various methods of hardness measurements, static indentation test is the popular and simplest, in which a steady load is applied to an indenter. The indenter may be a ball or diamond cone or diamond pyramid. The hardness is calculated from the area or the depth of indentation produced. The indenter is made up of a very hard material to prevent its deformation by the test piece so that it can cover materials over a wide range of hardness. For this reason, either a hardened steel sphere or a cone diamond pyramid is employed. In the process of static indentation test, the indenter is pressed perpendicularly into the surface of a sample by means of an applied load.

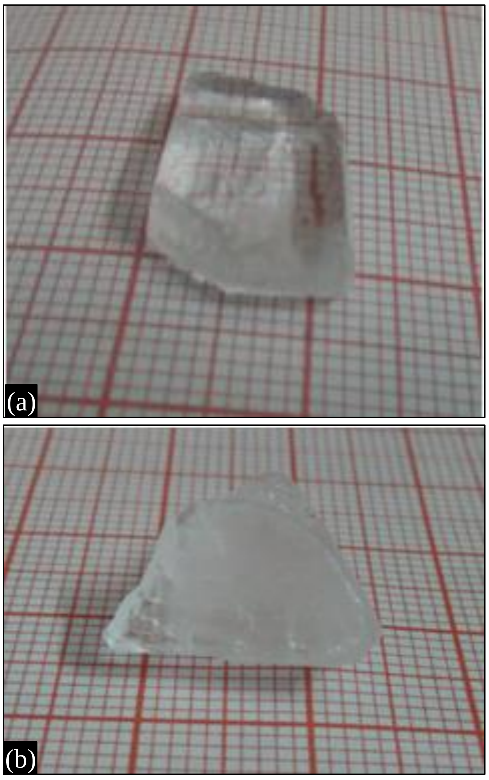

Fig. 1: *(a) A Single Crystal of Undoped ADP. (b) A Single Crystal of Strontium Chloride doped ADP.*

By measuring the correctional area or depth of indentation and knowing the applied load, the hardness number is evaluated. The undoped ADP and SCADP crystals were subjected to microhardness studies and for each load, several indentations were made and the average value of indentation length was found. Vickers hardness test was used to find the micro hardness number (H_v) and the relation employed is $H_v = (1.8544 * P) / d^2$, where P is the applied load and d is average indentation length and using the values of d, the micro hardness number is evaluated. The variations of average indentation length (d) and the applied load for undoped and strontium chloride doped ammonium dihydrogen phosphate (SCADP) are presented in Figure 2. The results reveal that the average indentation length increases with the increase of the applied load for both the samples and average indentation length are found to be less for SCADP crystal compared to that of undoped ADP crystal. The evaluated values of H_v for undoped and strontium chloride doped ADP crystals are presented in the Figure 3. The results indicate that the hardness shows a nonlinear behaviour with the applied load for both the samples. The hardness is found to be increasing with the increase of the applied load up to 50 g and it decreases up to 75 g and then again hardness increases until cracks are formed beyond 100 g. The increasing part is due to the tangled forest of dislocation lines and the work hardening mechanism. The subsequent decrease in hardness with an increase in load was due to work softening mechanism which resulted from the activation of cross slip and the movement of piled-up dislocations. The movement of dislocations is responsible for the increase and decrease behaviour of the hardness in the samples.

The work hardening coefficient of the samples was determined using Meyer's law. This law connects the average diagonal indentation length (d), the applied load (P) and the work hardening coefficient (n) and it is given by $P=ad^{n}$. Taking log on both sides, equation obtained is log P = log a + n log d where 'a' is a constant. Hence, if a plot of log P versus log d is drawn, the work hardening coefficient (n) could be found. Figure 4 and 5 show the plots of log P versus log d for undoped and strontium chloride doped ADP crystals and the obtained values of work hardening coefficient are 2.3992 and 2.2143 respectively. From careful observations on various materials, Onitsch and Hanneman pointed out that an 'n' lies between 1 and 1.6 for hard materials and it is more than 1.6 for soft materials. Hence, the grown crystals belong to the category of soft materials.

The yield strength of the samples was determined using the following formula.

$$\text{Yield strength } (\sigma_y) = (H_v/3)\,(0.1)^{n-2}$$

Where, σ_y is the yield strength and Hvis the hardness of the material. The elastic stiffness constant (C_{11}) for different loads was calculated using Wooster's empirical formula as given below.

$$C_{11} = H_v^{7/4}$$

The calculated values are of mechanical parameters like yield strength and stiffness constant of undoped and strontium chloride doped ADP crystals are provided in Tables 1 and 2. It is observed that these mechanical parameters increase with the increase of the applied load on the surface of the samples. These results indicate that the mechanical parameters are found to be more when ADP crystals are doped with strontium chloride and this is probably the presence of the dopant 'strontium chloride' in the interstitial position of the lattice enhances the mechanical strength of the doped sample.

***Table 1:** Values of Yield Strength and Stiffness Constant of Undoped ADP Crystal.*

P (grams)	σ_y(M Pa)	$C_{11}\times10^{14}$ Pa
25	84.4	19.4
40	90.2	22.9
50	94.6	27.2
70	89.1	25.7
90	95.4	27.9
100	103.3	32.3

***Table 2:** Values of Yield Strength and Stiffness Constant of SCADP Crystal.*

P (grams)	σ_y(M Pa)	$C_{11}\times10^{14}$ Pa
25	110.7	25.4
40	122.1	28.5
50	134.4	30.9
70	130.2	27.9
90	136.2	31.4
100	148.2	36.2

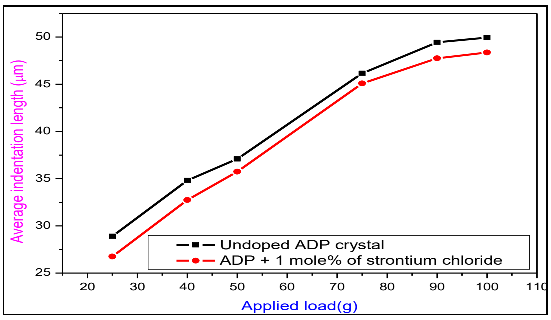

***Fig. 2:** Variation of Average Diagonal Indentation Length with Applied Load for Undoped and Strontium Chloride doped ADP Crystals.*

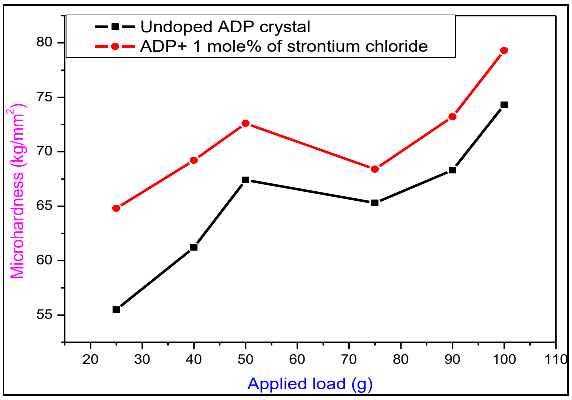

Fig. 3: *Variation of Hardness with Applied Load for Undopedand Strontium Chloride doped ADP Crystals.*

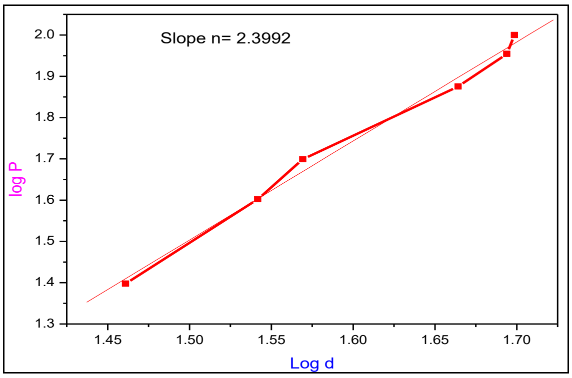

Fig. 4: *The plot of log (P) versus log (d) for Undoped ADP Crystal.*

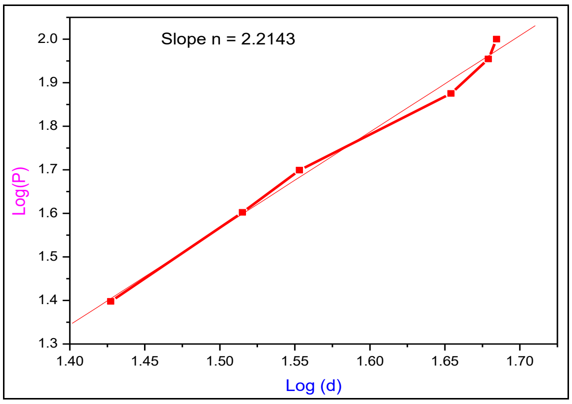

Fig. 5: *The plot of log (P) versus log (d) for Strontium Chloride doped ADP Crystal.*

Second Order NLO Studies

Second order NLO studies were carried out by Kurtz and Perry powder technique. The level of SHG response of a given material is inherently dependent upon its structural attributes. A Q-switched Nd: YAG laser beam of wavelength 1064 nm was allowed to strike the samples normally. The second harmonic signal generated in the crystal was confirmed from the emission of green radiation by the crystal. The SHG radiation of 532 nm was collected by a photomultiplier tube and converted into voltage output at the CRO. The input laser energy incident on the powdered samples was chosen to be 0.68 J/pulse, the output was found to be 16.8 mJ/pulse, 18.25 mJ/pulse for undoped and strontium chloride doped ADP crystals respectively and output of 8.8 mJ/pulse was observed from the KDP. Thus, the relative SHG efficiency of undoped ADP is found to be 1.9 and for SCADP is found to be 2.07 times that of KDP. Also, the relative SHG efficiency of SCADP is found to be 1.09 times greater than ADP.

FTIR Studies

Fourier Transform infrared (FTIR) studies were carried out for strontium chloride doped ADP crystal using a Bruker IR spectrometer in the wave number range 400-4000 cm^{-1}. This study helps to find the functional groups present in the sample. Here KBr pellet technique was adopted for the pelletized sample. The recorded FTIR spectra of undoped and strontium chloride doped ADP crystals are shown in the Figure 6 and 7. The broad absorption band in the wave number range 3400-3100 corresponds to OH stretching vibration of P-O-H group and N-H stretching of NH_4 group. The peak at 2369 cm^{-1} is due to P-H stretching in the case of SCADP crystal. But in the case of undoped ADP crystal, P-H stretching occurs at 2357 cm^{-1}. The peak at 1653 cm^{-1} is related to P=O stretching. The absorption peak at 1403 cm^{-1} is due to the bending vibration of NH_4. The IR absorption peak at 1283 cm^{-1} corresponds to the bending vibration of PO_4. The peak at 1106 cm^{-1} is related to bending vibration of the P=O group. The peak observed at 903 cm^{-1} corresponds to bending vibration of P-O-H group. The peak at 539 cm^{-1} is due to bending vibration of PO_4 and the small peak at 461 cm^{-1} is also related to bending vibration of PO_4. Compared to FTIR spectrum of undoped ADP crystal (Figure 6), the absorption peaks/bands in the FTIR spectrum of SCADP crystal are slightly broadened or narrowed and this is due to the incorporation of strontium chloride in the host ADP crystal.

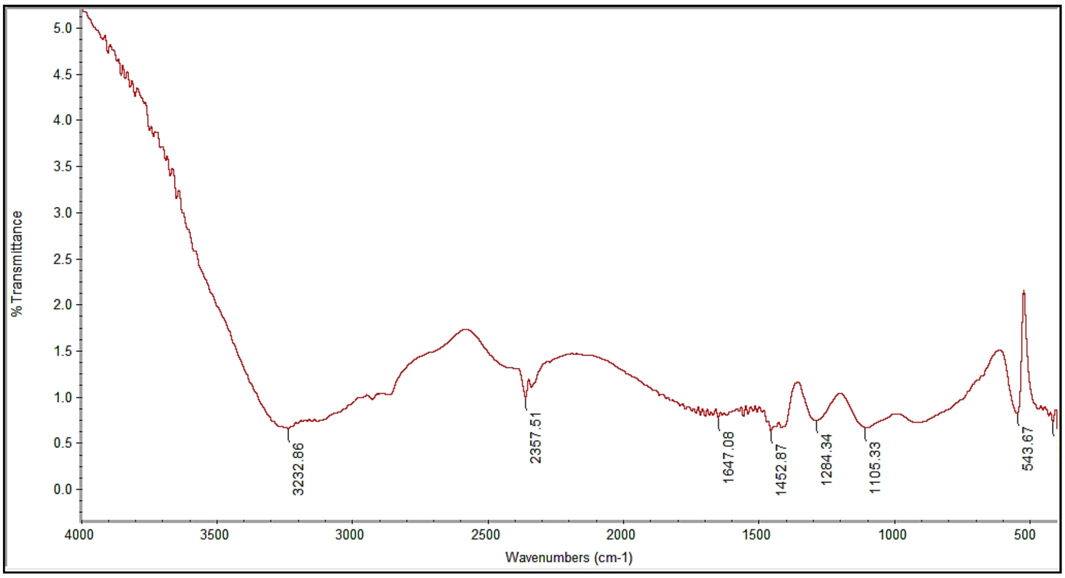

***Fig. 6:** FTIR Spectrum of Undoped ADP Crystal.*

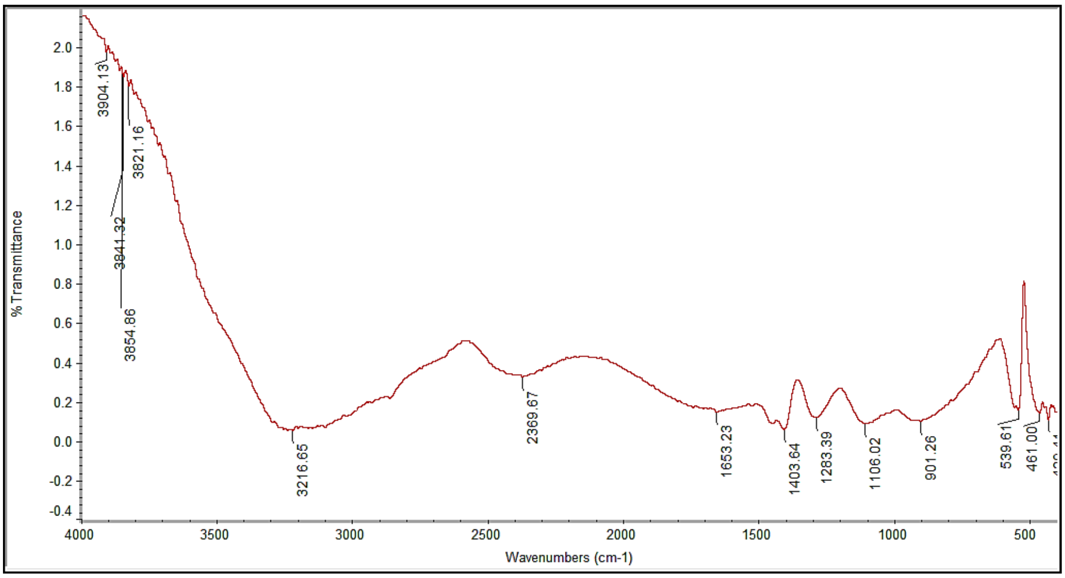

***Fig. 7:** FTIR Spectrum of SCADP Crystal.*

EDAX Studies

Energy Dispersive Analysis by X-rays (EDAX) is a technique that detects X-rays emitted from the sample during bombardment by an electron beamto characterize thethe elemental composition ofthe of thethe analyzed volume. The datagenerated by EDAX analysis consist of spectra showingpeaks corresponding to theelements making up the true composition of the sample being analyzed. When the sample is bombarded by the SEM's electron beam, electrons are ejected from the atoms comprising the sample's surface. The resulting electron vacancies are filled by electrons from a higher state, and an X-ray is emitted to balance the energy difference between the two electrons states. The X-ray energy is characteristic of the element from which it was emitted. The EDAX detector measures the relative abundance of emitted X-rays versus their energy. The spectrum of X-ray energy versus counts is evaluated to determine the elemental composition of the sample. The sample X-ray energy values from the EDAX spectrum are compared with known characteristic X-ray energy values to determine the presence of an element in the sample. In this work, the EDAX spectrum of SCADP crystal was recorded using a computer controlled scanning electron microscope and it is shown in Figure 8. From the spectrum, it is confirmed that the elements such as Sr, Cl, O, N and P present in the grown SCADP crystal. It is to be noted here that the element H cannot be identified by EDAX method. The presence of the elements like Sr, Cl, O, N and P in the doped ADP crystal indicates that the dopant strontium chloride has entered into the lattice of the host ADP crystal. The weight percentage and atomic percentage of various elements present in SCADP crystal are provided in Table 3.

Table 3: *Weight Percentage and Atomic Percentage of Elements in SCADP Crystal.*

Element	Series	wt%	at%
O 8	K-series	43.17	66.44
P 15	K-series	22.63	17.99
N 7	K-series	8.56	15.04
Cl 17	K-series	0.64	0.44
Sc 38	K-series	0.32	0.09

TG/DTA Studies

Thermal analysis is necessary to check the thermal stability of a sample and this analysis is related to change in the heat and mass. There are two important methods viz., TG and DTA methods. The measurement of change in the mass of a sample on heating is called Thermogravimetric analysis (TG) and the measurement of change in heat is known as the differential thermal analysis (DTA). There are endothermic and exothermic events when the temperature of the sample is changed. When the heat is absorbed by the sample, it is called as the endothermic event and if the heat is evolved, it is known as the exothermic event. In this work, TG/DTA thermal curves were recorded for SCADP crystal using a thermal analyzer in the temperature 35-600°C in the nitrogen atmosphere. The recorded TG/DTA curves for strontium chloride doped ADP crystal are shown in the Figure 9. It is observed from the results that there is a little weight loss in the temperature range 100-200°C and this is due to eliminating of free water molecules from SCADP crystal. The endothermic peak at 210°C is due to the decomposition/melting point of the sample. It is noticed that about 35% of the weight of the grown crystal is lost at 600°C. The broad exothermic event in the DTA curve beyond 300°C is due to evolving the gaseous particles from SCADP crystal. The literature indicates that the undoped ADP crystal decomposes at 194°C [8, 9]. Therefore, the melting/decomposition point of SCADP crystal at 210°C is found to be higher than that of undoped ADP crystal and thermal stability is more for SCADP crystal.

UV-visible Spectral Studies

The optical transmission and absorbance spectra of strontium chloride doped ADP (SCADP) crystal were recorded using a UV-vis-NIR spectrophotometer in the range of 200-800 nm. A good quality crystal of SCADP is used in this study. The transmittance spectrum of SCADP crystal is shown in the Figure 10. From the transmittance spectrum, it is observed that the absorbance is very low in the visible region and the lower cut-off wavelength is found to be at 222 nm. Using the formula E_g = hc/□□□□the optical band gap of the sample is determined and it is found to be 5.58 eV. Since the absorbance is low in the wavelength range 250-800 nm, this sample can be used as a second harmonic generator for NLO applications.

Using the transmittance data, the values of the optical absorption coefficient for different values of wavelength for SCADP crystal were calculated using the formula

$$\alpha = (2.303 \log (1/T))/t$$

Where, T is the transmittance and t is the thickness of the crystal and these values are given in Figure 11. From the results, it is noticed that the absorption coefficient is low in the visible region and it is an important parameter for NLO effects like a second harmonic generation. The extinction coefficient is the fraction of energy lost and it is determined using the relation $K = \lambda\alpha / 4\pi$ where λ is the wavelength and α is the absorption coefficient. ~~figure~~ Figure 12. From the result, it is observed that the extinction coefficient increases with an increase in wavelength after the cut-off wavelength. Tauc's plot (Figure 13) for SCADP crystal is drawn using the Tauc's relation as given below.

$$\alpha = (2.303 \log (1/T))/t$$

Where, E_g is optical band gap of the crystal, A is a constant and hν is the optical energy. Here n is equal to 2 for the direct band gap material like SCADP crystal. Using this plot, the optical band gap of SCADP crystal was found to be 5.6 eV.

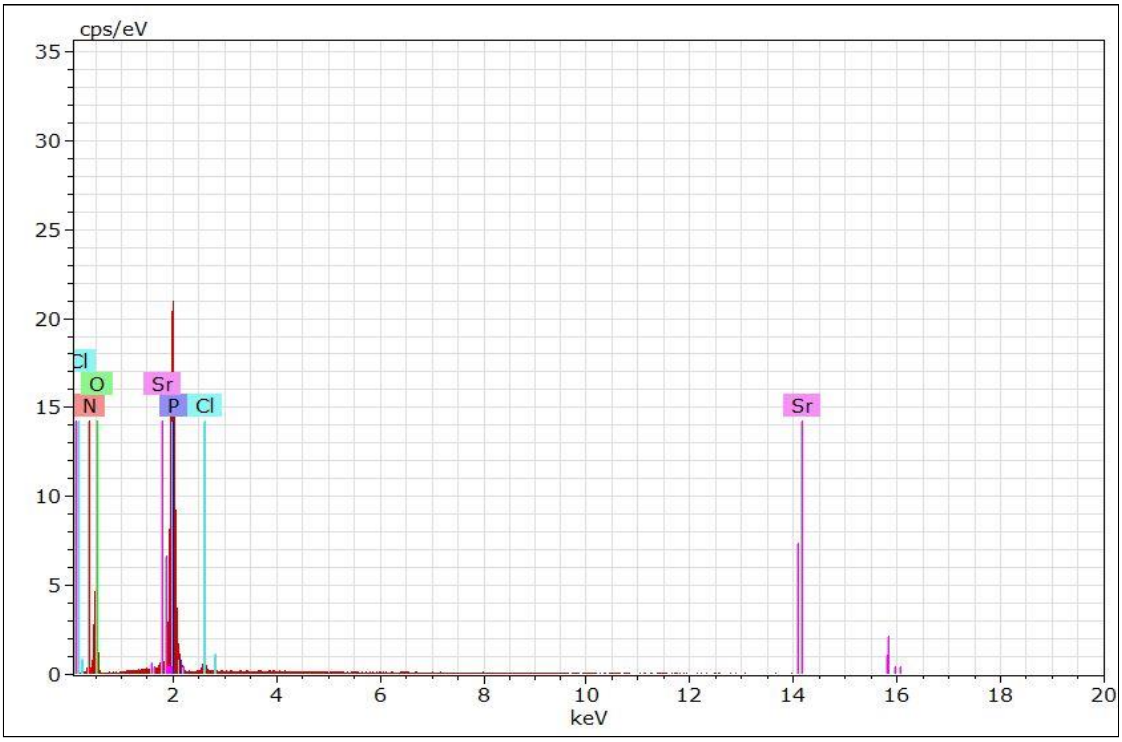

Fig. 8: *EDAX Spectrum of Strontium Chloride doped ADP Crystal.*

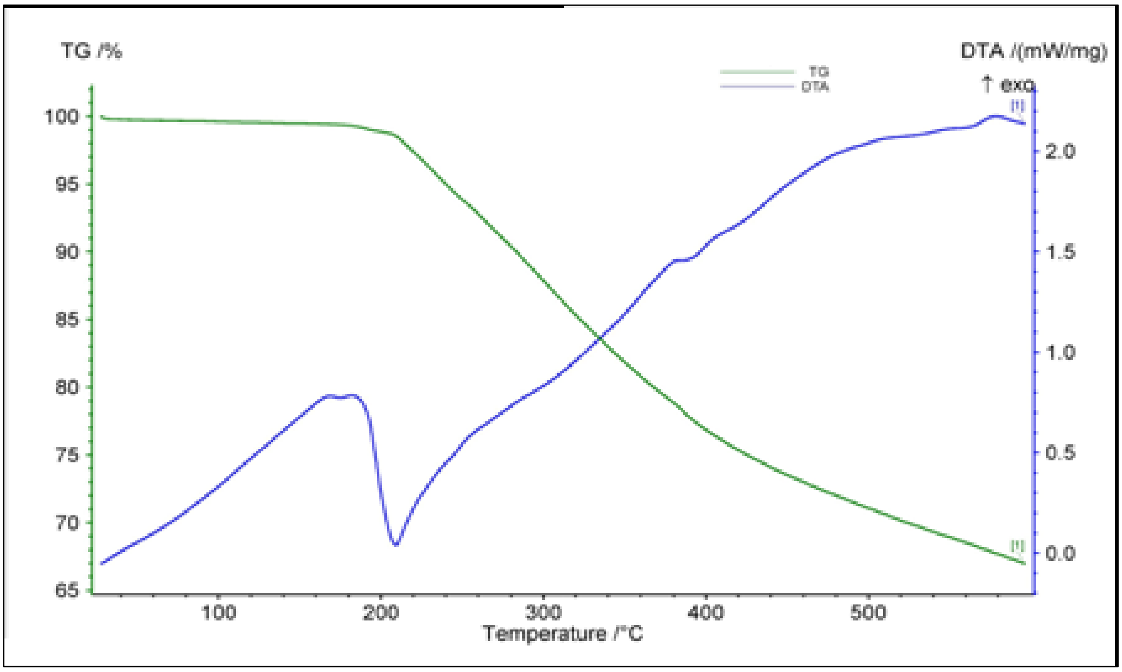

Fig. 9: *TG/DTA curves for Strontium Chloride doped ADP Crystal.*

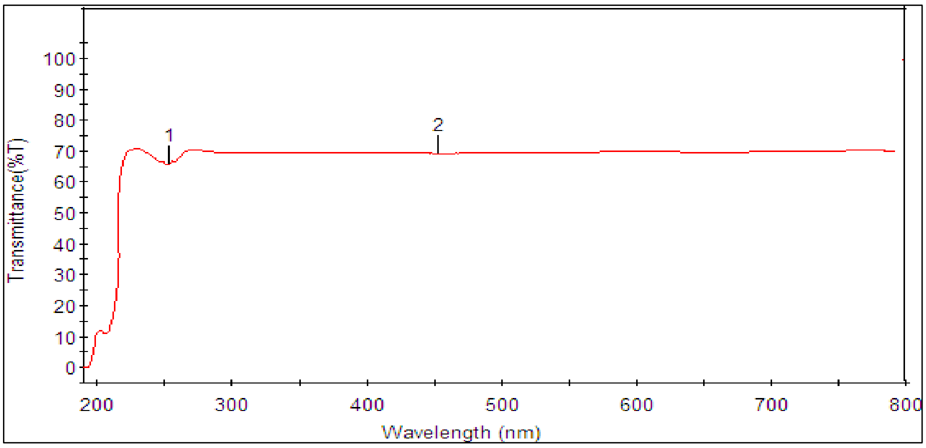

Fig. 10: *UV-visible Transmittance Spectrum of SCADP Crystal.*

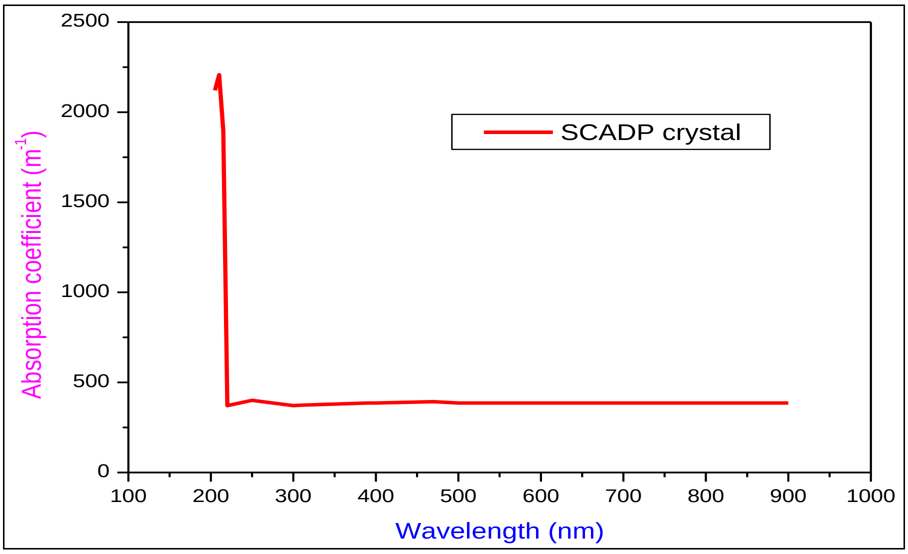

Fig. 11: *Variation of Absorption Coefficient with Wavelength for SCADP Crystal.*

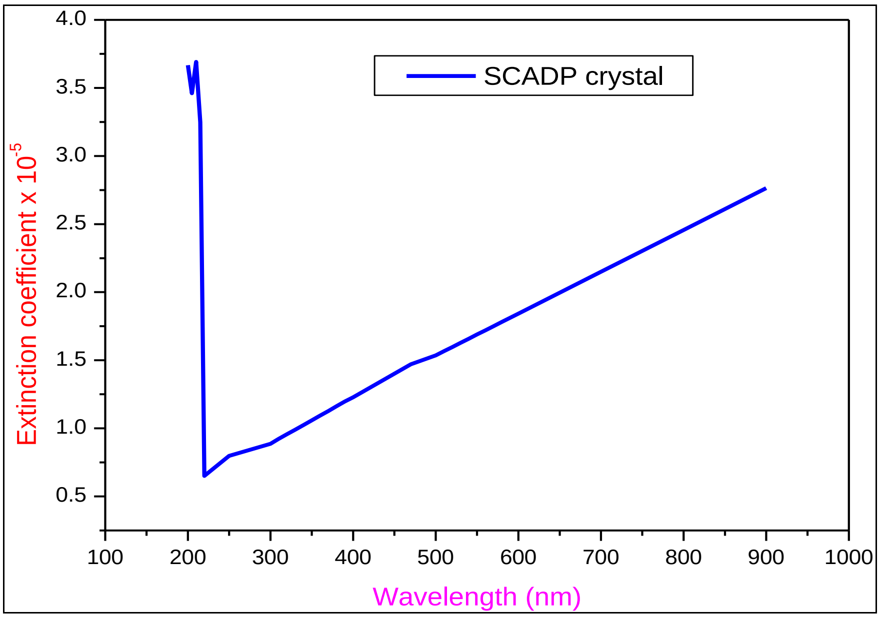

Fig. 12: *Variation of Extinction Coefficient with Wavelengthfor SCADP Crystal.*

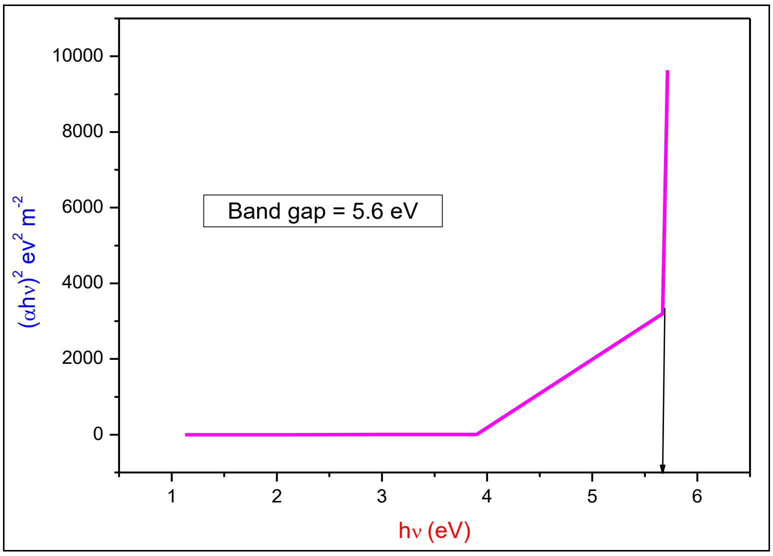

Fig. 13: *Tauc's Plot for SCADP Crystal.*

LDT Studies

Laser damage threshold (LDT) studies are important for NLO materials. As the laser is used to carry the NLO studies and NLO applications, the material must withstand the high intensity laser light. Hence, the laser damage threshold value is an important parameter in the characterization of NLO crystals. If LDT value of a crystal is low, it cannot be used for NLO applications. To find the LDT value of SCADP crystal, a Q switched Nd: YAG laser of pulse width 10 nanoseconds and repetition rate of 10Hz was used. The laser beam was focused and the sample was moved step by step into the focus along the optical axis of the crystal. The energy of the laser beam was measured by a Coherent energy/power meter (Model No. EPM 200). LDT value is determined using the formula $P = E/ \tau \pi r^2$ where E is the energy in mJ, r is the radius of the spot in mm and τ is the pulse width [10, 11]. The calculated value of LDT of the grown SCADP crystal is 0.695 GW/cm^2. Hence, the LDT value is nearly 3.47 times more than potassium dihydrogen phosphate crystal. Here it is to be mentioned that the LDT value of KDP crystal is 0.20 GW/cm^2.

Third Order NLO Parameters

It is known that third order susceptibility $\chi^{(3)}$ is responsible for third-order NLO phenomena like third harmonic generation (THG). The third order NLO parameters such as nonlinear refractive index, the nonlinear absorption coefficient and third-order nonlinear susceptibility of SCADP crystal were determined by using Z-scan technique and this technique is a simple and sensitive single beam technique for measuring the change in phase induced on a laser beam upon propagation through a nonlinear optical material. Additionally, this method is used to determine the nonlinear absorption coefficient and the nonlinear refractive index of optical materials. There are two modes in the Z-scan analysis namely, open and closed aperture modes. In the closed aperture method, an aperture is placed in front of the detector to prevent some of the light from reaching the detector. Hence, only the central region of the cone of light reaches the detector. The detector is now sensitive to any focusing or defocusing that a sample may induce. In the open aperture method, the aperture is removed to allow all the light to reach the detector and hence sets the normalized transmittance. This method is used in order to measure the nonlinear absorption which arises due to the absorption of two photons. A polished crystal of SCADP of the thickness of 0.55 mm and He-Ne laser of wavelength 632.8 nm were used in this method. Measurements of the normalized transmittance by varying the sample position (Z) have been made for the grown crystals in open and closed aperture modes. The obtained open and closed aperture Z-scan curves for strontium chloride doped ammonium dihydrogen phosphate (SCADP) crystal are shown in the Figure 14 and 15. From the results, it is observed that there is a pre-focal transmittance peak followed by a post-focal transmittance valley in the closed Z-scan curve of the sample. The transmission difference between peak and valley (ΔT_{p-v}) is written in terms of phase shift (($\Delta\phi$)and it is given by

$$\Delta T_{p-v} = 0.406\,(1-s)^{0.25}|\Delta\phi|$$

Linear transmittance aperture (S) is determined using the following relation

$$S = 1\text{-}\exp\left(\frac{-2r_a^2}{\omega_a^2}\right)$$

Where, r_a is the radius of the aperture and ω_a is the beam radius at the aperture. The third-order nonlinear refractive index (n_2) of the crystal was calculated by the following relation.

$$n_2 = \Delta\phi / (K\, I_o\, L_{eff})$$

Where, I_o is the intensity of the laser beam at the focus (Z = 0) and K = $2\pi/\lambda$ (λ is the wavelength of the laser beam).
The effective thickness can be calculated using the relation

$$L_{eff} = [1\text{-}\exp(-\alpha L)] / \alpha$$

Where, α is the linear absorption coefficient and L is the thickness of the sample. The nonlinear absorption coefficient (β) can be calculated using the following relation

$$\beta = \frac{2\sqrt{2}\Delta T}{I_0 L_{eff}}$$

Where, ΔT is the one peak value at the open aperture Z-scan curve. The value of β will be negative for saturable absorption and positive for two-photon absorption process. The real and imaginary parts of the third order nonlinear optical susceptibility ($\chi^{(3)}$) are defined as

$$\text{Real part of-}\ \chi^{(3)} = (10^{-4}\varepsilon_o\, c^2\, n_o^2\, n_2) / \pi\ \ (\text{esu})$$

$$\text{Imaginary part of-}\ \chi^{(3)} = (10^{-2}\varepsilon_o\, c^2\, n_o^2 \lambda\, \beta) / 4\pi^2\ \ (\text{esu})$$

Absolute value of $\chi^{(3)}$ = [{Real part of $\chi^{(3)}$}^{2}+ {Imaginary part of $\chi^{(3)}$}^{2}]$^{1/2}$ (esu). Here ε_o is the vacuum permittivity, n_o is the linear refractive index of the sample and c is the velocity of the light in vacuum. Using the above equations, nonlinear refractive index, third-order susceptibility and the nonlinear absorption coefficient of the crystals could be determined [12-15] and the obtained values are provided in Table 4. From the results, it is observed that the closed Z-scan curve indicates the transmittance peak followed by transmittance valley and hence third-order nonlinear refractive index is negative and this is due to the self-defocusing nature of the sample. Using the open aperture Z-scan curve, the nonlinear absorption coefficient was determined. The results of nonlinear absorption and nonlinear refraction of the sample reveal that these phenomena always coexist as they have resulted from the same physical mechanisms. The third order nonlinear parameters are useful to understand the third order NLO phenomena that are taking place in SCADP crystals.

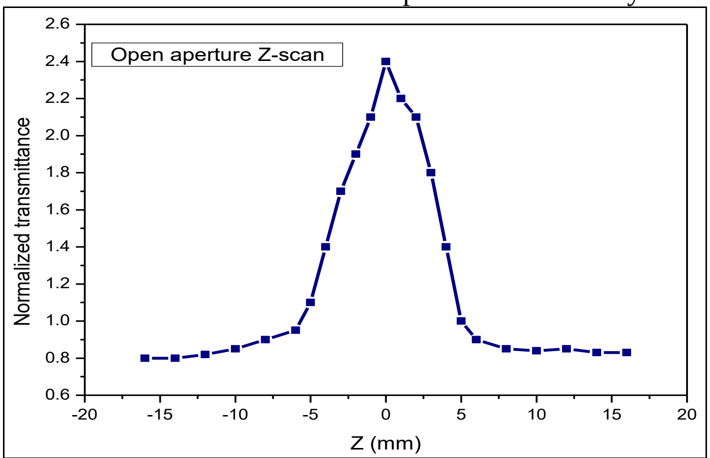

Fig. 14:*Open Z-scan for Strontium Chloride doped ADP Crystal.*

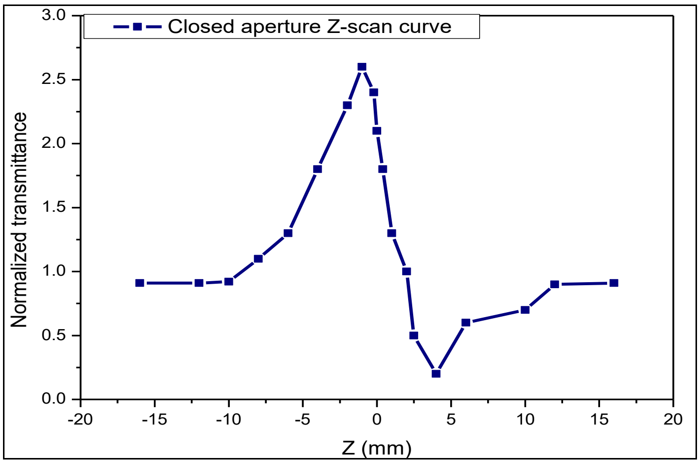

Fig. 15: *Closed Aperture Z-scan for Strontium Chloride doped ADP Crystal.*

Table 4: *Third-Order NLO Parameters for SCADP Crystal.*

Parameters	**Values**
Wavelength of laser	632.8 nm
Power of the laser	5 mW
The focal length of the convex lens used	30 mm
Nonlinear absorption coefficient □□□	3.184×10^{-4} m/W
Nonlinear refractive index (n_2)	-5.048×10^{-11} m^2/W
The real part of the nonlinear susceptibility	3.175×10^{-11} esu
The imaginary part of the nonlinear susceptibility	5.093×10^{-8} esu
The absolute value of nonlinear susceptibility	5.932×10^{-8} esu

CONCLUSIONS

Solution method with slow evaporation technique was adopted to grow the single crystals of undoped and strontium chloride doped ADP and the structure of the grown crystals was found to be tetragonal. From microhardness studies, the values of work hardening coefficient of the pure ADP and SCADP crystals were found to be 2.3992 and 2.2143, respectively. FTIR spectral studies of the samples were carried out to find the functional groups. The LDT value of SCADP crystal is found to be 0.695 GW/cm^2 and the optical band gap of the grown SCADP crystal is obtained to be 5.6 eV. The elements of the sample were found by EDAX studies. The third order NLO parameters were determined by using Z-scan studies. The melting/decomposition point of SCADP crystal is observed to be at 210°C and it is found to be more than that of undoped ADP crystal and thermal stability is enhanced when ADP crystal is doped with strontium chloride.

ACKNOWLEDGMENT

The research supports obtained from various research institutions like IIT, Chennai, Cochin University, Cochin, VIT, Vellore and St. Joseph's college, Trichy was gratefully acknowledged.

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Cite this Article
J. Velins Stanly, C. Muthukrishnan, U. Rajesh Kannan. Growth and Studies of Strontium Chloride Doped Ammonium Dihydrogen Phosphate Crystals. *Research & Reviews: Journal of Physics.* 2019; 8(2): 1–8p.

