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Studies on Structural and Optical Properties of Pure and Lanthanum Oxides Doped: L-Alanine Alaninium Nitrate (LAAN) Organic Nonlinear Optical Single Crystal

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Abstract:- Single crystals of pure and Lanthanum oxides (La_2O_3) doped L-Alanine Alaninium Nitrate (LAAN) crystals were grown by slow evaporation method. The lattice parameters were analyzed by single crystal X-ray diffraction technique. The slight changes in the lattice parameters were observed for the doped crystals compared to pure LAAN crystal. The presence of functional groups of the pure and dopant LAAN molecules were studied using Fourier Transform Infrared (FTIR) spectra. The identification of Lanthanum ion in the doped crystals was done using the energy dispersive X-Ray (EDX) spectrum. The UV-Vis transmission spectra of La_2O_3 doped LAAN showed excellent transmittance from 255 nm to 800 nm. The optical band gap energy of the grown crystals were also calculated. Improvement in the second harmonic generation (SHG) efficiency was studied by the Kurtz and Perry method. The SHG efficiency was found to be more for La_2O_3 doped LAAN comber to pure LAAN . The various studies showed the incorporation of the impurity La_2O_3 into LAAN crystal and the investigations indicated that the impurity played an important role in the changes of the properties of LAAN crystals.

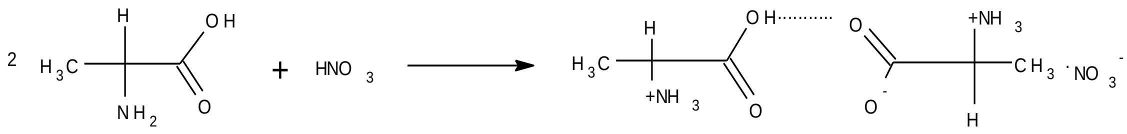
Key word: *Nonlinear optics, Organic materials, Doping effects, X-ray diffraction, Optical properties*

I. INTRODUCTION

During the last three decades, much progress has been made in the development of new and better nonlinear optical (NLO) materials having large nonlinear optical coefficients. Nonlinear optical (NLO) materials are of much importance because of its extended applications, especially to develop new laser sources [1]. An impurity ion in the crystalline lattice acts as a foreign particle, chemical composition which of differs from the main crystal morphology. In many crystallizing procedures, the presence of impurities even in the ppm range substantially affects the growth parameters and habit of crystals. Even though ion impurities exert their influences in any stage of crystal growth, their specific action induces changes in the growth rate and growth kinetics. When the impurity ion is incorporated into the crystalline lattice, decreasing the rate of crystal growth is a cause of retarded growth processing and created considerable changes in the physical parameters. Therefore selective impurities could be applied to modify the physical properties of crystals for the special purposes. Selective additives have been applied vastly in crystal growth industry in order to change the appearance shape of crystals and modify their qualities. Study of influence of impurities on the crystal growth procedure has been the subject of many researches in recent years [2-5]. The L-alanine alaninium nitrate (LAAN) belongs to the family of organic nonlinear optical material and grown from its aqueous solution by slow evaporation technique at room temperature. The characterizations of the grown single crystals were investigated by many researchers [6-10]. The L-alanine alaninium nitrate (LAAN) was first crystallized by Manuela Ramos Silva [11], which belongs to the monoclinic crystal system with noncentrosymmetric space group $P2_1$ with cell parameters of a = 7.8578 (5) (Å), b = 5.4516 (6) (Å), c = 12.8276 (7) (Å) and β = 94.73(4) $^{(\circ)}$. Thus satisfying one of the basic and essential material requirements for the SHG activity of the crystals. In the present investigation, influence of different concentration of Lanthanum oxides dopant on the structural and optical properties of L-Alanine Alaninium Nitrate (LAAN) has been studied successfully. We also compared the transmission spectra and SHG efficiency of La_2O_3 added LAAN crystal with the pure LAAN crystal.

II. EXPERIMENTAL PROCEDURE

Aqueous solution of pure LAAN were prepared by dissolving stoichometric L-alanine ($C_3H_7NO_2$) and nitric acid (HNO_3) taken in the ratio 2:1 in double distilled water. The required quantity of L-alanine and nitric acid was estimated according to the following chemical reaction:



L-alanine + nitric acid → L-alanine alaninium nitrate

The solution was heated at 50˚C until the synthesized salt of pure LAAN was obtained. The same procedure is applied to prepare the La_2O_3 doped LAAN crystals by adding different concentration (2 mol%, 4 mol% and 6 mol %) of La_2O_3 to the LAAN solution respectively. The solubility of the pure and doped LAAN in double distilled water was studied gravimetrically at different temperatures (30, 40, 50, 60 and 70°C). The solutions of pure and doped LAAN were prepared separately and kept at constant temperature for two hours with constant stirring. The homogeneous solutions were kept in the container for an hour without any disturbance. A 10 ml saturated solution of each sample was pipetted out, dried in oven and weighed to measure the dissolved solute. The same process was repeated for different temperatures range from 30 to 70°C in steps of 10°C. The solubility curves of the pure and doped LAAN crystals are shown in **Figure 1**. It can be seen that the solubility increases with temperature for pure and doped LAAN, thus the double distilled water was used as a solvent throughout the experiment. The solubility of doped crystals was found to be less than that of pure crystal. The pure and doped LAAN crystals were grown by slow evaporation technique at room temperature. In accordance with the solubility data, saturated solutions of the synthesized salts of pure and doped LAAN were prepared separately. The solution was filtered and then allowed to evaporate in a dust free atmosphere. After three to four weeks, colourless and transparent crystals were harvested. The grown crystals are shown in **Figure 2**.

III. RESULTS AND DISCUSSION

3.1. Single Crystal X-ray Diffraction Analysis

The pure and doped LAAN crystals were subjected to single crystal X-ray diffraction studies using Nonius CAD-4/MACH3 diffractometer with Mo–Kα radiation (λ= 0.71073A˚) to determine the unit cell parameters. The obtained lattice parameter values of the undoped and doped crystals are tabulated in **Table 1** and the values of pure LAAN are well matched with the reported literature [11, 12]. It was seen that the pure and doped grown crystals were crystallized in monoclinic system with the space group $P2_1$, a well known noncentrosymmetric space group, thus satisfying one the basic and essential material requirements for the SHG activity of the crystal [13,14]. The single crystal XRD study reveals that the presence of dopant has not altered the basic structure of the LAAN crystal. There are slight variations in the lattice parameters and cell volume of the doped crystals. These variations are due the lattice strain in the grown crystals due to the incorporation of La_2O_3 in the LAAN crystal lattice [1,15]

3.2. Powder X-ray Diffraction Analysis

Powder X-ray diffraction studies were carried out for the pure and doped gown crystals using a Rigaku-Miniflex diffractometer with Cu-Kα (λ= 1.5406 Å) radiation. The samples were scanned for 2θ values from 10˚ to 90˚ at a rate of 5˚ /min. **Figure 3** shows the Powder XRD pattern of the pure and doped LAAN crystals. Powder XRD spectra (**Fig. 3**) for the pure and doped crystals revealed that there are slight shift in the Bragg angle of the doped crystals compared to the pure LAAN crystal. This may be attributed to strains on the lattice by the absorption or substitution of dopant [16].

3.3 FT-IR Spectral Analysis

Fourier Transform Infrared Spectroscopy (FTIR) is an analytical technique used to identify mainly, the functional groups present in organic materials. FTIR analysis provides information about the chemical bonds and molecular structure of a material. In order to qualitatively analyze the presence of functional groups in the grown crystals, the FT-IR spectrum were recorded in the range 400-4000 cm^{-1} using FTIR-8400S spectrophotometer, SHIMADZU model under a resolution of 4 cm^{-1} and with the scanning speed of 2 mm/sec with KBr pellets method. FT-IR spectra for the pure and doped LAAN crystals are shown in **Figure. 4.** It was observed that a strong absorption occurred at 3088.14cm^{-1} corresponding to the stretching bonds of the NH^+_3 ion of the amino acid. The strong carbonyl absorption at 1234.48 cm^{-1} confirmed the asymmetric stretching of COO^- group of the compound. The frequencies with their relative intensities obtained in FTIR of pure and doped LAAN and their most probable assignments are presented in **Table 2**. The presence of additional peaks in the lower frequency region in the doped spectra may be due to the presence of Lanthanum in the coordination sphere. Although the spectrum of La^{3+} doped LAAN provides similar features as that of undoped LAAN, there is a slight shifting observed for all the peaks suggesting a wide range of interactions for the functional groups. It was also observed that there was broadening or narrowing of some absorption peaks in the FTIR spectrum of doped LAAN comber that of undoped LAAN and this may be due to the incorporation of La^{3+} in the lattice of LAAN [16, 17].

3.4. Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX) Analysis

Scanning electron microscope (SEM) analysis was carried out to investigate the morphology of compounds. The morphology of the growth surfaces were observed by a scanning electron microscopy (SEM) using S-3400N scanning electron microscope. The SEM images of pure and lanthanum oxides doped LAAN compounds are shown **in Figure 5.** It shows that both compounds are porous and agglomerated in nature [5, 16]. The SEM photos exhibit the effectiveness of the impurity in changing the surface morphology of LAAN crystal. Energy dispersive X-ray analysis (EDX) is important tool for determining the elements present in the compounds. The chemical composition of the grown crystals were observed by Quanta 200 with Genesis eds software. The recorded EDX spectra for pure and lanthanum oxides doped LAAN are shown in **Figure 6** which confirms the presence of lanthanum in the compounds[2, 5, 13, 18]. The weight percentages (wt%) of carbon (C), nitrogen (N), Oxygen (O), lanthanum (La) as obtained from EDX analysis for pure and doped crystals are presented in **Table 3**. From the experimental data, the presence of dopants in the doped crystals can be easily identified. It appears that only a small quantity is incorporated into the lattice of the LAAN crystal.

3.5. UV-Visible Spectroscopy

The UV-visible spectroscopy of the pure and doped LAAN crystals were performed by using a Varian (Cary 5000) UV–Vis spectrophotometer in the range of 200–800 nm covering the entire near ultraviolet and visible regions. The absorption and transmittance spectrums of the pure and doped crystals are shown in **Figure 7** and **Figure 8** respectively. From Figure 7, it can be seen that there is very little absorption at the wavelength of 532 nm, which can improve the second harmonic generation [16]. The pure and doped LAAN crystals have good transmittance in UV as well as in visible region (**Fig. 8**) which is an added advantage for the crystals to be used in optoelectronic applications and it is one of the additional key requirements for having efficient NLO characteristics [19,20]. In the spectrum, transmission percentage increases due to additive of Lanthanum oxide in LAAN crystal [13,16,21]. For the undoped LAAN the UV cutoff wavelength is found at 260 nm and the doped LAAN shows a slight shift in UV cutoff wavelength. This was attributed due to incorporation of the dopant and showed broad transmission in UV region. So these materials can be used in the ultraviolet region for the device applications [17]. The optical energy gap of the pure and doped grown crystals is determined from the transmittance spectrum using the equation;

$$\alpha h\nu = N(h\nu - E_g)^m$$

where N is a constant which varies with transitions, E_g is the band gap of the material and m is an index which can have the values 1/2, 3/2, 2, or 3 depending on the nature of the electronic transitions. Here, the value of m is assigned as 1/2 for an allowed direct transition and α is the absorption coefficient, which is calculated from the equation;

$$\alpha = \frac{Abs}{t}$$

where Abs is the absorbance of the material and t is the thickness of the sample. From the transmittance spectrum, a graph is drawn between $h\nu$ and $(\alpha h\nu)^2$ and is displayed in **Figure 9.** The band gap energy of pure and doped crystals is evaluated by extrapolating a straight line in the linear region of the graph at $(\alpha h\nu)^2 = 0$. The UV transparency cut-off wavelength and band gap energies for the pure and doped LAAN crystals are tabulated in **Table 4**.

3.6. SHG Efficiency Measurement

The SHG conversion efficiency of pure and doped LAAN crystals were estimated using modified setup of Kurtz and Perry method [22] was employed. In this experiment Q-switched, mode locked Nd:YAG laser of wavelength 1064 nm having pulse energy 2.15 mJ, pulse duration 10 ns and repetition rate 10 Hz was used. The pure and doped LAAN crystals with 2, 4 and 6mole% Lanthanum oxide doped LAAN were powdered with a uniform particle size and then packed in a micro capillary of uniform bore and exposed to laser radiations. The output from the sample was monochromated to collect the intensity of 532 nm component, and to eliminate the fundamental wavelength. Second harmonic radiation generated by the randomly oriented micro crystals was focused by a lens and detected by a photomultiplier tube. The generation of second harmonic was confirmed by the emission of green light. The optical signal generated from sample is converted into electrical signal and was measured on oscilloscope. The output was measured at 532 nm wavelength. The SHG efficiency of pure and doped crystals was estimated with respect to standard potassium dihydrogen phosphate (KDP) and is given in **Table 5**. From **Table 5**, the SHG efficiency was found to be increased with the concentration of the lanthanum oxides [18, 23,24]. Due to the presence of dopant in the crystal lattice, there is an increase in polarizability of the molecule, which tends to increase the SHG efficiency. The increased SHG efficiency can be taken as better candidate for NLO applications [25, 26] .

IV. CONCLUSION

Good quality single crystals of pure and Lanthanum oxide doped L-Alanine Alaninium Nitrate (LAAN) crystals were grown successfully by slow evaporation technique. Single crystal X-ray diffraction studies were carried out, and the lattice parameters are calculated. FTIR spectrum determines the various functional groups present in the compounds. The shifting in bands gives indirect evidence for doping of La_2O_3 in LAAN . The scanning electron microscope pictures show the porous and agglomerated in La_2O_3 added LAAN crystals. The presence of La was confirmed by EDX analysis. The transmission spectrum reveals that the crystal has sufficient transmission in the entire visible and UV region. Also this spectrum reveals that lower UV cut-off wavelength for La_2O_3 doped LAAN is lower than pure compound which indicates material has good optical transmittance in entire visible region. The presence of dopant has improved the nonlinear optical (NLO) properties of the grown crystals and these crystals can be promising material for nonlinear device fabrication.

Figures:

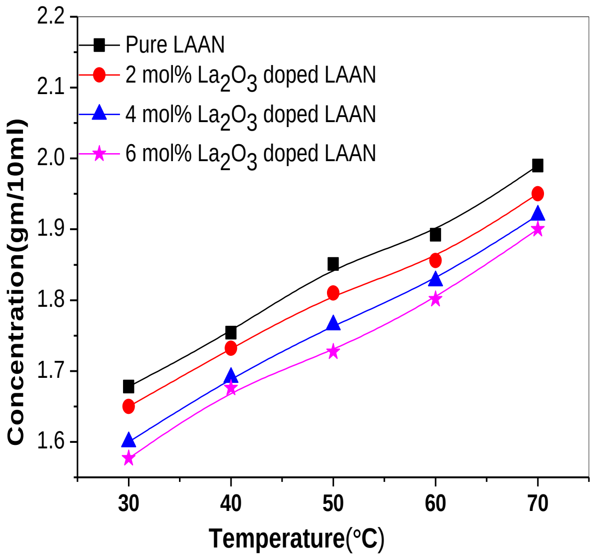
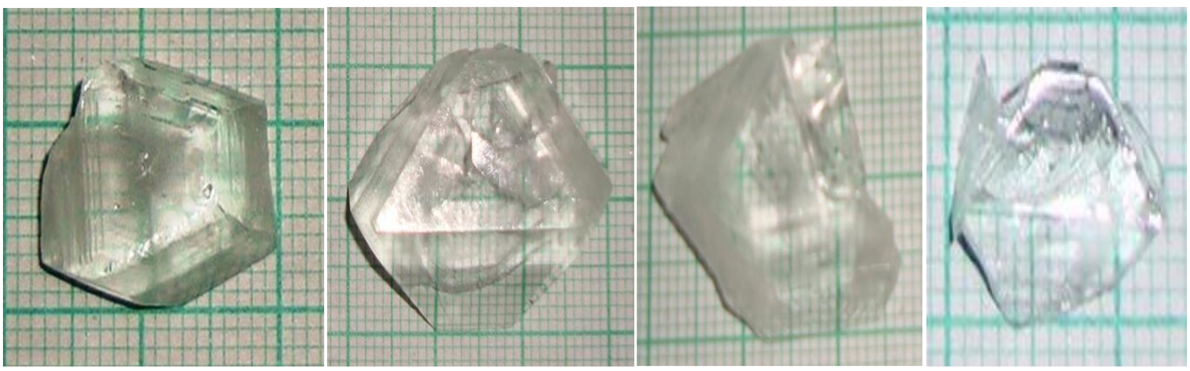


Fig.1. Solubility curves of pure and lanthanum oxides doped LAAN crystals



Pure LAAN	2 mol% La_2O_3 doped LAAN	4 mol% La_2O_3 doped LAAN	6mol% La_2O_3 doped LAAN

'Fig.2. Photographs of the as grown pure and lanthanum oxides doped LAAN crystals

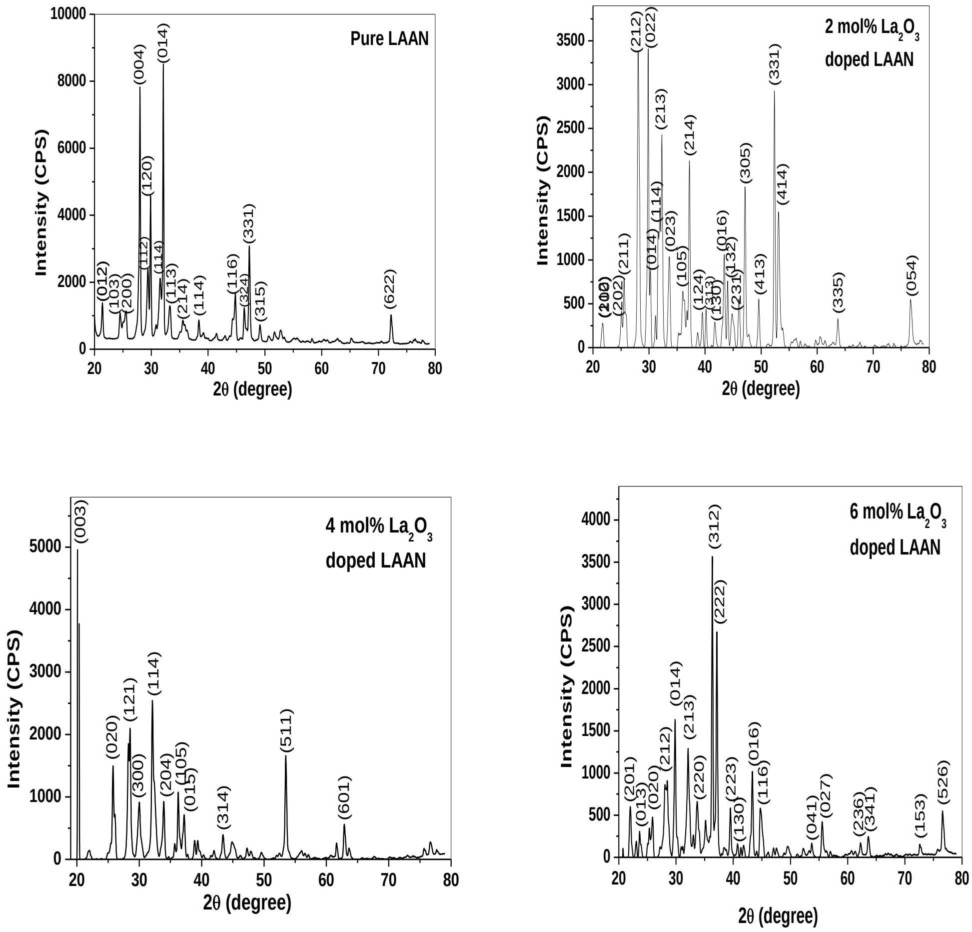
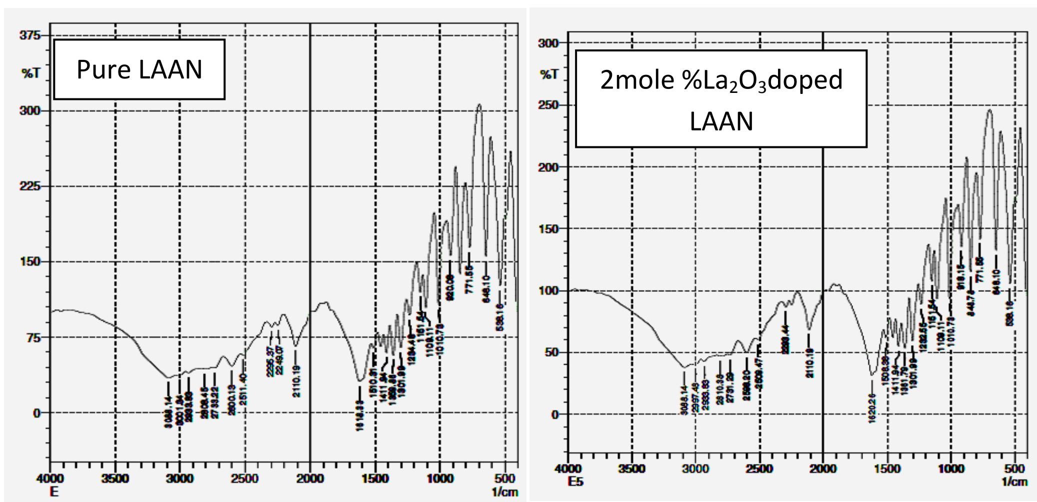


Fig. 3. The Powder X-ray diffraction patterns of pure and lanthanum oxides doped LAAN crystals



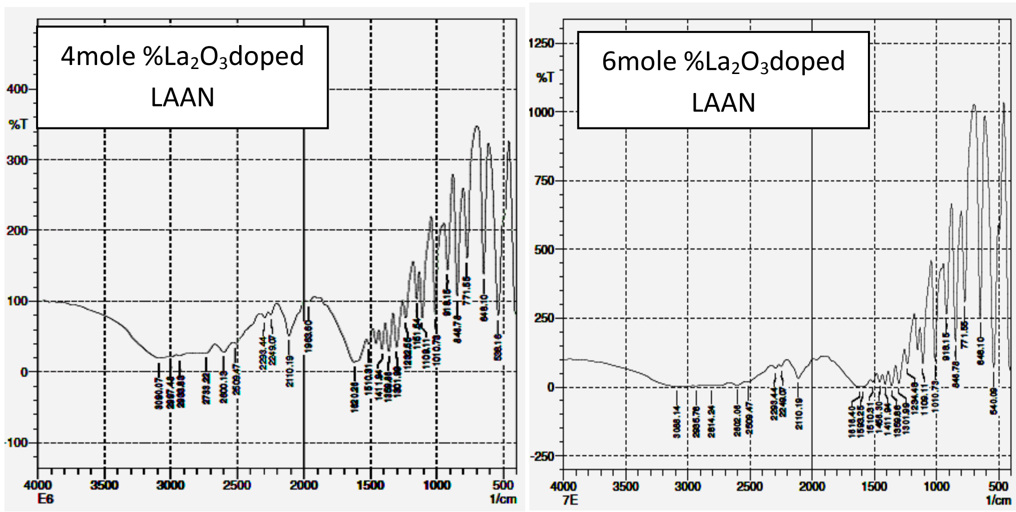


Fig. 4. FT-IR spectrum of a pure and lanthanum oxides doped LAAN crystals

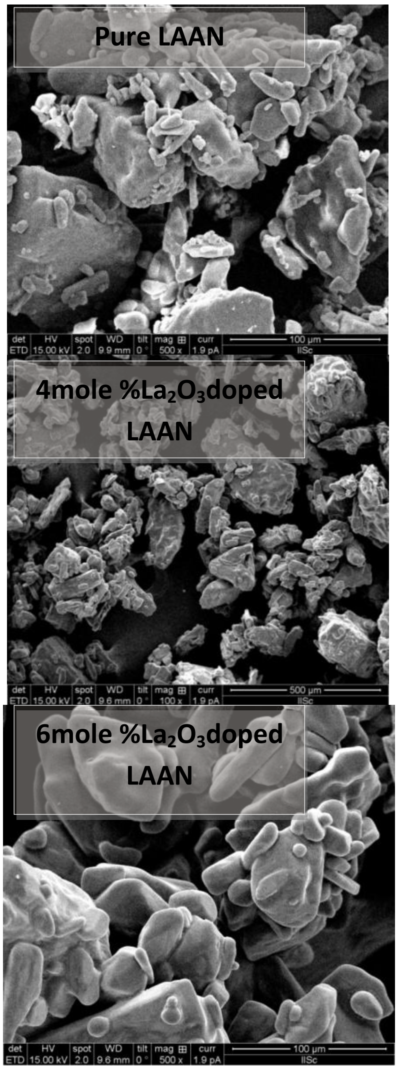


Fig. 5 . SEM images of pure and lanthanum oxides doped LAAN crystals

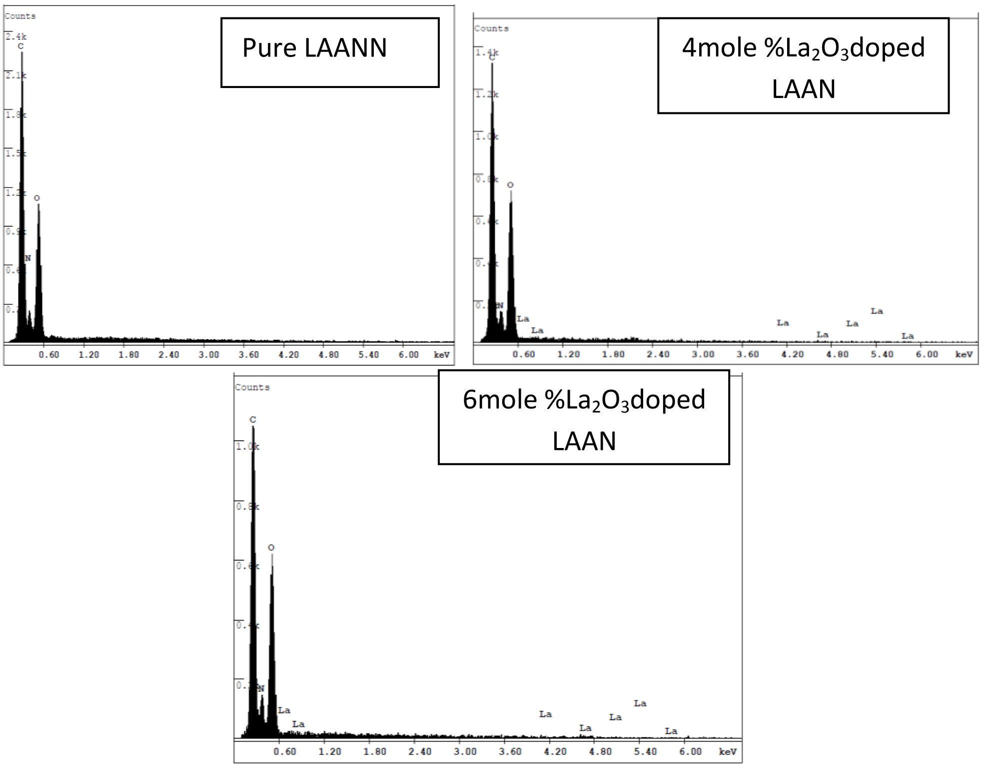


Fig. 6. EDX **of** pure and lanthanum oxides doped LAAN crystals

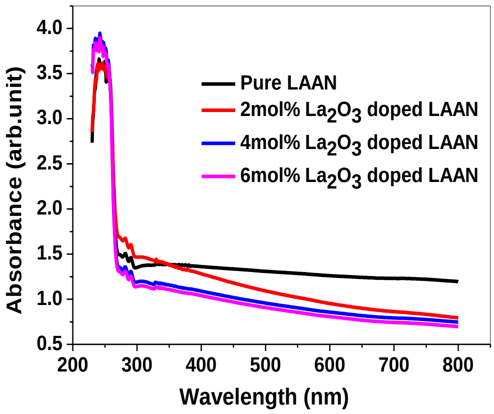


Fig.7. The absorption spectrums of pure and lanthanum oxides doped LAAN crystals

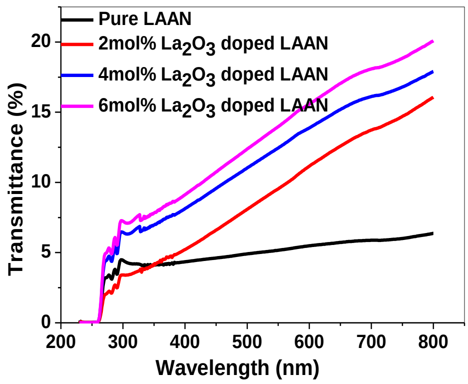


Fig 8. The transmission spectrums of pure and lanthanum oxides doped LAAN crystals

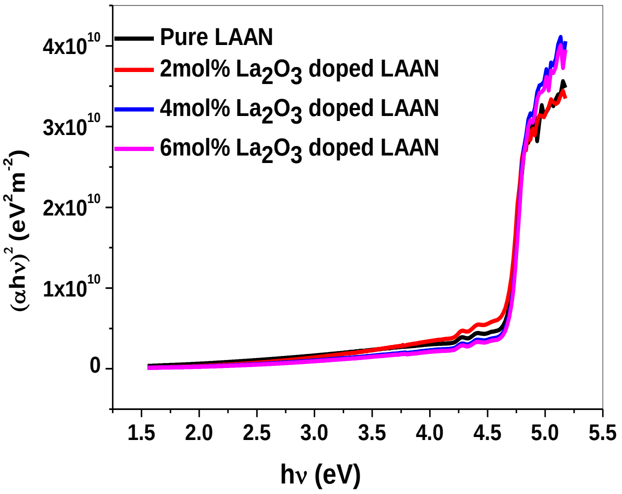


Fig9. $(\alpha h\nu)^2$ vs photon energy (hν) plot of pure and lanthanum oxides doped LAAN crystals

Table 1. Unit cell parameters of pure and lanthanum oxides doped LAAN crystals

Crystals	a (Å)	b (Å)	c (Å)	β (°)	Volume ($Å^3$)	**Crystal system**	**Space group**
Pure LAAN	8.034	6.092	12.908	94.73	629.606	Monoclinic	P21
2 mol% La_2O_3 doped LAAN	8.523	6.723	13.062	94.65	745.990	Monoclinic	P21
4 mol% La_2O_3 doped LAAN	8.912	6.923	12.935	94.73	795.343	Monoclinic	P21
6 mol% La_2O_3 doped LAAN	8.842	6.902	13.162	95.89	799.003	Monoclinic	P21

Table 2. FT-IR frequency assignments of pure and doped LAAN crystals

Pure LAAN $\{cm^{-1}\}$	2mole%	4mole%	6mole%	Assignments
	La_2O_3 doped LAAN $\{cm^{-1}\}$			
3088.14	3088.14	3090.07	3088.14	NH stretch of NH_3
2933.83	2933.83	2933.83	2935.76	CH_2 asymmetric stretch
2600.13	2598.20	2600.13	2602.08	CH_2 asymmetric stretch
2110.19	2110.19	2110.19	2110.19	Combination band of NH_3^+ degenerate mode and of NH_3^+ torsion
1618.33	1620.26	1620.26	1616.40	asymmetric NH_3 vibration
1510.31	1503.38	1510.31	1510.31	NH_3^+ sym. stretch
1411.94	1411.34	1411.94	1411.94	ν (COO)
1359.86	1361.79	1359.36	1359.86	CH_3 bending and COO^- symmetric vibrations
1301.99	1301.99	1301.99	1301.99	presence of NO_3^-
1234.48	1232.55	1232.55	1234.48	COO^- symmetric stretch
1151.54	1151.54	1151.54		COO^- sym. str. ; NH_3^+ rock
1109.11	1109.11	1109.11	1109.11	COO^- vibrations
1010.73	1010.73	1010.73	1010.73	C-N stretching stretch
920.08	918.15	918.15	918.15	C-C-N symmetric stretch
771.55	771.55	771.55	771.55	ϕ NO2
648.10	648.10	648.78	648.10	C-O in plane deformation
538.16	538.16	538.16	540.09	δ ring

Table 3. EDX quantification data of pure and lanthanum oxides doped LAAN crystals

Pure LAAN	Element	C	N	O			Total
	Wt (%)	48.45	15.35	36.20			100.00
	Atomic%	54.56	14.83	30.61			100.00
4% mole of Lanthanum oxide doped LAAN	Element	C	N	O	La		
	Wt (%)	45.57	15.40	38.01	1.02		100.00
	Atomic%	52.14	15.11	32.65	0.10		100.00
6% mole of Lanthanum oxide doped LAAN	Element	C	N	O	La		
	Wt (%)	45.25	15.66	37.17	1.91		100.00
	Atomic%	52.16	15.48	32.16	0.19		100.00

Table: 4. The UV transparency cut-off wavelength and band gap energies of the pure and lanthanum oxides dope LAAN crystals

Crystals	Cut-off wavelength (nm)	Band gap energy (eV)
Pure LAAN	260	4.62
2 mol% La_2O_3 doped LAAN	256	4.62
4 mol% La_2O_3 doped LAAN	257	4.63
6 mol% La_2O_3 doped LAAN	258	4.63

Table 5. SHG efficiency of the pure and doped LAAN crystals

Crystals	SHG signal (mV)	Efficiency with respect to KDP (11.5 mV)
Pure LAAN	6.6	0.57
2 mol% La_2O_3 doped LAAN	7.1	0.62
4 mol% La_2O_3 doped LAAN	8.3	0.72
6 mol% La_2O_3 doped LAAN	9.0	0.78

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