Investigation on the physico-chemical properties of Sodium L-Alanine Cadmium chloride crystal and assessment for their Non Linear Optical response

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Abstract

Non linear optical crystals L-Alanine Cadmium Chloride (LACC) and sodium admixed LACC have been grown as semi-organic single crystals using the slow evaporation crystal growth process at pH 5.0. Densities of the crystals are measured using Flotation method. The host lattice of LACC has sodium compounds, according to the Atomic Absorption Spectroscopy (AAS) technique and Energy Dispersive X-ray (EDAX) Analysis. From the Single Crystal X-ray Diffraction (SCXRD) studies, the crystalline structure and associated crystallographic axes were calculated. To distinguish between the various functional groups present Fourier transform infrared (FTIR) spectroscopic method is applied. Thermo gravimetric analysis determines the thermal properties of the pure and sodium admixed LACC crystal. The micro hardness test is used to determine the nature of the formed crystals. Absorbance percentage of sodium mixed LACC crystals were recorded using Ultra - Violet Spectrophotometer instrument and the calculated optical band gap confirms that the crystals are insulators. Using Nd-YAG LASER source, non linear optical phenomena of the prepared crystals was confirmed and sodium doped LACC crystals shows higher Second Harmonic Generation (SHG) efficiency than the pure LACC crystal. Dielectric properties were reported using the dielectric studies and found that the AC activation energy is higher at 100 Hz.

Keywords

L-Alanine Cadmium choride; Activation energy; Density; Micro Hardness.

INTRODUCTION

In modern days the growth of semi organic crystals with nonlinear optical properties are mostly utilized in the research areas like optical data storage devices, optical signal processing technique, optical switching purpose etc., [1]. An amino acid crystal has wide NLO properties due to the presence of carboxyl group which act as a proton donators and amino group as proton acceptors. Sodium atom is an ionic compound having only one electron in the valance shell [2]. Because of its unique properties of low atomic mass and large atomic radius, sodium has least dense compared to all elemental metals which act as a best insulators and electrides [3]. Sodium compounds have immense commercial industry

applications such as glass, paper, soap and textiles. Sodium is an electrolyte used for de-icing, anti-icing, as food preservatives, bioavailability and which plays important role in nerve pulse and muscle contractions [4]. Sodium forms different compounds with metals like group 11 and 12 elements, different compounds are produced because of its higher polarity of the C-Na bonds. L-Alanine is the simplest and neutral amino acid which exists as zwitterions; it possesses high transparency with favorable physic chemical properties [5].

The pure compound namely (LACC) was initially crystallized by the team Kathleen et al. [6] and latter the crystals optical phenomena's are reported by the team Dhanuskodi et al. [7]. Further the optical properties of the parent compound were reported by Kalaiselvi et al. [8]. The properties of the parent compound have been altered by adding the impurities. The improved dielectric properties are obtained when potassium is doped with the main compound reported by K.C. Bright et al. [9]. The magnetic properties of the main compounds are changed by adding impurities such as cobalt and nickel [10, 11].The improved linear optical properties when copper is incorporated in the lattice of L-Alanine cadmium chloride [12]. Earlier we have reported the effect of strontium on the LACC crystal [13]. In extension this paper we presented the brief account of influence of sodium admixture on the growth process, structural, thermal, mechanical and optical phenomena of LACC crystals suitable for NLO applications.

EXPERIMENTAL METHODS

In this present work, the simple slow evaporation crystal growth technique is utilized for the growth of semi organic L-Alanine cadmium chloride (LACC) and sodium ions doped LACC crystals. For the experimental growth process, AR grade Cadmium Chloride Monohydrate, L-Alanine, Sodium chloride and doubly ionized water was used. The equimolar ratio of calculated amount of L-Alanine amino acid and metal cadmium chloride is completely dissolved with the solvent and stirred well for about 2 hours. Then the homogeneous mixture was heated about at temperature of 333 K. In order to suppress the possible decomposition, the samples were dried completely then dissolved with the water to make saturated solutions. Saturated mixture solution was allowed to crystallize at pH of 5.0 in the normal atmospheric pressure about 303 K for four weeks. The single crystals of 12 x 7 x 2 mm^3 dimension with good transparency were collected.

For sodium chloride doped LACC crystallization, in addition to the equimolar quantities of L-Alanine cadmium chloride, 0.02 and 0.04 mole of sodium chloride is added as a dopant. Same procedure is used for the growth of sodium doped LACC crystals. The single crystals of 12x 11 x 3 mm^3 (2 mole %) and 15 x 14 x 4 mm^3 (4 mole %) dimension with good transparency were collected within five weeks are shown in the Fig.1.

Fig.1 Grown LACC and Na ions doped LACC crystals.

RESLUT AND DISCUSSION

Densities of LACC and the sodium admixed LACC crystals were accurately calculated by floatation method since the method gives precise measurement. Flotation

density measurement technique is highly responsive to the point defects and not sensitive to the dislocation of the grown LACC crystal. The measured density values of LACC is 2.139 g/cc and 2, 4 mole percentage of Na^+ ions doped LACC crystals are 2.175 and 2.176 g/cc respectively. Even though sodium is a least dense metal, the density measurement confirms that due to doping the density of the crystal increases.

Atomic Absorption Spectroscopy (AAS) is considered as an important technique to find the concentration of impurity atoms in the sodium doped crystals. An AAS result reveals the concentration of Na^+ ions in 2, 4 mole percentage of doped LACC crystals are 1427 ppm and 1479 ppm respectively. Moreover the AAS result confirms that as the mole percentage of the sodium increases the impurity concentration also increases.

Elemental identifications were carried out in addition to AAS to confirm the presence of impurities and the corresponding EDAX spectra of LACC and Na^+ incorporated LACC crystals are shown in Fig.2,3 and the corresponding elemental compositions are given in tables 1and 2 respectively.

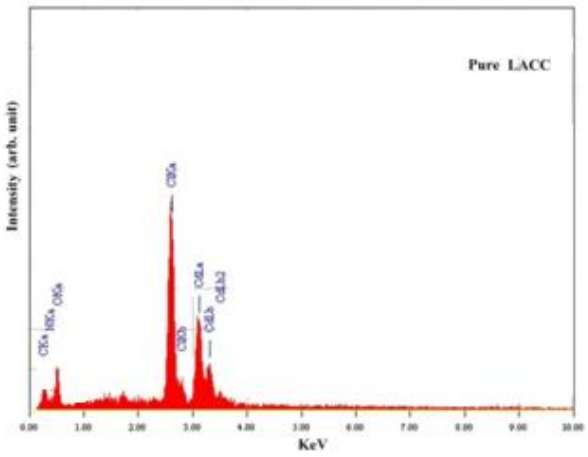

Fig.2. EDAX spectra of LACC crystal.

Table.1 Elements present in pure LACC crystal.

Elements Present	Energy (keV)	Mass (%)	Atom (%)	K
C K	0.277	23.37	36.22	6.6274
N K	0.392	8.72	11.58	10.4883
O K	0.525	35.94	41.81	24.3768
Cl K	2.621	14.18	7.44	26.1827
Cd L	3.132	17.8	2.95	24.0775
Total		100	100	

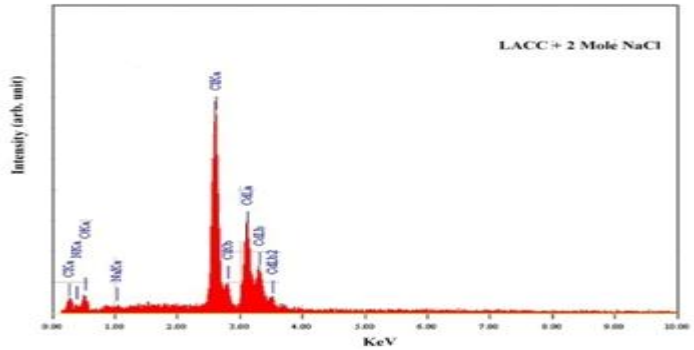

Fig.3. EDAX spectra of 2 mole % NaCl admixed LACC crystal.

Table.2 Elements present in 2 mole % NaCl admixed LACC.

Elements Present	Energy (KeV)	Mass (%)	Atom (%)	K
C K	0.277	23.56	39.64	5.5592
N K	0.392	9.63	13.89	10.3745
O K	0.525	25.27	31.91	14.11
Na K	1.041	0.21	0.18	0.1906
Cl K	2.621	17.79	10.14	32.2413
Cd L	3.132	23.55	4.23	30.6041
Total		100	100	

EDAX results very clearly confirmed the presence of sodium on the LACC crystalline lattice. Mass percentage of 2 mole % sodium doped LACC was 0.21 furthermore it was identified that the ionic radii of the sodium decreases with increasing the mass percentage of metals in LACC compound. Thus greater ionic radii of sodium easily enter into the lattice site of the host materials.

The structural properties of grown LACC and sodium admixed LACC crystals were analyzed using computer interfaced Enraf Nonius SCXRD. Using the Molybdenum-K alpha radiation of wavelength λ= 0.71073 Å, computed unit cell parameters of pure LACC crystals are a = 16.331 Å, b = 07.323 Å, c = 07.987 Å, α =90°, γ = 90°, β = 116.45° and volume V=845.79 Å^3. For sodium admixed LACC crystals, the computed unit cell parameters are calculated a = 16.643 Å, b = 07.313 Å, c = 07.998 Å, α =90°, γ = 90°, β = 116.58° and volume V=862.35 Å^3. Both the pure and sodium incorporated LACC crystals belongs to monoclinic crystal system, C_2 space group and effective number of atoms is 4.

Phase identification of the power LACC crystal was performed using Philips X'pert Pro PXRD interfaced with X'pert High score software. Copper K-alpha radiation with a tube voltage 40 kV and current 30 mA was supplied for the continuous scanning in the 2θ range between 10 to 50^0 in 0.01 degree / step. Fig.4 depicts the powder XRD spectrum of LACC and sodium doped LACC crystals.

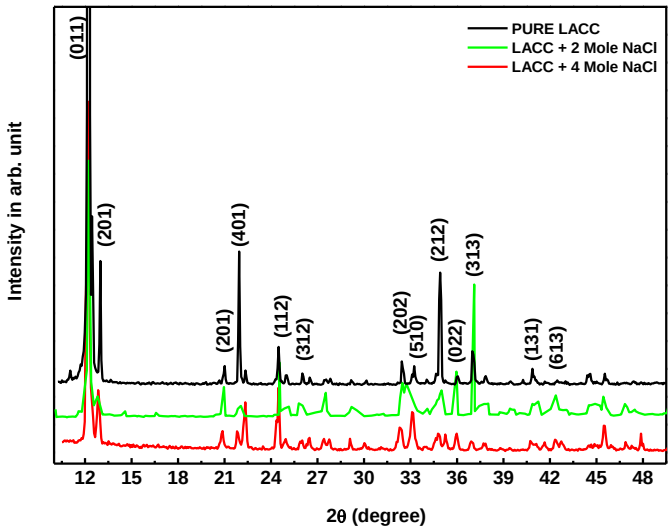

Fig.4 Powder XRD pattern.

Powder XRD spectrum confirms the crystalline nature of the powder sample. For monoclinic crystal system the inter planar distance (d) can be calculated form the following relation [14],

$$\frac{1}{d^2} = \frac{1}{\sin^2\beta}\left(\frac{h^2}{a^2}+\frac{k^2\sin^2\beta}{b^2}+\frac{l^2}{c^2}-2hl\frac{\cos\beta}{ac}\right) \quad (1)$$

The lattice constants a, b and c values are deduced from the powder XRD data and the cell volume is calculated using the relation [14],

$$V = a\,b\,c\sin\beta. \quad (2)$$

The calculated lattice parameters correlated with the single crystal XRD result and the calculated values are depicted in table.3.

Table.3 Calculated lattice parameters of crystals.

Crystal	Cell parameters	Cell volume
Pure LCC	a = 16.28 Å, b = 07.27 Å, c = 07.97 Å α = γ = 90°, β = 116.38°	843.79 Å^3
LACC + 2 mole % NaCl	a = 16.314 Å, b = 07.315 Å, c = 08.038 Å α = γ = 90°, β = 116.48°	856.27 Å^3
LACC + 4 mole % NaCl	a = 16.334 Å, b = 07.320 Å, c = 07.978 Å α = γ = 90°, β = 116.57°	853.14 Å^3

Fig.5 and 6 gives the Fourier transform infra red spectrum of pure and two mole percentage sodium doped LACC recorded using FTIR spectrophotometer. The spectrum was recorded in the IR range using potassium bromide pellet technique. L-Alanine amino acid consist of amine group, it is protonated due to the carboxyl group and gives NH^{3+} amine and COO^- carboxyl groups. Broad bands in the wave number ranges such as 3044–3250 cm^{-1}, 3425–3500 cm^{-1} in LACC and 3039–3225, 3425–3570 in sodium admixed LACC is due to NH^{3+} symmetric and asymmetric stretching vibrations. Strong absorption peak around 1615

cm^{-1} in both the spectrum is assigned due to NH^{3+} bending mode of vibration [15]. Absorption of IR radiation in the region of 2500 to 3050 cm^{-1} in both the spectrum is assigned as the combinations of multiple overtone bands. The absorption peak at 1410 cm^{-1} is assigned due to carboxyl molecules symmetric stretching [16]. The COO^- bending and rocking vibrations are assigned in the positions at 762 and 536 cm^{-1} respectively. Moreover the absorption at 1343 and 1001 cm^{-1} are identified due to CH_3 symmetric bending and rocking mode in the amino group respectively. The absorption peaks around 850 to 900 cm^{-1} are assigned due to the C–C–N symmetric stretching vibrations. FTIR spectrum concludes that the vibrations experienced in both the crystals are same but there is a slight shift, broadening and narrowing of absorption peaks in sodium ions doped LACC crystals compared to pure LACC crystal, which is mainly due to the addition sodium into host LACC crystalline compound.

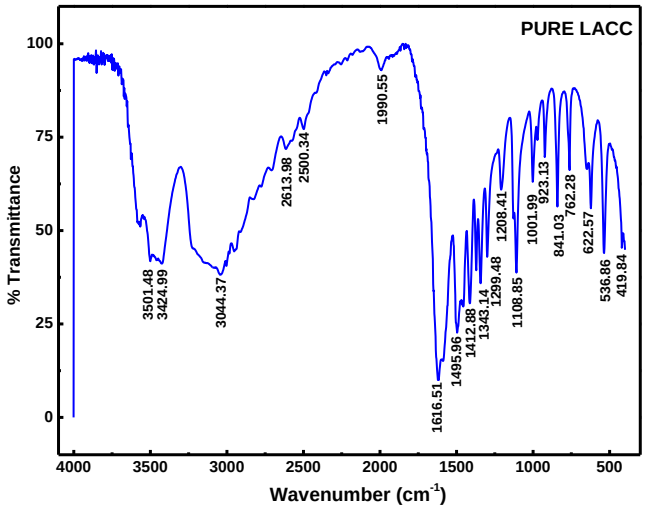

Fig.5 Recorded IR spectrum of pure LACC.

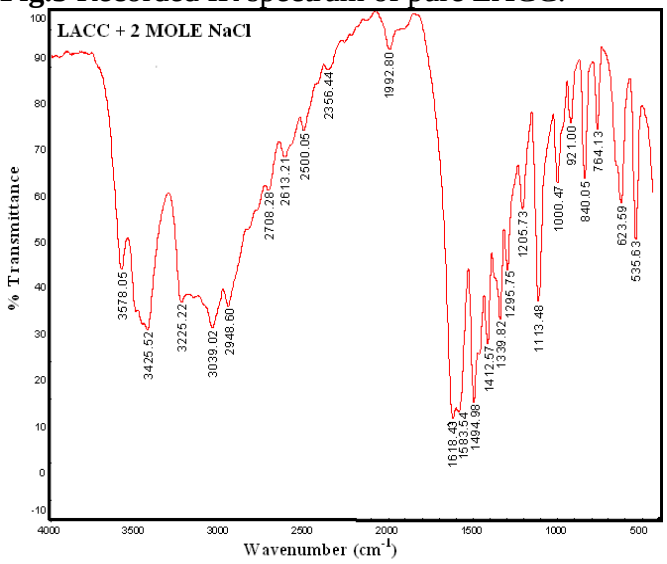

Fig.6 Recorded IR spectrum of sodium doped LACC.

The SEM images of pure LACC and 2 mole percentages of sodium ions doped LACC crystals have been obtained with JEOL-JSM model SEM. The corresponding microscopic images are depicted in Fig 7-8. From the images it was observed that the porosity decreases,

grain size and alignment improved by sodium doped as compared with LACC and the improved grain size, porosity and alignment may arise due to the assemblage of trace metal grains in the region of the sodium doped atoms.

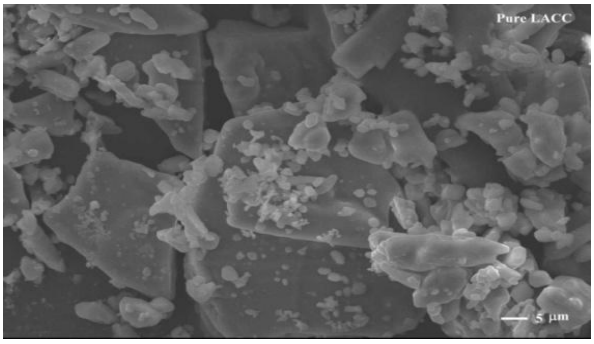

Fig.7 Microscopic image of LACC.

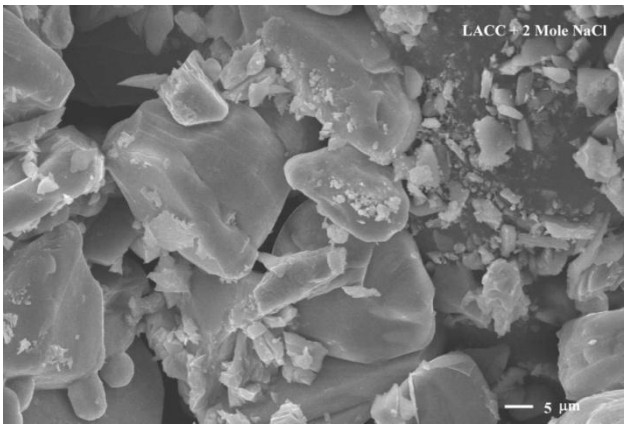

Fig.8 Microscopic image of 2 mole % NaCl doped LACC.

The TG/DTA analysis was performed with a heating rate of 283 K / min in the nitrogen atmosphere. The Thermo graphs of LACC and Na^+ admixed LACC crystals were given in Fig.9 and 10 respectively.

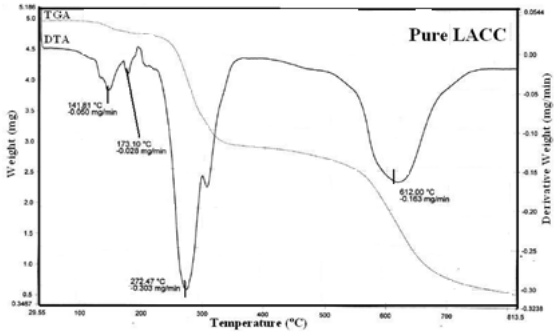

Fig. 9 TGA/DTA graphs of pure LACC

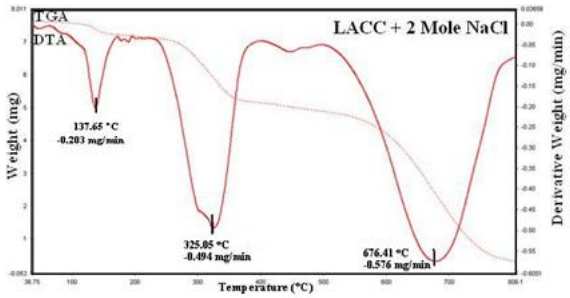

Fig. 10 TGA/DTA graphs of NaCl doped LACC.

From the pure TG/DTA curve, it was confirmed there is no loss of weight experienced up to the temperature 115°C hence it was clearly identified that LACC is thermally more stable up to this corresponding temperature. A strong endothermic peak at the temperature 272.47°C is due to the degradation of amino acid present in the parent compound. The gradual weight loss in the temperature range 112.5 – 140.3°C and 320.4 – 550.3°C. The major degradation of the LACC compound at 612.5°C is due to the liberation of various molecules like CO, CO_2, NH_3 and Cl_2. Furthermore TG/DTA curve of sodium doped LACC crystal the endothermic peaks obtained at 137.65°C, 325.05° C and 676.41°C corresponds to the liberation of OH, CO, CO_2, NH_3 and Chlorine molecules present in the dopant crystal. It was also proved that the sodium chloride admixed LACC crystals are thermally more stable compared to undoped LACC crystals. Thus the sodium doping enhances the thermal stability of the parent crystal. The final residue left at the end beyond heating 800°C is the metal cadmium.

Mechanical properties of LACC and sodium doped LACC crystals are studied using a Leitz microhardness tester. Both the prepared crystals were subjected to the load in the range of 0.025 to 0.100 kg in the particular face (101) plane. For each load, the average diagonal length (d) is calculated. The micro hardness number (H_v) is determined using the relation as;

$$H_v = \frac{1.8544\ P}{d^2}\ \text{kg mm}^{-2} \quad (3)$$

Where P and d are the applied load and diagonal length respectively. The plot of calculated Vickers micro hardness number and the load applied is shown in Fig. 11. The plot proves that the micro hardness number of both pure LACC and doped LACC crystals increases with increasing load. Again, in the sodium doped crystals the micro hardness number is less than that of pure LACC and in this case the doping concentration has not much affect the micro hardness number this may be increased by increasing the doping concentration. The variations in the micro hardness number are due the addition of the sodium impurities into the crystal lattice of LACC crystals.

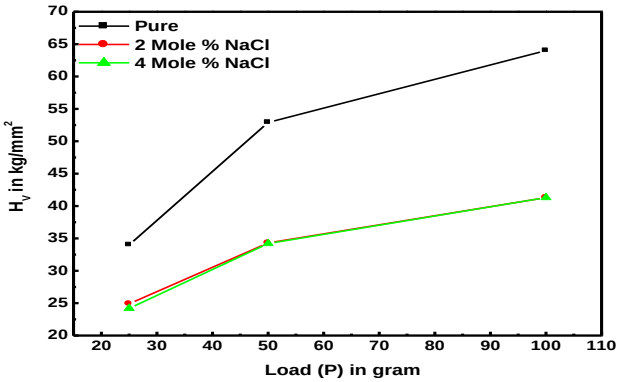

Fig. 11 Variation of micro hardness number with applied load

Work hardening coefficients are determined from the following Mayer's relation,

$$P = kd^n \quad (4)$$

Here k is proportionality constant and Meyer's index number (n) otherwise called as work hardening coefficient. The work hardening coefficients are calculated from the slope of the Fig. 12. The obtained value of n for the grown crystals was estimated using least square fit method. The value of n for pure LACC is 3.5466 and for two and four mole percentages of sodium doped LACC crystals were 3.1253 and 3.2185 respectively. Therefore the calculated n values are greater than 1.6. Based on Onitsch [17] and Hanneman, the value of n is greater than 1.6 all the prepared material belongs to the category of soft materials. Hence it was concluded that the pure LACC and doped LACC crystals belong to the category of soft material.

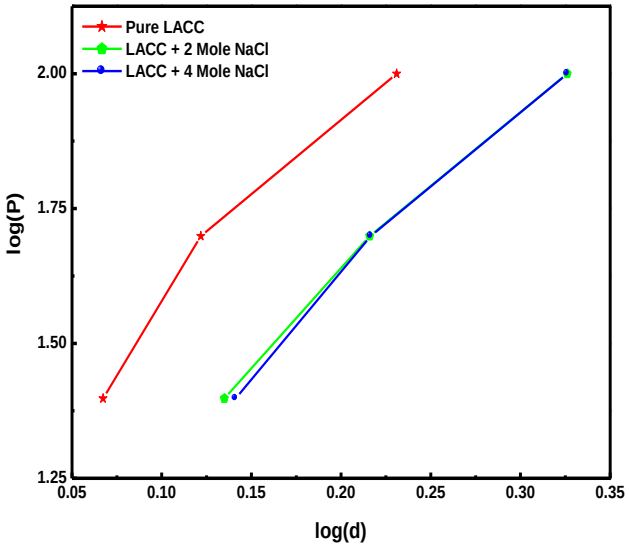

Fig.12 Mayer's plot for un doped and NaCl doped LACC crystals.

The optical absorbance spectrum of sodium doped LACC crystals were recorded using UV-Vis spectrophotometer in the UV-Vis region is shown in Fig.13.

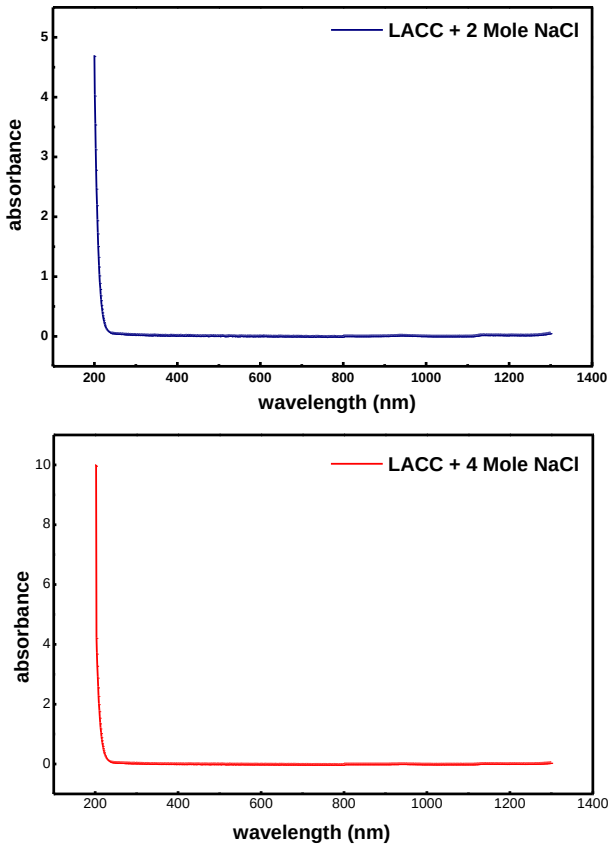

Fig. 13 Absorbance spectra of NaCl doped LACC crystals.

The UV spectrum confirms that there was no significant absorption in the entire UV-Visible region. No such absorption arises because of the presence of amino acids in the parent compounds where the conjugated bonds are absent. Lower absorbance percentage is due to the optical energy transition from 3p to 3s orbital's. For the pure and sodium admixed LACC, the cut off wavelength experienced around 200nm enhances the optical transparency which makes LACC for optoelectronic applications again it is considered as essential parameter for NLO applications. Compared to pure LACC [7] there is small variation arises in cut-off wavelength of sodium doped LACC crystals. Hence sodium dopant changes the optical transmittance and absorbance percentage. Optical band gaps (E_g) were calculated using the following relation;

$$E_g = hc/\lambda \quad (5)$$

Where h Plank's constant, λ wavelength and c is velocity of light. Calculated band gap values obtained for both the crystals E_g is around 5.87 eV and the value suggested that the prepared crystals are good insulators [18]. The optical properties of this particular crystal suggested that it can be utilized as a display in electronic devices. The SHG measurements of LACC and Na^+ ions incorporated LACC were carried out using simplest Kurtz and Perry powder technique [19]. Input LASER source of energy 4.3 mJ /5.3 mJ per pulse was used as input. The potassium di phosphate (KDP) crystalline powder was taken as the standard reference. The SHG efficiency of pure undoped LCC crystal is 0.57 times compared to KDP and for 2 and 4 mole Na^+ ions doped LCC crystals SHG efficiency is 0.47 and 0.352 times compared to KDP. This result confirms the non linear optical properties of the grown undoped and Na^+ ions doped LACC crystals, again the SHG efficiency of LACC is very much changed by the

sodium dopants due to its change in polarizability of the metal ions present in the doped material. Hence the materials can be used as an efficient frequency conversion material. An organo sodium metal produces high polarity due to C-Na bonds. It was already reported that the LASSER emitting sodium is can be as an artificial laser guiding stars.

Optical studies confirms about the grown crystals are good insulators, to understand more insight about the dielectric properties of the crystals, capacitance value and dielectric loss measurements are carried out with Agilent 4284 A, LCR meter between the frequency 100Hz -1MHz at the temperature range between 30 – 110^{o}C. Fig.14 shows the dielectric constant Vs varying frequencies of pure LACC and sodium added LACC crystals. The dielectric constant values of LACC and Na^{+} doped LACC crystals increases linearly with increase in temperature due to its polarization mechanism. At lower frequencies, the maximum value of static permittivity noted proves the presence of higher amount of space charge polarization present in the prepared sample. When an applied frequency increases, the space charge polarization cannot sustain themselves therefore the polarization values decreases [20, 21]. Furthermore the increasing dielectric constant with temperature is mainly due to the occurrence of space charge polarization near the grain boundary interfaces [22]. Moreover the electrical conductivity is larger at lower frequency due to the presence of hydrocarbons that was present in the amino acid. It was also reported that the polarization increases because of the sodium ions addition with smaller ionic radii and more highly charged [23].

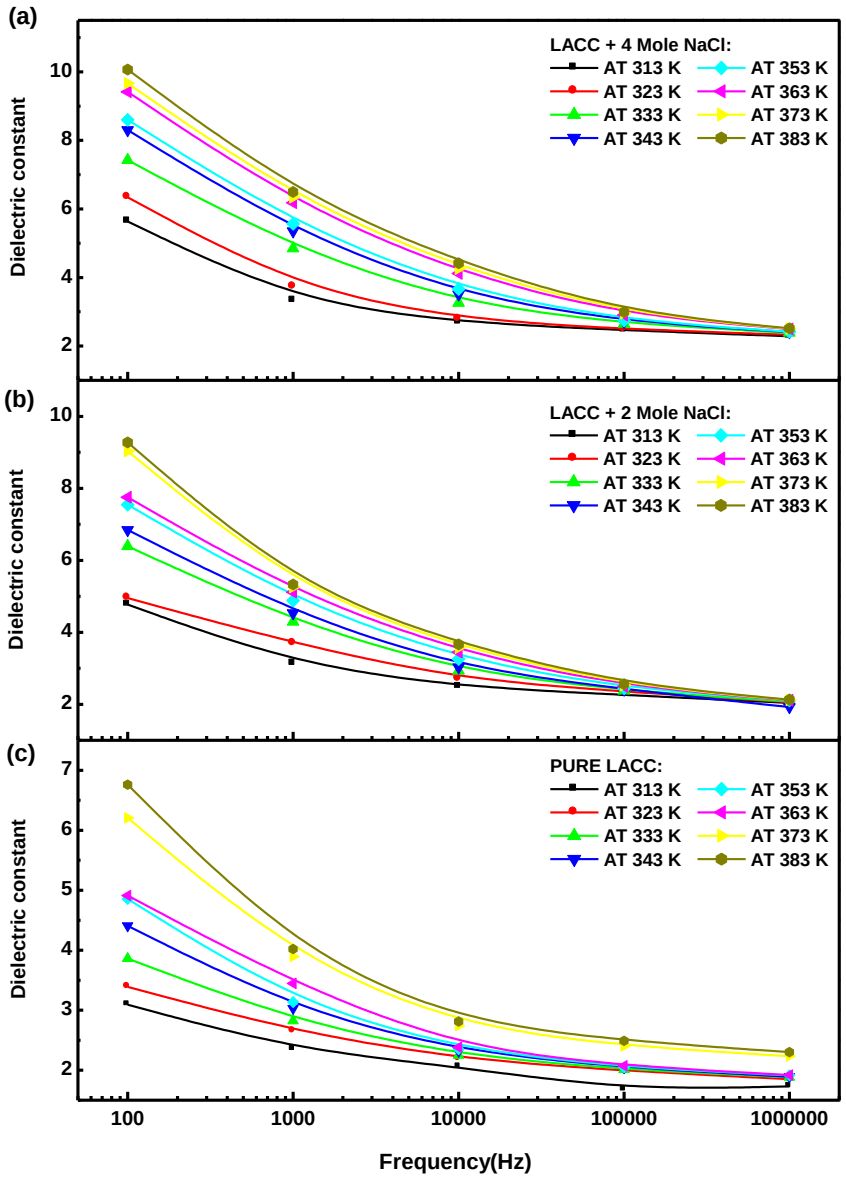

Fig. 14 Dielectric constant Vs frequencies of pure and sodium added LACC crystal.

When an a.c. voltage is applied to an insulator part of the electrical energy is absorbed by the material and losses in the form of heat energy. The corresponding energy loss in the form of heat is called dielectric losses. Nowadays dielectric losses is considered as a very important engineering problem as for as the application level is concerned and its significant involvement in the heat energy loss plays a versatile platform in the high energy storage applications [24]. The corresponding losses with respect to frequency response of LACC and Na^+ ions incorporated LACC crystals are depicted in the following Fig.15.

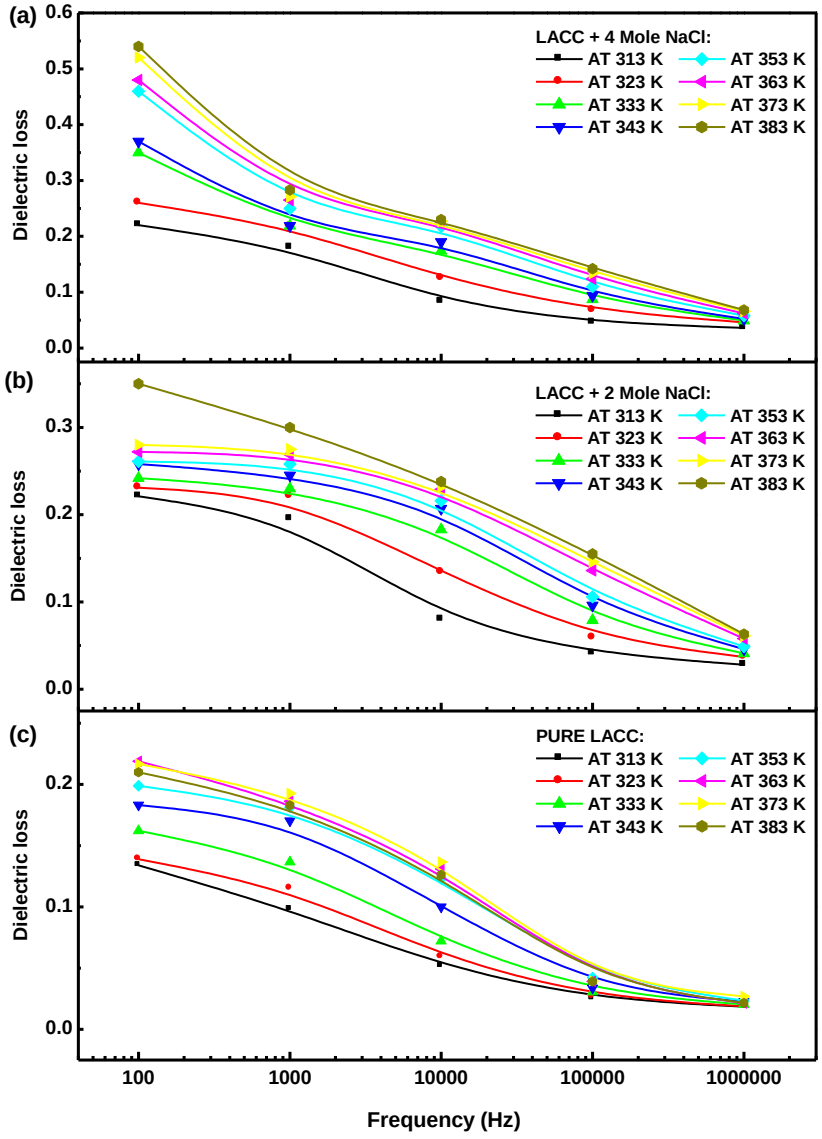

Fig. 15 Dielectric loss Vs frequencies of pure and sodium doped LACC crystal.

For LACC and sodium doped LACC, dielectric losses decreases with increasing frequencies. Sodium doped LACC crystals shows sight enhancement in the dielectric loss compared to the pure crystal. Compared to sodium doped LACC crystals pure crystals have lesser number of dipole moments per unit volume. When sodium ions were added as an impurity, the number of dipoles per unit volume doped crystals increases. Therefore sodium crystal experiences minimum losses [25]. Therefore the value of dielectric losses of the material decreases at higher frequencies. The lower value of dielectric losses at higher frequencies results that such crystals possess good optical quality and can be use as NLO applications [26-28]. Since the material is organometallic, the dielectric properties have applications application in the medical field such as EEG recording, transponders and electroshock therapy.

The calculated dielectric constant and the dielectric losses are utilized to find out the AC conductivity. The Arrhenius plot is drawn between 1000/T and ln σ_{ac} are given in Fig. 16

to 18. The slope of the Arrhenius plot gives the value of activation energy and is given in table 3.

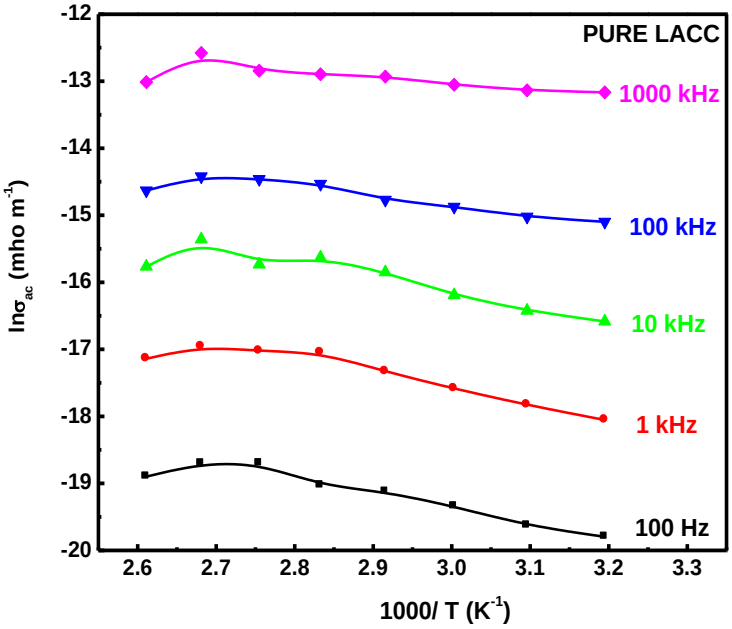

Fig. 16 The 1000/T vs. ln σ_{dc} plot for pure LACC.

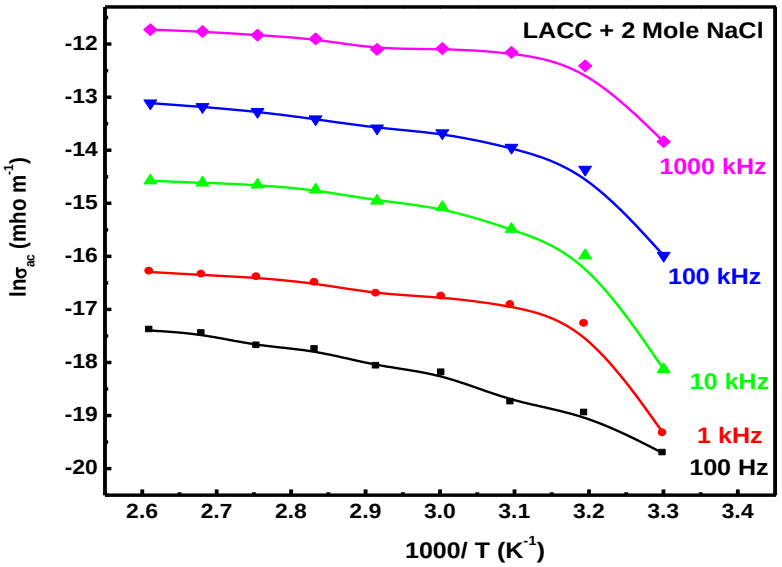

Fig.17 The 1000/T vs. ln σ_{dc} plot for 2 mole % sodium doped LACC.

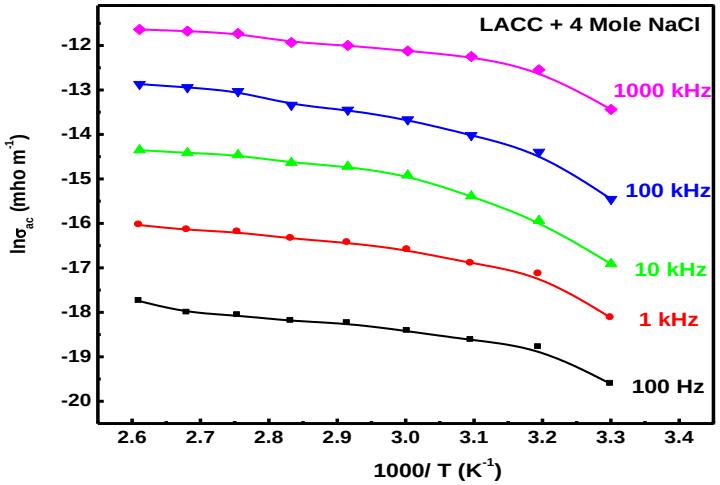

Fig.18 The 1000/T vs. ln σ_{dc} plot for 4 mole % sodium doped LACC.

Table 3 AC activation energy (E_{ac}) of undoped and Na^+ ions doped LACC crystals.

Crystal	E_{ac} at 100 Hz	E_{ac} at 1000 KHz
LACC	0.16 eV	0.1247 eV
LACC + 2 mole % NaCl	0.222 eV	0.1943 eV
LACC + 4 mole % NaCl	0.2772 eV	0.1943 eV

From the table it was known that the calculated activation energy at frequency 100Hz value is greater than 1000 KHz value. Thus it was clearly concluded as follows; when the frequency of applied a.c. voltage is low much more energy is required to activate the materials. At higher frequencies lesser energy is enough to activate the atoms or molecules. Also the calculated activation energy for the sodium doped LACC crystals is more than that of the pure LACC crystals. Thus the sodium impurity present in the prepared crystal increases the activation energy of doped crystals.

CONCLUSION

Semi organic compounds namely, L-Alanine Cadmium Chloride (LACC) and sodium admixed LACC crystals successfully prepared at a pH of 5.0. Calculated densities, Atomic Absorption Spectroscopy and EDAX studies confirm the addition of sodium metals on the parent crystalline lattice. Both the prepared crystals experiences monoclinic crystal system with good optical transparency. Due to doping thermal stability of the crystal enhances. Mechanical studies suggested that the grown crystals belong to soft material category. NLO studies reveal that sodium admixture enhances the SHG efficiency. Dielectric properties tuned by sodium is a high resistance dielectric material used for mechanical actuation, cap sense, strain gauge, textile applications and as a photosensitive material for charge storage in laser printer and copying machines.

Statements and Declarations

Competing Interests:

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contribution:

All authors contributed equally to the study, conception, design and all the authors commented on the previous versions of the manuscript. All authors read and approved the final manuscript.

Declaration of Competing Interests:

The author declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Research Data Policy and Data Availability Statements:

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Supplementary information:

Not Applicable

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This article does not contain any studies involving human participants or animals performed by any of the authors.

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