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Studies on the structural, thermal, optical and nonlinear optical characteristics of a novel organic glycine doped L-arginine adipate crystals

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ABSTRACT

Pristine and glycine doped L-arginine adipate (LAA) crystal, is a novel NLO materials were proficiently grownup by a simple slow evaporation way at ambient environment. The lattice parameters and crystalline nature for the as-synthesized crystals have approved in powder X-ray diffraction (XRD) statistics, while exhibited the as-grown crystals have monoclinic crystal system through precise space group. FT-IR and ^{1}H NMR spectroscopy designate that the plausible assortment kinds of molecular vibrations/configuration of as-grown crystals. Linear optical effects were originating via UV–Vis absorption spectral probe, and the cut-of wavelength has ~ 205 and 208 nm for LAA and GLAA crystals exclusively. The UV–Vis optical transmission inquiry exposed that the glycine doped LAA (GLAA) crystal had precise high optical transparency into visible-region and the virtual bandgap were calculated. Photoluminescence (PL) belongings tested for the grown LAA and GLAA crystals were detected fluorescence band emission at ~ 400–650 nm. Auxiliary, the developed crystals have scrutinized for thermal assets by TGA/DTA study and the results are inferred. Besides, the NLO belongings by Kurtz's and Perry technique which screening on the grown GLAA crystal was conversed to indorse the increases of SHG efficacy.

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1 Introduction

In current existences, the non-linear optical (NLO) materials consume fascinating attention towards the development of energetic and functional devices [1]. The organic NLO materials by high conversion adeptness further highly efficient optical harmonic generation possessions would lead to unique progress of frontier industrial applications in modern engineering optic, solid-state lasers, photonics and optoelectronic devices etc., [2]. Since, when related to inorganic NLO crystal materials, organic crystals remain greatly effectual for liable abundant physicochemical things, intended for amassing proper materials for varied device fabrication and uses. The NLO originated material by delicate non-linear absorption and vigorous refraction has broad courtesy for their prospective uses in optical limiting and signal processing. The interspersing potentialities of different organic and inorganic resources by good NLO goods comprise its application over the terahertz wave generation, information processing, optoelectronics modulators, etc., in manufacturing processes [3]. Mostly, amino-acids centered single crystals appeal a more concern owed to the chiral carbon atom and ought non-centrosymmetric natured molecular alignments which sort them favorable aspirants for NLO applications [4]. Amino-acids encompass a proton donor carbonyl-acid (COO–) group and alike proton acceptor (NH_2^+) amine itself, later its retains huge optical nonlinear susceptibility, rapid retort and with abundant optical laser damage threshold [5]. Then it could custom zwitterion in their structure have illustrated decent mechanical stuff, molecular chirality, high optical transparency, when it's softened in relevant solvents and proficient founding salt by further counter ions of any inorganic of organic ingredients [6].

Typically, prime amines act as strong bases amongst carboxylic acid clusters for might certainly make bond amid diverse organic acids. Abundant of amino acid built NLO crystals were stated and their belongings were deliberated [2, 5, 7]. From the family of amino acid crystals, L-arginine (LA) is a kind of such favorable NLO material, as well newly the LA invented crystals with exciting optical, and physicochemical things might assist as vibrant material (polyhedron feature of (100)) which offers huge amounts of results for crafty photonics, electro-optic device and optoelectronic applications [8]. Moreover, the LA has amino acid cross-chain by a 3-carbon aliphatic straight-chain, the distal expiration which was overlaid via guanidinium group, also the delocalization in positively charges by a conjugation amid the dual bond and the nitrogen (N) ion sets [9, 10]. Later the manifold hydrogen bonds construction is facilitated [11]. Likewise, the organic materials deal further kindness owed to their skilful application in an adipic acid (AA) over it to exist an appropriate second harmonic generation (SHG) material for NLO uses. The AA facilities of L-arginine and DL-arginine are designed by zwitterionic, expressly twofold negatively charged adipate ions and positively charged arginium ions, with a 2:1 stoichiometry ratio [12]. The crystal structure of L-arginine adipic acid/adipate (LAA) was described [13], and its crystallizes into the monoclinic crystal system, devising $P2_1$ space group, with values of a = 12.494(4) Å, b = 5.9510(7) Å, c = 16.719(5) Å, β = 105.977(5)°, Z = 2, and V = 1195.1(6) $Å^3$; $\alpha = \gamma = 90° \neq \beta$ reasonably [14]. Equally, glycine ($C_2H_5NO_2$) is the fortunate amino-acid just by a hydrogen atom as being cross-chain, consequently, it has tried as the optimal dopant to contract dielectric constant, which augment the good optical and NLO belongings into altering LAA crystals beliefs [15].

In the existent study deals through the objective of realizing novel NLO useful materials for academic and industrial usages, an effort has been made up the glycine revised L-arginine adipic acid/adipate (LAA) single crystals via simple slow evaporation way on the ambient environment. Also the structural, thermal, linear and NLO optical possessions via powder X-ray diffraction (PXRD) pattern, Fourier Transform Infrared (FT-IR) spectroscopy, ^1H Nuclear magnetic resonance (NMR) archives, Differential Thermal Analysis (DTA) and Thermogravimetric Analyzer (TGA) scrutiny, Photoluminescence (PL) spectra, the UV–Vis spectra, and Kurtz-Perry powder technique for (SHG) NLO applications on the typical belongings of grown pristine LAA and GLAA crystals. Moreover, the impact of glycine on LAA crystal has to findings these outcomes and these surveys are interpreted and deliberated.

2 Experimental procedures

2.1 Growth of pristine and glycine doped LAA crystals

L-Arginine (LA) and adipic acid (AA) (AR grade) were acquired from Merck Pvt. Ltd. The glycine compound was obtained from SRL chemicals Pvt. Ltd. For the parental complexes of LA and AA were attained via equimolar ratio (1:1) and it has dispersed in an aqueous solution with 5 mol % of glycine dopant was added auxiliary. This blend was stirred by a continuous magnetic stirrer for ~ 4 h with Double-distilled water (DDW, 20 mL) as a crucial solvent. After 4 h period, the solution conquers a homogeneous combination and is filtered with Whatman filter paper to eliminate impurities extant in their solution [16]. Finally, this was enclosed discretely and kept stable for conventionally slow evaporation to the ambient room environment ensue [17]. Later 15 days, the optically unique of GLAA crystals are well harvested and are sensibly recrystallized to achieve additional purity and transparency. The harvested LAA and GLAA crystals are shown in Fig. 1a and b, also the complete reaction process for LA responds with AA (i.e.) formation of LAA crystal was exposed in Eq. (1).

$$C_6H_{14}N_4O_2 + C_6H_{10}O_4 \rightarrow 2C_6H_{15}N_4O_2 . C_6H_8O_4 \quad (1)$$

2.2 Characterization techniques

The as-grown crystals were exposed to the crystalline structure, functional group, thermal, studies, linear optical and NLO belongings. The PXRD analysis of as-grown both pristine LAA and GLAA crystals are conceded over Rigaku Miniflex II with Cu Kα (λ = 1.5406 Å) radiation, for the material revelation of crystallinity, crystalline structure and phases also. To authorize the function groups and stretching/bending vibrations were qualitatively supported through FT-IR spectral analysis (Perkin Elmer RX-1 model spectrophotometer) by KBr pellet way into 4000–400 cm^{-1} series. The ^1H NMR spectra of the as-grown crystals data were proved in a magnetic field of 11.75 T (300 MHz) using Bruker FT NMR spectrometer device at room temperature for validation of the molecular chemical structures. The optical transparency and bandgap (E_g) were inspected by UV–Vis spectrophotometer (UV–Vis; Perkin Elmer) by the wavelength array from 200 to 800 nm. The PL spectra sustained to comprehensive via Horiba IHR-550 tool with Xe lamp at an excitation series at ~ 320 nm. The as-grown crystals are crushed to on fine powder, the TGA and DTA examine were executed over SDT Q600 V20.9 Build 20 model thermal analyzer at heated in stages of 20 °C/min with temperature array up to 30–600 °C in an inert nitrogen (N_2) atmosphere. The measurement of the NLO possessions of SHG efficacy was determined undergone Kurtz and Perry developed powder system over the Q-switched to

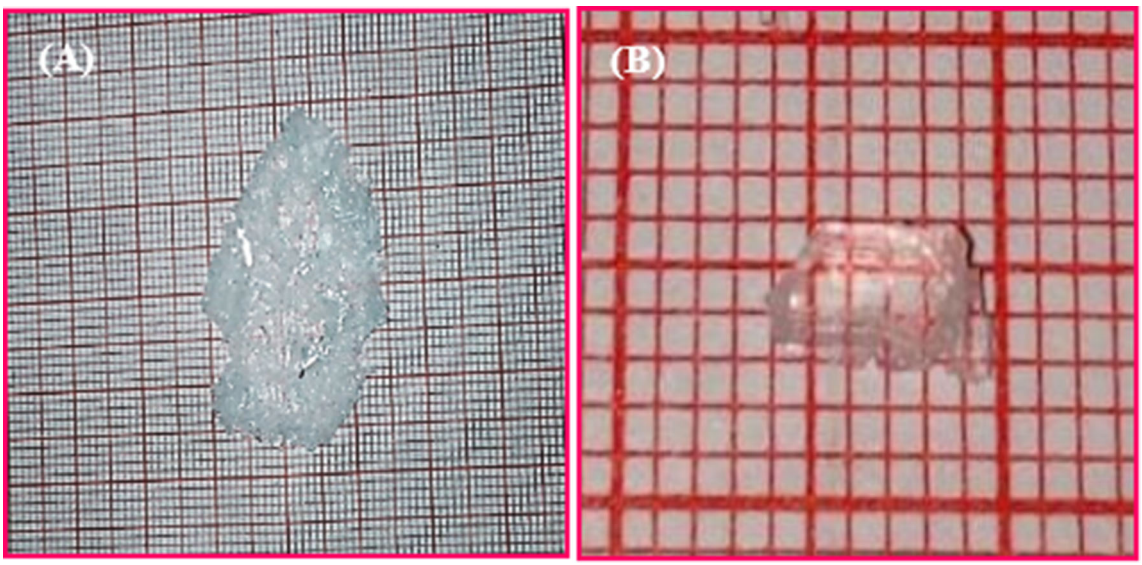

Fig. 1 The photographs of as-grown crystals of **a** LAA and **b** glycine doped LAA

Nd: YAG laser source by an input pulse of ~ 11 mJ and emission of green light (~ 532 nm) relatively.

3 Results and discussion

3.1 Powder XRD studies

The PXRD pattern of as-grown pristine LAA and glycine doped LAA (GLAA) crystals were denoted in Fig. 2a and b. Besides, the as-attained crystals consume good crystallinity [18]. The PXRD pattern was well-indexed through *hkl* values whereas crystalline structure and lattice parameters were intended and obtainable in Table 1. It is witnessed the lattice parameters of LAA and GLAA together crystals retains are monoclinic crystal system by $P2_1$ space group and these are well contracted with reported morals [13]. There are substantial variants detected in the lattice parameters, intensities and cell volume of GLAA when likened to pristine LAA crystal owed the integration of supreme glycine dopant [9]. However, the existence of glycine has reformed slightly in the lattice parameters and there are no momentous variations in the basic structure of as-grown crystals.

3.2 FT-IR analysis

FT-IR spectroscopic scrutiny is a vital system to authorize the existence of acquired functional groups and numerous vibration types of the as-grown crystals in a typical molecule absorb [19]. The FT-IR spectrum of succeeded LAA and GLAA crystals are revealed in Fig. 3a and b. Usually, the projected broad envelope among 2500 and 3400 cm^{-1} wavelength comprises of stretching vibration kinds owing to the superimposed N–H and C–H bonds, chiefly, the cyclical N–H bonds were detected that there is a distinctive absorption band on 3184 and 3193 cm^{-1} fairly. As a wide extent inter or intramolecular forces, hydrogen bonds has a fabulous influence on the swing of FT-IR spectrum.

Besides, the stretching vibration of C=O band drops to 1660 cm^{-1} series for the carboxyl groups deeds as a proton donor in the molecules. The solid absorption peaks at 1390 and 1326 cm^{-1} are owing to symmetric and asymmetric stretching types on C=O stretch of –COO– vibrations, and CH_3 groups disparately approve the deprotonation of a carboxyl group, separately [3]. Intense and sharp peaks are perceived at 3341 and 3346 cm^{-1} remains to stretch vibration of surface fascinated hydroxyl (O–H) groups of the as-grown LAA and GLAA crystalline material. The aliphatic C–H stretching manners are decided at 2928 and 2934 cm^{-1} (asymmetric) wavelength [20]. Although, the manifold adequate structures at the inferior energy mode of the envelope specifies that the sturdy hydrogen bonding interface of $–NH_3^+$ group with $–COO^-$ groups in these as-grown crystals. The bands at 2106 and 2098 cm^{-1} are owed to blend of asymmetrical $–NH_3^+$ bending vibration of LA molecule and it's out of plane

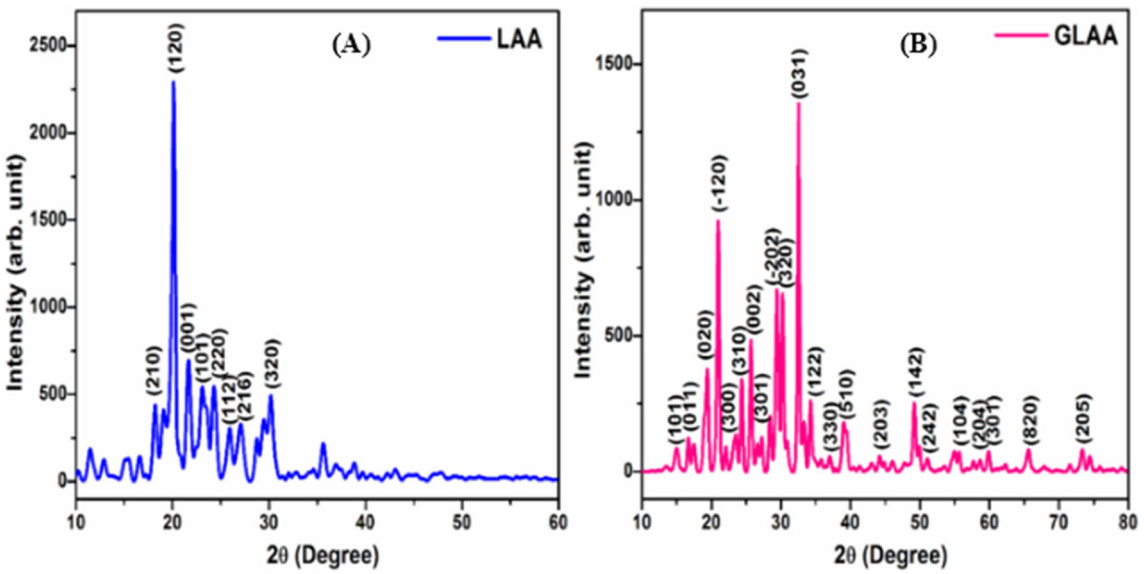

Fig. 2 Powder XRD pattern of as-grown **a** LAA and **b** GLAA crystals

Table 1 The lattice parameters and identified crystal structure of as-grown crystals

Grown crystal	Lattice parameters			α, γ, β	Crystal structure
	a (Å)	b (Å)	c (Å)		
LAA	11.95576	9.149527	6.756624	$\alpha = \gamma = 90° \neq \beta$	Monoclinic
GLAA	11.21359	9.704490	4.120999	$\alpha = \gamma = 90° \neq \beta$	Monoclinic

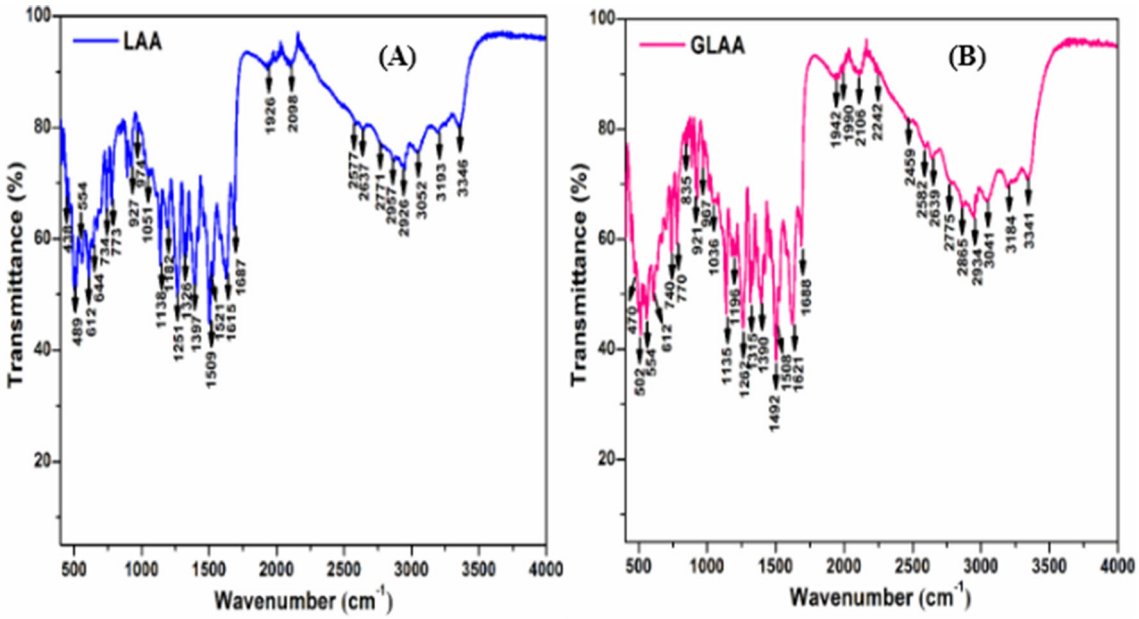

Fig. 3 FTIR spectrum of as-grown **a** LAA and **b** GLAA crystals

Table 2 FTIR spectral assignments for grown LAA and GLAA crystals

Wavenumber (cm^{-1})		Corresponding vibration modes
LAA	GLAA	
3346	3341	O–H stretching
3052	3041	C–H stretch
2926	2934	C–H asymmetric stretching
2577	2582	N–H symmetric stretch
2098	2106	$-NH_3^+$ bending vibration
1687	1688	C=O stretching
1521	1508	NH_2 in-plane bending
1397	1390	CH_3 symmetric plain bending
1326	1315	C=O stretch of $-COO^-$
1251	1262	C–N stretching
1138	1135	C–C–C symmetric stretching
1051	1036	C–H in-plane deformation
927	925	C–C stretching
773	770	–C≡C–H stretching
554	554	C–O bending

bending [21]. The vibrational assignments are itemized in the Table 2. Likewise, there is no substantial difference have detected in the peaks incidence of doped crystals. The peak at 1051 and 1036 cm^{-1} are consigned to the C–H in-plane deformation kinds of vibration. The C–O bending and C–C stretching sensations of actually absorption bands seem at 612, 644, 770, 835 and 921 cm^{-1} are owed to the vibrations of profound functional groups and carbon chain of LA molecules reasonably.

3.3 NMR studies

To define the structure of conquered organic compounds and chemical shifts of the as-grown crystals, proton (^1H) NMR spectrum is a vibrant method to be verified at room temperature. The ^1H NMR spectra of acquired LAA and GLAA crystals are displayed in Fig. 4a and b. In both the ^1H NMR spectrum the advent of an intense signal at δ = 4.78 ppm owing to the incidence of NH_2 and NH_3^+ groups on D_2O solvent [16]. Since these spectra, there have two triplet

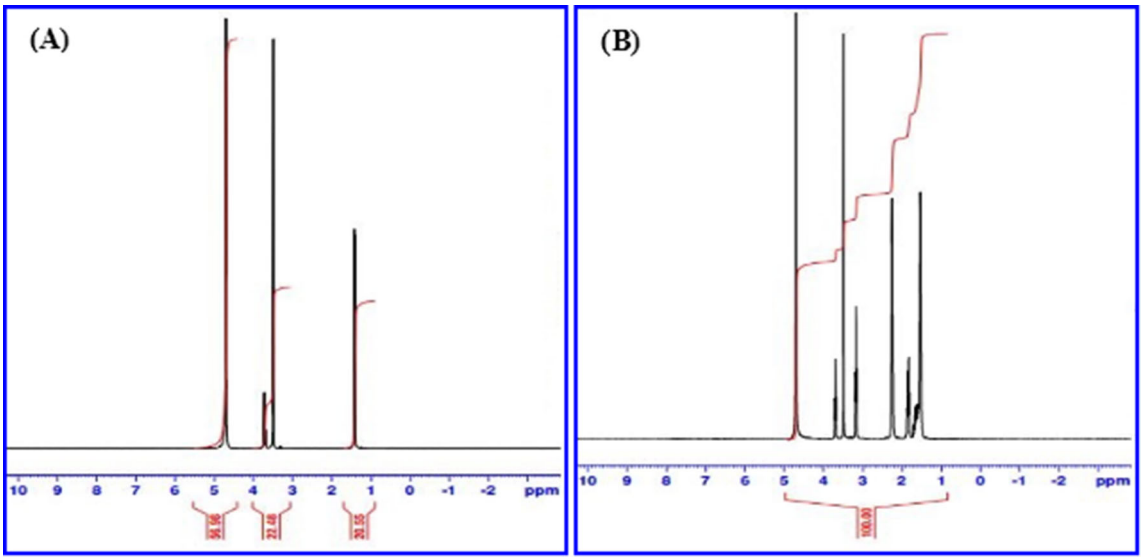

Fig. 4 ^{1}H NMR spectrum of as-grown **a** LAA and **b** GLAA crystals

peaks at δ = 3.57 and 3.08 ppm agrees to the hyperfine splitting of adjacent C–H and CH_2 protons, likewise the methylene carbon composed to carboxyl groups in LA for the crystalline components [22]. Furtherly, two multiplets intensity peaks in the series of δ = 1.75 and 1.552 ppm agree to the two CH_2 groups of carbon transport terminals into own amino-acids. Equally the CH_3 protons sustained terminal of adipate acted at δ = 2.121 and 1.39 ppm singly. These as-obtained chemical shifts are signified into δ ppm and consigned via web literature ideals. There are no specific peaks seemed for electron extracting NH_2 and –COOH groups signals in the ^{1}H NMR spectrum for nearby to the –CH carbon atom, later its leads to the delocalization of electrons to excuse for the dipolar nature [15], which specifies that they are ionic nature and are intricate in the subordinate services. Besides, the high δ value of this triplet peak has owed to the existence of electron together to the –CH carbon atom thus indicates to the delocalization of electrons to the reason for the dipolar nature. The amino-acid features are therefore very well carried out. Henceforth, the persistent functional groups were existent in the novel compounds are thus obviously authorize its purity and these outcomes are well inferred.

3.4 UV–Vis spectral analysis

It is authoritative to have noble optical transparency in an NLO crystal in the precise visible region. Optical absorption and transmittance spectra for the as-grown crystals were verified in the range from ~ 200 to 800 nm has exposed in Fig. 5a. From the UV–Vis absorption spectrum of pristine LAA and GLAA crystals they have discloses that the lower cut-off wavelengths are found to be ~ 205 and 208 nm individually [6]. Then, the lesser percentage (%) of UV absorption designates that the GLAA crystal gladly agrees the sharp UV cut-off wavelength perceived at ~ 223 nm has owing to the p–p* transition in this virtual crystalline material [18]. This auxiliary screening the UV optical transmission spectrum (insert Fig. 5a) of doped GLAA crystal has progressed optical transparent effects allied with pristine LAA crystal. These belongings are augmented in the glycine doped GLAA crystal is suitable for generating SHG stuff as well. The as-grown GLAA crystal expressions high optical transparency (87%) related than pristine LAA sample owed to the integration of glycine matters [23].

An optical band gap is additional essential optical parameter might be assessed since UV–Vis absorption spectra. It is kind of dynamic eminence learning for the requisite of absorption coefficient (α) on photon energy ($h\nu$), however, eventually, profits to calculating the optical bandgap (E_g) values of as-considered crystals were consuming the typical Tauc relation; $(\alpha h\nu)^2 = A(h\nu - E_g)$ [24]. However, E_g, A and h stand for an optical bandgap of the material, relation constant and Planck's factors also. The appraised optical band gap (E_g) were originated to be ~ 5.04

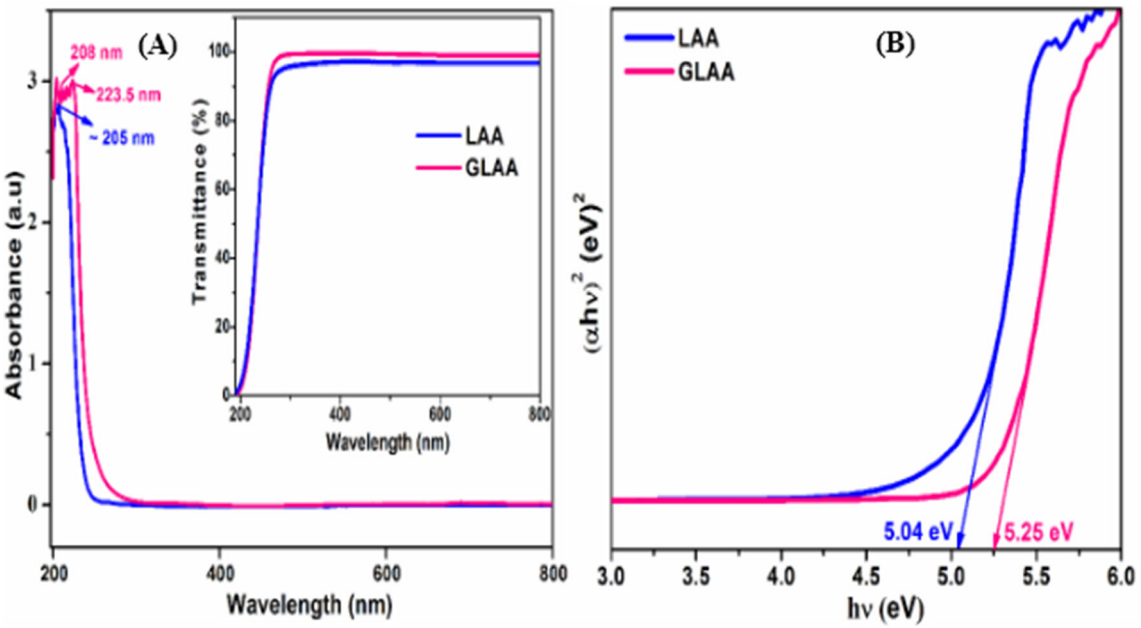

Fig. 5 **a** UV–Vis Absorbance, (insert) Transmittance spectra and **b** Tauc's plot of as grown LAA and GLAA crystals

and 5.25 eV for as-grown pristine LAA and GLAA crystals are publicized in the Tauc plotting chart of Fig. 5b. The glycine doped GLAA crystal have slightly broader band gap which likens than pristine LAA crystal. Meanwhile, the enhances of the optical transparency and bandgap energy might be owed the realization of hydrogen bonding amid the amino-acids of glycine and LAA molecules would possess and boosts the double bond personalities of N…C bonds also hinder thereby transitions of electrons (e^-) states which presenting absorption into UV region [22]. From the validation of perceived that the as-grown glycine influenced LAA crystals to have admirable transparency in the entire visible region and short cutoff wavelength facilitates. Hence its could signify the suitable material for probable NLO and optoelectronic applications and the SHG (frequency conversion) belongings as of Nd: YAG laser [25].

3.5 Photoluminescence studies

The photoluminescence (PL) study is a hopeful apparatus to recognize the intrinsic impurities, luminescence manners related to the compounds through explicit color centered emissions of the as-grown optical materials. PL emission spectra of the as-grown pristine LAA and GLAA crystals have been surveyed in the series from 400 to 650 nm at room atmosphere [9]. Though the photo-excited wavelength of ~ 320 nm and the emission spectrum of each crystal sample was demonstrated in the visible region at ~ 440 nm (Fig. 6a and b). The PL analysis exposes that the color centered a strong emission of consistent to LAA crystal is greater/broader at 439 nm on violet-colored emission was detected. The doping of glycine assisted that substantially enhances into actual intense emission strengths and likewise marginal shift in the peak emission wavelength (~ 447 nm) of GLAA crystal. The PL result signposts that the GLAA crystal has extensive violet-blue fluorescence colored emission [17]. Consequently, the auxiliary intermediary emission of as-grown both LAA and GLAA crystals are at ~ 502, 608 and 503, 608 nm (green and orange colored emission) congruently. The maximum and broad PL centred peak at ~ 447 nm for GLAA crystal might be ascribed to n-π^* transition which occurrence and also protonation of the amino to the carboxyl group fairly [26]. The broadness of PL intensity and peak exposes the crystalline nature of as-obtained materials. Accordingly, GLAA crystal has a blue colored emission and it advises that it could be used as a novel blue color radiating material, besides it might be used as the noticeable material for the devising violet-blue color lasers.

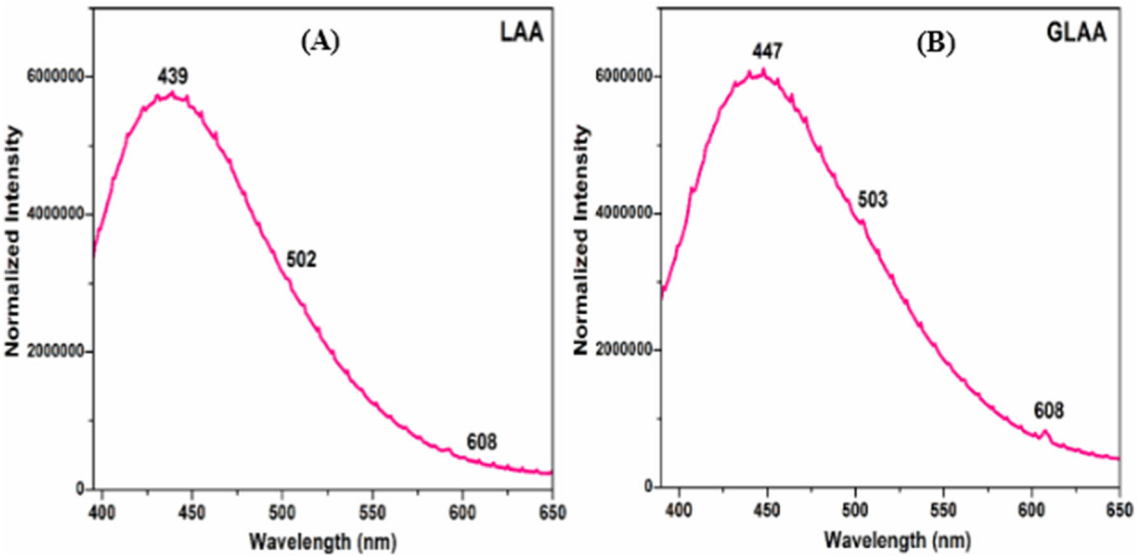

Fig. 6 PL emission spectrum of as-grown LAA and GLAA crystals

3.6 Thermal studies

Thermal analysis was engaged to realize the weight % and energy variations in the crystal samples concerning the temperature (°C) [8]. The thermal things of prepared LAA and GLAA crystals were explored from TGA/DTA studies and the ensuing thermogram traces are publicized in Fig. 7a and b. Both the pristine and GLAA crystals illustrate two stages of weight loss, 78.5% and 81% in the first stage and 21.2% and 18.6% in the second stage arises in the range of 100–260 °C individually. This outcome has sustained by the DTA analysis where an endothermic response is perceived at 230–280 °C of both crystals. For reductions in the decomposition temperature of the doped GLAA crystal is owed to the bond energy assumed by the dopant of glycine content. Similarly, the decomposition temperature of the pristine and GLAA crystals together started on 247.8 °C and 241.1 °C separately, then the solitary melting point is the noticeable manifest for whole crystallinity of these grown crystals. The DTA curve has numerous exothermal peaks in 220–320 °C temperature range that agrees to residual yields of dehydration of the as-grown crystals [17]. The continual loss of weight was witnessed at primary temperature (TGA) which relates to the vaporization of physically adsorbed water (H_2O) molecule extant in the as-grown crystals, which is also directed in the DTA arc by a sharp endothermic peak at 102 and 223.5 °C. Through the absolute stages, accompanying discharges of CO_2, CO– and amine molecules by the right condensation reaction of simplest amino-acids glycine, amine and carboxyl groups are progressive on auxiliary heating for above 260 °C, and thus the resultant perhaps residue undergoes degradation [15]. The consequences designate the thermal steadiness of GLAA crystal up to 240 °C and establish its aptness to endure the high temperatures in laser testing and also suitable for exploiting this material for NLO applications.

3.7 Second harmonic generation (SHG) studies

SHG test for the as-grown powder crystals of LAA and GLAA was considered by engaging Kurtz-Perry powder technique [27]. The SHG archives are required to study the optical belongings, expressly transmission at essential and so second harmonic wavelengths formerly deciding applicability of the as-acquired crystals [28]. The powdered crystal samples which were illuminated via Nd: YAG laser beam source by filtered in the vital harmonics output wavelength of ~ 1064 nm on passing pulse width of 10 ns simultaneously. The SHG efficiency of as-grown crystals was equated with that of standard KDP from the output bases [20]. Consequently, the SHG efficiency established that the as-grown LAA and glycine doped GLAA crystals have virtually 0.72 and 1.24 times greater efficiency than reference KDP material since it's specified that the glycine dopant

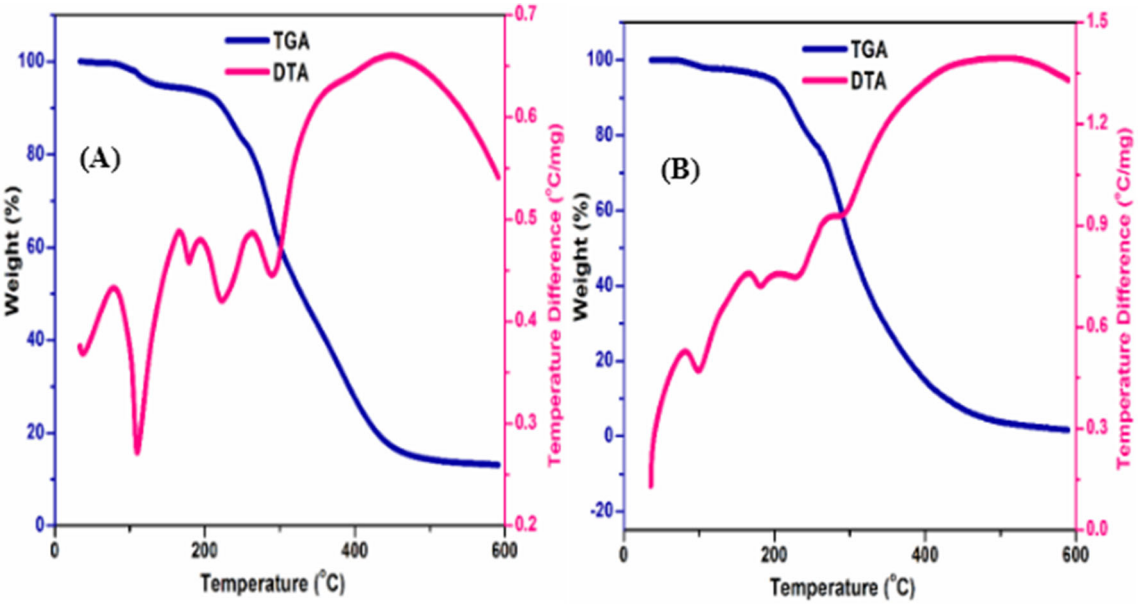

Fig. 7 The TGA and DTA curve as-grown **a** LAA and **b** GLAA crystals

has superior the optical NLO belongings of pristine LAA crystal [24]. The increases in the SHG effectiveness of as-grown GLAA crystals might be owed to the formation of multiple hydrogen bonds amid the amino-acid and huge optical nonlinearity which glycine amended environment locally in the LAA crystal. Equally, the superior delocalization of π-electron over donor–acceptor bonding system is the belief phenomenon of thus actual grownup of GLAA crystal ensuing to high NLO response. Hence this glycine doped LAA crystalline material is deliberated for NLO and photonic applications.

4 Conclusion

In summary, the good quality glycine doped LAA (GLAA) single crystals were been harvested effectively through facile slow evaporation route crystallized by evident explicit crystal system which endorses by PXRD studies. The numerous functional groups and chemical shifts in with the selective site of the as-grown crystals were recognized and consigned qualitatively by FT-IR and ^{1}H NMR spectrum analysis. From the UV–Visible spectrum inquiry, the grown GLAA crystal has high optical transparency over entire UV–Vis region for an accumulation of glycine dopant and the inferior absorption is originate to be ~ 208 nm, also the reliable optical band gap (~ 5.25 eV) was assessed. PL spectrum spectacles that the as-grown crystal material has extensive violet-blue colored fluorescent emission on 447 nm. The thermal steadiness of the samples was deliberate through ensured TGA/DTA curves and establish that the molecules of the as-grown crystal are thermally steady up to 241 °C. The Kurtz-Perry method discovered that GLAA crystal parades augmented the SHG efficiency of 1.24 and 0.72 times superior that of reference KDP and also pristine LAA crystal, while the occurrence of glycine dopants has upgraded the NLO properties. Hence from the upstairs surveys, the good crystalline and optical eminence of grown GLAA crystal were explored and establish to be promising aspirant in future photonic, optical limiting, and NLO device fabrication.

Compliance with ethical standards

Conflict of interest The authors have declared no conflict of interest.

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