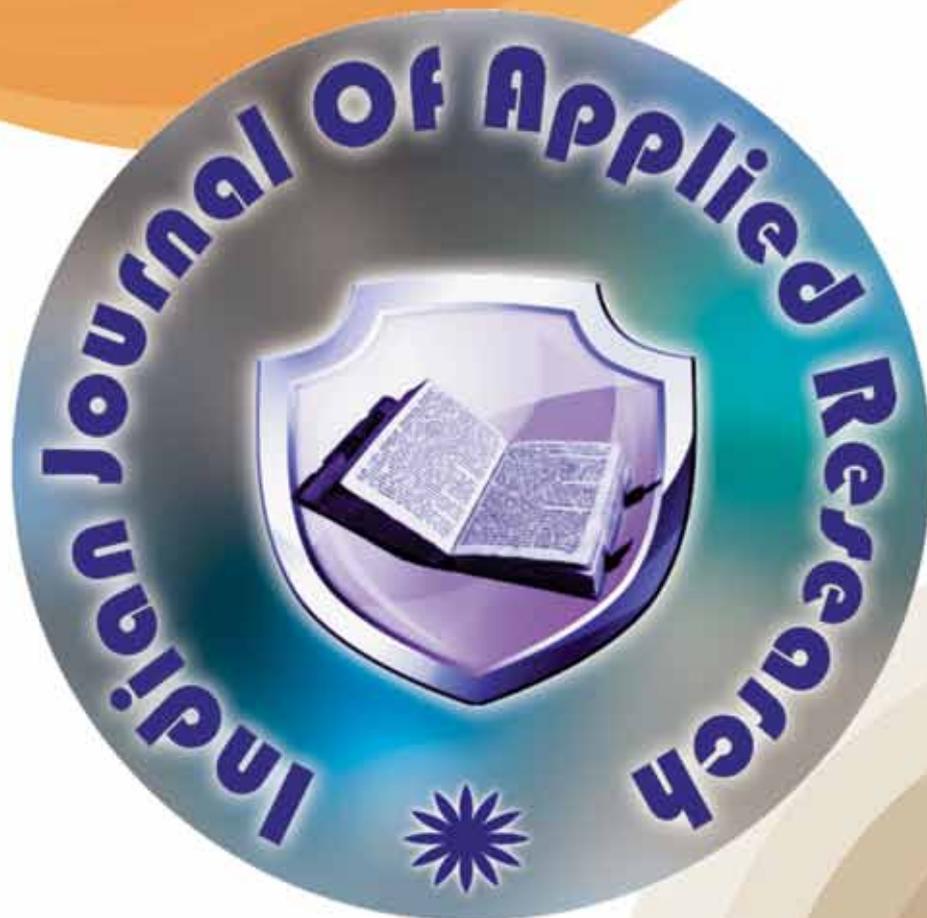


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Crystal Growth, Thermal and Optical Studies on L-arginine Based Nonlinear Optical Material

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ABSTRACT

L-arginine trifluoroacetate (LATF) an organic nonlinear optical material has been synthesized in aqueous solution at 55°C and characterized by FT-IR, and FT-NMR spectroscopic studies. Solubility of LATF has been determined in water and ethanol in the temperature range 30-50°C. The single crystal with dimensions 10x7x4 mm³ were grown by slow evaporation technique at 30°C. The grown single crystal has been subjected to single crystal X-ray diffraction to determine the unit cell dimensions. The TGA and DTA reveal that the material has good thermal stability in the temperature range 35-700°C. The linear (UV-Vis) and nonlinear optical (SHG efficiency) properties of this material have been evaluated.

Keywords : Crystal growth; Organic NLO materials; SHG Efficiency, UV-Vis Transmittance

1. Introduction

There has been intense research and development on blue-green laser light sources, which provide high density recording capacity for compact disks and other optical data storage systems. Semiconductor laser diodes are now considered as prime candidates because of their potential advantages of compactness and low cost. However, beyond harmonic generation, SHG devices have the advantages of power emission, choice of wavelengths and reliability, which make them desirable for practical applications [1-3]. Inorganic NLO materials have very high thermal and mechanical stabilities but possess modest optical nonlinearity due to the lack of extended p-electron system between donor and acceptor groups. For example potassium niobate has the nonlinear coefficient $d_{31} = 15.8$ pm/V and it is suitable for frequency doubling of diode lasers (e.g. GaAs, $\lambda = 808$ nm) [4].

Organic materials are attracting great attention due to their fast and large nonlinear response over a broad frequency range, inherent synthetic flexibility and high optical damage threshold. It was proposed that the presence of electron donating and electron accepting groups on benzene and stilbene systems would result in strong second harmonic generation [5]. Among the organic NLO materials, amino acid (L-arginine, L-histidine and L-alanine) derivatives have been extensively investigated due to their molecular chirality, wide transparency and donor-acceptor molecules with large hyperpolarizabilities. For example L-arginine phosphate has SHG efficiency 1.56 times of KDP with optical transparency in the range 250 –1500 nm [6-9].

2. Experimental

2.1 Synthesis and Solubility

LATF (C₇H₁₄N₄O₂·CF₃COOH) was synthesized by of L-arginine and trifluoroacetic acid was thoroughly dissolved in double distilled water. The resulting solution was evaporated to dryness by heating carefully at 55°C in a temperature controlled water bath to avoid any possible decomposition. A white synthesized crystalline salt of LATF was obtained with the yield of 95% then the material was purified by repeated recrystallization process in double distilled water. The melting point of the compound was found to be 215 ±1°C.

The solubility test was carried out for LATF in water and ethanol in the temperature range 30-50°C. The solution was prepared in water at 30°C with continuous stirring using a magnetic stirrer to ensure homogeneity of temperature and concentration over the entire volume of the solution. On

reaching saturation, the content of the solution was analyzed gravimetrically and this process was repeated for every 5°C for water and ethanol. The solubility curves of LATF are shown in Fig.1. It is observed that LATF have a positive temperature gradient and the solubility of these materials is high in water and hence water was used as the solvent for crystal growth.

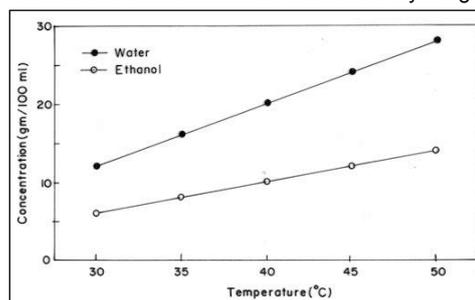


Fig.1 Solubility curves of LATF.

2.2 Crystal Growth

The synthesized salt of LATF was thoroughly dissolved in double distilled water using a magnetic stirrer. The solution was saturated at 30°C with pH 3.7. The solution was filtered twice to remove the suspended impurities and allowed for slow evaporation at the same temperature. A single crystal of the size 10 × 7 × 4 mm³ was grown within 30 days with an approximate growth rate of 0.3 mm/day (Fig.2). The grown crystal found to be stable, nonhygroscopic and unaffected by the environment.



Fig.2 Grown single crystal of LATF.

3 Characterizations

3.1 Single Crystal X-ray Diffraction Analysis

The grown crystal of LATF was subjected to single crystal X-ray diffraction using an Enraf Nonious CAD4 X-ray diffractometer with MoK α radiation ($\lambda=0.7107 \text{ \AA}$) to find out the unit cell dimensions. The cell dimensions are $a = 10.571(1) \text{ \AA}$, $b = 5.713(5) \text{ \AA}$, $c = 10.866(2) \text{ \AA}$ and $b = 106$ with volume 628 \AA^3 . The LATF belongs to monoclinic system with space group P21. The noncentrosymmetric space group thus satisfies the basic and essential material requirement for the SHG activity of the crystal.

3.2 FT-IR Spectral Analysis

The FT-IR spectrum of LATF was recorded using a JASCO 460 Plus FT-IR spectrometer by KBr pellet technique in the range $400\text{--}4000 \text{ cm}^{-1}$ to confirm the molecular structure of the synthesized compounds (Fig.3) and compared with L-arginine.

The N-H stretching of guanidyl group ($+(H_2N)_2CNH$) in L-arginine appears at 3289 cm^{-1} whereas in LATF it is shifted to 3380 cm^{-1} , due to the protonation of nitrogen in the guanidyl group. Similarly, in L-arginine the C=O stretching appears at 1661 cm^{-1} , but it is shifted to 1640 cm^{-1} in LATF, due to the deprotonation of carboxylic group [9,10]. The strong band at 1195 cm^{-1} is assigned to C-F stretching frequency of LATF. The vibrational bands at 3171 cm^{-1} is assigned to C-H stretching and CH₂ bending appears at 1365 cm^{-1} in LATF. The other characteristic vibrational frequencies are presented in table 1. From the spectral analyses, it is confirmed that the guanidyl and amino (NH₃) groups are protonated and thus balance with the negative charge of the carboxylate (COO⁻) functionality [11, 12].

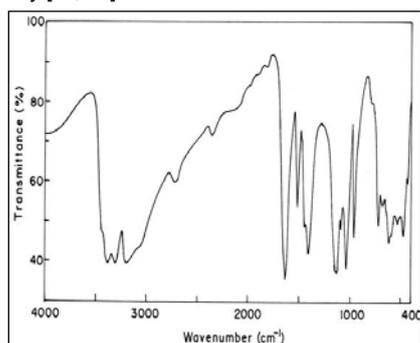


Fig.3 FT-IR spectrum of LATF.

Table 1 The FT-IR spectral band assignments of LATF.

Wavenumber (cm^{-1})		Assignments [11-1]
L-arginine	LATF	
3289	3380	N-H stretching
3155	3171	C-H stretching
1681	1693	N-H asym. bending
1661	1640	COO- sym. stretching
1441	1441	CH ₂ sym. bending
1377	1365	CH ₂ bending
1330	1327	C-H bending
.....	1195	C-F stretching
1136	1133	C-C-N asym. stretching
667	648	COO- out of plane bending

3.3 FT-NMR Spectral Analysis

The ¹H-NMR spectrum of LATF were recorded using a JEOL GSX -300 MHz FT-NMR spectrometer with DMSO as the solvent (Fig.4). In the ¹H-NMR spectrum of LATF, the proton in the CH₂ group gives a signal at $\delta = 3.44 \text{ ppm}$ due to the influence of adjacent CH₂ group. Another triplet at $\delta = 2.92 \text{ ppm}$ is attributed to the CH₂ group with the influence of neighboring methine group. The area under these groups is 1.00 and 2.093 indicating the proton population as 1:2 [11, 12]. The other chemical shifts in ¹H-NMR spectrum of LATF

is presented in table 2.

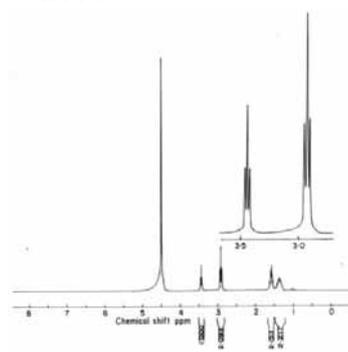


Fig. 4 ¹H-NMR spectrum of LATF.

Table 2 ¹H-NMR spectral assignments of LATF.

Spectrum	Assignments [12,13]	
¹ H - NMR	1.372	NH of L-arginine
	1.554	CH of L-arginine
	1.600	CH ₃ of acetic acid
	2.921	CH ₂ of L-arginine
	3.440	CH ₂ of L-arginine

3.4 Thermal Analyses (TGA and DTA)

Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) for LATF were carried out using a Seiko Thermal analyzer to determine the thermal stability of the grown crystals in the temperature range $35\text{--}700 \text{ }^\circ\text{C}$ (Fig.5). A ceramic crucible was used for heating the sample and the analyzes were carried out in nitrogen atmosphere at a heating rate of $20 \text{ }^\circ\text{C}/\text{min}$.

In Fig.5, the endothermic peak in the DTA curve at $215 \text{ }^\circ\text{C}$ represents the melting point of the LATF compound. The TGA curve shows that there is a weight loss of about 40% in the temperature range $250\text{--}270 \text{ }^\circ\text{C}$ due to the decomposition of the compound. There is no significant endothermic or exothermic peak above $250 \text{ }^\circ\text{C}$ in the DTA whereas TGA shows a complete weight loss (5%) at $700 \text{ }^\circ\text{C}$. This is due to the step-by-step decomposition and the release of volatile substances (probably ammonia and carbon dioxide) in this compound.

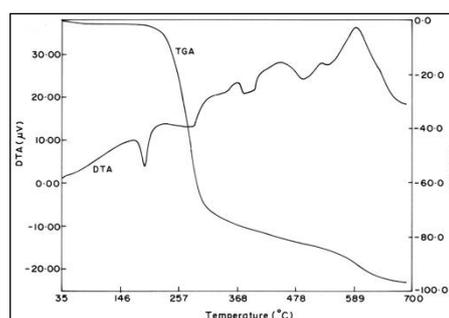


Fig. 5 TGA and DTA curves of LATF.

3.5 UV-Vis Optical Transmittance

The UV-Vis transmittance spectrum of LATF was recorded using a Varian Cary 5E- UV-Vis spectrophotometer in the range $200\text{--}800 \text{ nm}$ (Fig.6). The figure shows that the material has a wide optical transmittance in the entire visible region ($400\text{--}800 \text{ nm}$) and the lower cutoff wavelength is down to 280 nm due to $p\text{-}p^*$ transition in the compound [13]. From the UV-Vis spectral analysis, it is noted that there is no significant absorption in the UV and visible region thereby confirming the advantage of the crystal for optical applications. The large transmission in the entire visible region and the lower cutoff wavelength enable them to be the potential candidates for

second and third harmonic generation of Nd:YAG laser fundamental and for the generation of the higher harmonics of the GaAlAs laser diode emitting the fundamental in the vicinity of 800 nm for the achievement of blue lasers.

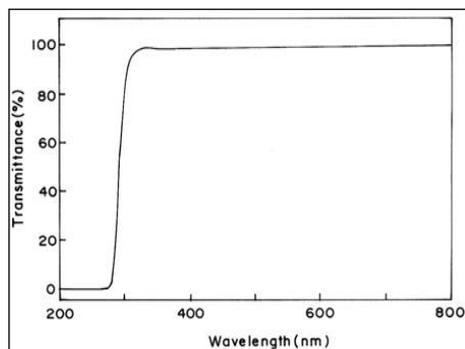


Fig. 6 UV-Vis transmittance spectrum of LATF.

3.6 Measurement of Powder SHG Efficiency

The powder SHG efficiency of LATF was measured following the Kurtz and Perry technique [14] using an Nd:YAG laser

(1064 nm, 8 ns, 10 Hz). The sample was ground and sieved into different particle sizes (<106, 106-125, 125-150, >150 mm). All the powdered samples were tightly packed into the separate microcapillary tube of uniform bore of 1.5 mm diameter. The laser power incident on the capillary tube was 6 mJ. The SHG at 532 nm (green light) was collected by the photomultiplier tube (PMT). The result obtained for LATF shows powder SHG efficiency 0.35 times that of a well-known organic NLO material urea. The SHG efficiency for various particle sizes shows that SHG intensity increases with particle size, thus giving a proof for the phase matching property of LATF.

4. Conclusions

Single crystals of LATF were grown in aqueous solution by the slow evaporation technique at 30°C. The molecular structure of the synthesized compounds is confirmed by FT-NMR and FT-IR spectroscopic techniques. The unit cell parameters are evaluated by single crystal X-ray diffraction technique and the crystal belongs to monoclinic system with the space group P21. The TG-DTA shows a good thermal stability of the grown crystals and it establishes the suitability of the grown crystal for device fabrication. The UV-Vis transmittance spectrum highlights a good transmittance in the entire visible region and the lower cutoff wavelength is 280 nm. The powder SHG efficiency is measured which indicates the phase matchability of the materials.

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