**L-Histidine family single crystal for frequency matching applications**

Indu

Research Scholar- Department of Physics

Baba Mastnath University

Haryana, India

induarya0@gmail.com

Sonia

Assistant Professor- Department of Physics

Baba Mastnath University

Haryana, India

ahlawat.sonia44@gmail.com

Babita

Assistant Professor- Department of Physics

Baba Mastnath University

Haryana, India

babitaphy@gmail.com@gmail.com

Meenakshi

Assistant Professor- Department of Physics

Baba Mastnath University

Haryana, India

meenakshi4phy@gmail.com@gmail.com

**ABSTRACT**

This article reviews the properties of L-histidine based single crystals.Here, the crystal growth techniques, through which better sized good quality single crystal has been grown in economic manner is also reported. A plethora of nonlinear compound of L-histidine has been reported, which confirms the suitability of this amino acid for nonlinear optical applications.This paper includes the structural review of the materials which gives the cell parameters of the compound. UV-VIS-NIR spectroscopy was discussed for determining the optoelectronics applications of crystal. Mechanical strength of the material is determined through Vickers hardness. Nonlinear behavior has been analyzed by the SHG efficiency available in reported literature.

**Keywords:** Nonlinear optics;crystal growth techniques**;** structural analysis; optoelectronics application; Frequency Conversion Studies

1. **INTRODUCTION**

Young researchers have been inspired to look for novel materials with improved capabilities for photonics applications by the quick and high-speed data processing, retrieving, and transfer prevalent in today's information technology environment [1]. All these terms pointed towards the nonlinear optics (NLO). Actually, nonlinear optics is the study of how a strong electromagnetic field interacts with materials to produce modified fields that differ from the original field in phase, frequency, or amplitude [2]. When exposed to light, some materials undergo changes based on the direction, temperature, light wavelength, etc. In the present scenario, these materials become the essential part of the various applications like optical data processing, photonics, terahertz generation, ultra-fast switches, laser amplifiers etc. [3,4]. With the increasing demand of these materials, researchers pay their progressive attention for the discovery of new NLO materials which fulfill today’s requirement. On the basis of the properties of materials, these materials are categorized in three categories as– Organic, Inorganic and Semi-organic nonlinear optical materials [5]. Inorganic materials are thermally and mechanically stable but possessing low nonlinearity, whereas organic material is just opposite to them, which have high nonlinearity but low mechanical and thermal stability [6]. According to the new scenario and increasing demands of various industries, the properties of organic nonlinear materials can be modified with the use of chemical engineering techniques [7]. Need of improving the properties of these materials leads to existence of new category materials known as semi organic NLO materials which have high nonlinearity along with excellent mechanical and thermal stability. These materials are the crystalline solids which are of anisotropic nature i.e., they have different properties in different directions. The nonlinear material crystallizes in non-centrosymmetric space group which is essential for NLO behavior.

Amino acids are best suited to form NLO materials because of the two main properties i.e., zwitter ion formation and chirality [8,9]. As the amino acids has ability to form zwitter ions. The zwitter ionic behaviour of the molecule is responsible for the crystal hardness which is the main concept behind choosing this material [10]. One of the main properties of amino acids are that these have electron donor carboxylic group and electron accepter amino group [11,12]. One such amino acid that produces numerous variants of single crystals with NLO property is L-histidine. A number of researchers have recently grown and examined the characteristics of L-histidine derivative crystals, and identified them as potential candidate for nonlinear applications.

L-histidine is the amino acid with α-amino group. It has the special ability of having imidazole side chain. This side chain plays an important role in the reaction by acting as proton donor/ proton acceptor or nucleophilic reagent [13,18]. A plethora of nonlinear optical material of L-histidine has been reported by researchers. Some of these materials are as - L-histidine tetrafluoroborate (L-HFB) [14,15], L-histidine perchlorate [15], L-histidine nitrate (LHN) [16], L-histidine trifluoroacetate (LHTF) [17], L-Histidinium-4-nitrophenolate 4- Nitrophenol (LHPP) [18], L-histidine hydrochloride monohydrate (LHHC) [19], L- histidinium dinitrate [20], L-histidinium iodide [21] and many more. Single crystals of these materials have been grown by the researchers and checked their usefulness for various applications under structural, optical, thermal, mechanical and nonlinear property of the material. Structural properties of the material were studied through X-ray diffraction method. Researchers have also carried out UV-VIS-NIR spectroscopy for the studied compound. Under this spectroscopy technique transmittance, absorbance and reflectance were determined which are used to find the transparency, absorbance, energy band gap and refractive index of the compound [22]. Energy band gap is measured through Tauc’s curve which is the curve between (αhν)2  and energy where α is the absorption coefficient, it can be verified by Plank’s equation [23] i.e.,

Eg = $\frac{hc}{λ}$

Kurtz- Perry powder technique is used to calculate the second harmonic generation (SHG) value of nonlinear material. The SHG value represent the nonlinear behaviour of compound with reference to other material. Generally, Potassium dihydrogen phosphate (KDP) is taken as reference material for that purpose. Mechanical stability of the material is determined by subjecting the sample crystal against various indenter loads. Under Vickers hardness technique the Meyer’s index was calculated which signifies whether the material comes under the category of soft or hard material.

1. **NONLINEAR OPTICS**

Nonlinear optics is the study of the behaviour of highly intense light in the nonlinear medium. When intense light pass through this type of materials then new light of different wavelength has been collected from the other side [24]. This incoming light may have different frequency, phase, polarization and path as compared to the incident light. In the years thereafter, plenty of nonlinear effects in light-matter interaction have been experimentally confirmed and, in many cases, quickly found their way to market applications in a variety of disciplines ranging from telecommunications to imaging for health care and characterisation [25,26]. As the electromagnetic radiation passes through nonlinear materials then the electric vector of radiation effect it’s polarization (**P**). In this case polarization is expressed in power series of **E.**

**P**(t) **=** ɛ0**[** χ1 **E** (t) **+** χ2 **E2** (t) **+** χ3 **E3** (t) **+ …….]** [27]

Here, **P** is the net polarization, ɛ0 - absolute permittivity, χ – susceptibility coefficient, **E** stands for electric field vector.

In the above equation, first term has only single power of susceptibility coefficient and **E** (electric field vector) which shows the linear behaviour of the compound whether the second and third term has higher powers of χ and **E**, shows the nonlinear behaviour. Term having second power represent Second harmonic generation, on the other hand, third power term represent third harmonic generation of frequency by the compound. These terms in the series confirms the nonlinearity presents in the compound [28-30]. Fig. 1 represent the nonlinear phenomenon which shows the changing of single frequency to double frequency.



**Fig. :- 1 Nonlinear phenomenon**

1. **CRYSTAL GROWTH TECHNIQUES**

In order to crystallize the material, there are various crystal growth techniques ranging from simple & low-cost technique to complex & costly technique represented in figure -2. These techniques are broadly categorized into three categories which are as follows- vapour growth, melt growth technique, and solution growth technique.

1. **Vapour growth technique**

In this technique vapours are used to grow the crystals. Thus, phase change is from vapours to solid. There are two methods under this category- physical vapour transport and chemical vapour transport [31].

1. **Melt growth technique**

This technique contains two primary methods to grow the crystal i.e., fusion and solidification. Firstly, the solid raw salts are melted through fusion and the solidification of the material takes place to form the crystal. Here phase transition takes place from solid to solid [32].

1. **Solution growth technique**

It is the most common method of crystal growth. This method is based upon the precipitation from saturated solution. Here phase change takes place from liquid to solid. It is considered as superior method because it is the simplest and cheapest method by which good optical transparency of the crystal and uniform mixing of the raw salts in the lattice achieved easily [33-35].

Raw reagent Salt Synthesis Solution Crystal

The simplest method in this technique is Slow Evaporation Solution Growth technique (SEST). In which saturated solution of raw reagents has been prepared by taking them in particular sociometric ratio. This solution is then filtered through Whatman filter paper for separating the undissolved contaminants. This solution is covered with perforated polythene and left at room temperature for slow evaporation. After few days, single crystals have been collected from the solution.

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**Fig. :- 2 Crystal Growth Techniques**

1. **NONLINEAR OPTICAL CRYSTAL OF L-HISTIDINE FAMILY**

In the emerging area of photonics, nonlinear optics play an important role [36]. As with the demand of industries like frequency conversion, data processing, telecommunication and laser etc increases then NLO materials comes as a boon to fulfil their demands [37-39]. Many of the applications require materials with high second-order nonlinearities, small lower cut-off wavelengths, and stable physicochemical properties. For the use of material in various application the main requirement is that the materials must be transparent not only to the laser frequency but also to the newly created frequency, in addition to having significant nonlinearity. These materials should be resistant to optical damage, have suitable mechanical hardness, thermally and chemically stability, capable of being produced in usable sizes, and have the requisite phase - matching properties. For this purpose, properties of L-histidine family of materials are discussed in this article and are presented below.

1. **L-histidine bromide (LHB)**

L-histidine bromide NLO material was grown as single crystal through slow evaporation solution growth technique by Reena Ittyachan et al. [40] in 2003. LHB single crystal belongs to orthorhombic crystal system with P212121 space group. The lattice parameters of the grown crystal were determined through single crystal XRD analysis and found as- a = 7.0530 Å, b = 9.0409 Å, c = 15.2758 Å, α = β = γ = 90º, Whereas the cell volume comes out to be 974.0670 Å3. Optical studies of the compound show that for the titled compound the transmission takes place in visible region and the refractive index comes out to be 1.516. Nonlinear behaviour of material was determined through second harmonic generation test and SHG efficiency of LHB comes out to be 0.97 times that of KDP standard [41].

1. **L-Histidine-2-Fluoro-4-nitrophenolate 2-Fluoro-4-nitrophenol (LHFPFP)**

A novel single crystal of L-Histidine-2-Fluoro-4-nitrophenolate 2-Fluoro-4-nitrophenol organic compound was grown using economic slow evaporation method at room temperature by : R. Dhanjayan et al. [42] . The grown crystal has the cell parameter as- a = 8.9183 Å, b = 8.9258 Å, c = 12.3953 Å, α = γ = 90º and β = 102.712 º volume = 962.50 Å3. The material synthesized with monoclinic crystal system and P21 space group. Through optical analysis, band gap of the material is calculated which is found to be 2.54 eV. The SHG efficiency of LHFPFP was 1.57 times the KDP standard.

1. **L-Histidine dihydrogenarsenate orthoarsenic acid (LHA)**

L-Histidinium dihydrogenarsenate orthoarsenic acid (LHA) has been grown as single crystal by H. Ratajczak et al. [43] in 2000 with monoclinic crystal system and P21 space group. The crystal parameters are that a = 9.264 Å, b = 8.929 Å, c = 8.874 Å, α = γ = 90º and β = 108.61º cell volume = 695.7 Å3. SHG efficiency of the titled compound was 0.46 times the KDP. The mechanical analysis shows that LHAS is soft material with Meyer’s index 1.97 [44].

1. **L-histidinium Fumarate Fumaric acid Monohydrate (LHFFAM)**

Organic nonlinear optical single crystal has been synthesized and grown through slow evaporation technique. The structural analysis reveals that the crystal was synthesized in monoclinic crystal system with C2 space group. Unit cell dimensions of the grown crystal was a = 15.6985 Å, b = 6.7466 Å, c = 16.7772 Å, α = γ = 90º and β = 98.748º and the cell volume V = 695.7 Å3 . SHG efficiency of the grown crystal was 0.92 times that of KDP [45]. The optical properties were analysed through UV-VIS spectroscopy. This study signifies that the band gap of LHFFAM crystal is 3.9 eV and refractive index comes out to be 1.4664. LHFFAM comes under the category of soft material with Meyer’s index 2.75 [46].

1. **L-histidine methyl ester dihydrochloride (LHMED)**

Semi-organic L-histidine methyl ester dihydrochloride single crystal was grown with lattice parameters a = 8.221 Å, b = 7.108 Å, c = 9.505 Å, α = γ = 90º and β = 94.56º, volume (V) = 555.42 Å3. The crystal synthesized with monoclinic crystal system and P21 space group. Optical analysis reveals that band gap of the LHMED compound is 5.35 eV. The SHG efficiency of the material was 1.6 times higher than KDP [47].

1. **L-histidinium thiocyanurate thiocyanuric acid**

A single crystal of organic compound L-histidinium thiocyanurate thiocyanuric acid was synthesized by Mauro A. Pereira Gomcalves et al. in 2015 [48]. Through single crystal XRD structural data was collected and found that the crystal belongs to monoclinic system with P21 space group. The cell parameters of the grown crystal were as follows- a = 11.3096 Å, b = 6.94250 Å, c = 14.2779 Å, α = γ = 90º and β = 98.9193º, volume (V) = 1107.503Å3. To determine the nonlinear behaviour of the compound Kurtz and Perry powder technique was used and found that SHG efficiency is 0.37 times that of urea standard.

1. **L-histidine trichloro zinc (HZC)**

First metal organic L-histidine based single crystal of L-Histidinium trichloro zinc (HZC) was grown through slow evaporation technique at room temperature by Radhakrishnan Anbarasan et al. in 2018 [49]. Structural analysis reveal that this compound was synthesized in orthorhombic system with P212121 space group. The cell parameters are $13.656×9.871×8.6661$ Å with volume 1167.00 Å3. For HZC compound the band gap energy was calculated which found the value 3.45 eV. The SHG efficiency of the titled compound was comes out to be 1.4 times to that of standard KDP .

1. **L-histidinium perchlorate (LHPCl)**

The crystalstructure of semi-organic LHPCl single crystal was determined through single crystal XRD and shows that the compound is of monoclinic type with P21 space group. The cell dimensions of the compound were given as- a = 5.052 Å, b = 9.194 Å, c = 10.388 Å, α = γ = 90º and β = 92.34º with volume 482.1 Å3 [50]. Band gap shown by the optical analysis was 3.9 eV. The nonlinear behaviour was determined through NLO studies under second harmonic generation test. The SHG efficiency of the compound comes out to be 3.19 times of KDP standard [51]. Mechanical behaviour of LHPCl compound was determined through Vickers microhardness test and it signifies that the compound has Meyer’s index 2.31 which shows that the compound comes in the category of soft material [52].

1. **L-histidinium l-tartrate hemihydrate (LHT)**

Novel organic single crystal of L-histidinium l-tartrate hemihydrate has been grown for nonlinear optical applications by M.K. Marchewka et al. in 2003. The compound crystallizes in non-centrosymmetric (C2) space group with monoclinic system. XRD analysis provide the structural information of the material. The crystal data of the material is as- a = 23.002 Å, b = 7.676 Å, c = 7.657 Å, α = γ = 90º and β = 96.91º with volume 1342.1 Å3 [53]. Calculated optical band gap of LHT material is comes out to be 5.39 eV. Mechanical studies reveal that LHT crystal belong to the category of soft materials represented by Meyer’s index, which have the value 3.5. The second harmonic generation efficiency of the material is 0.5 time that of standard KDP [54].

1. **l-Histidinium chloroacetate (LHCA)**

Christuraj Paul chinnappan et al. synthesized a new semi-organic singe crystal of L-Histidinium trichloroacetate with non-centrosymmetric space group P1 under triclinic system. The cell parameters are **a** = 6.883 Å, **b** = 8.981 Å, **c** = 15.386 Å, **α** = 90.014, **β** = 89.989º and γ = 89.99º with volume 951.10 Å3. LHCA is optically transparent material with band gap 3.52 eV. Through mechanical studies it is confirmed that LHCA is a soft material with Meyer’s index 4.28. The SHG efficiency is 1.8 times to that of KDP [55].

1. **L-histidinium tetrafluorophthalate (LHFP)**

LHFP material has been grown as single crystal by slow evaporation solution growth technique. Cell dimensions were determined through X-ray diffraction and is given as- a = 15.602 Å, b = 5.050 Å, c = 16.152 Å. α = γ = 90º, β = 92.328º, volume V= 1297.006 Å3. The transparency of the crystal is observed in entire visible region. Work hardening coefficient of LHFP is 4.3 which results that the titled compound is soft material. SHG efficiency is 0.2 times that of urea standard [56].

1. **L-histidine L-aspartate monohydrate (LHLAM)**

Semi-organic novel L-histidine L-aspartate monohydrate single crystal was synthesized and grown by CG Suresh et al. [57,58] at room temperature through slow evaporation method with P21 space group and cell dimensions $5.131×6.881×18.277 Å$ and cell volume V = 632.820 Å3. With angles α = γ = 90º, β = 94.19º. LHLAM crystal shows maximum transparency in UV and visible region. The SHG efficiency of LHLAM material is measured through powdered sample using Kurtz and Perry technique and found that it has value 3.8 times that of KDP standard [58].

1. **Results and discussion**

For the growth of the NLO crystals, one of the methods of crystal growth that is widely prevalent in the creation of many crystals, crystals from aqueous solution, was used. Slow evaporation solution growth technique seems economic and best suited for producing NLO crystals. Various properties of L-histidine based single crystals are investigated by researchers from application standpoint. Here, in this paper, the work on the L-histidine family of NLO single crystals is summarised. The structural information, space group and the relative SHG of grown crystals are summarized in Table -1

**Table-1 L-histidine family single crystal properties**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Compound** | **Cell dimensions (**$Å$**)** | **Space group** | **SHG****(KDP)** | **Band gap (eV)** | **Meyer’s Index** | **References** |
| LHB | 7.0530$×9.0409 ×15.275$ | P212121 | 0.97 | - | - | 40,41 |
| LHFPFP | 8.9183$ ×8.9258 ×12.3953$ | P21 | 1.57 | 2.54 | - | 42 |
| LHA | $$9.264 ×8.929 ×8.874$$ | P21 | 0.46 | - | 1.97 | 43 |
| LHFFAM | $$15.6985 ×6.747 ×16.777$$ | C2 | 0.92 | 3.9 | 2.75 | 45 |
| LHMED | $$8.221 ×7.108 ×9.505$$ | P21 | 1.6 | 5.35 | - | 47 |
| HZC | $$13.656 ×9.871 ×8.6661$$ | P212121 | 1.4 | 3.45 | - | 49 |
| LHPCl | $$5.052 ×9.194 ×10.388$$ | P21 | 3.19 | 3.9 | 2.31 | 50,51 |
| LHT | $$23.002 ×7.676 ×7.657$$ | C2 | 0.5 | 5.39 | 3.5 | 53,54 |
| LHCA | $$6.883 ×8.981 ×15.386$$ | P1 | 1.8 | 3.52 | 4.28 | 55 |
| LHFP | $$15.602 ×5.050 ×16.152$$ | - | 0.2(urea) | - | 4.3 | 56 |
| LHLAM | $$5.131 ×6.881 ×18.277$$ | P21 | 3.8 | - | - | 58 |

**Conclusion**

L-Histidine family of single crystals is analysed in this review article. With structural investigation it is found that all the compounds crystallized in non-centrosymmetric space group which confirm that it is good material for various nonlinear optical applications. The second harmonic generation study confirms the NLO applications of the material. This article provides new ideas to the researchers for discovering many and more NLO materials for satisfying the demands of various industrial applications.

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