L-HISTIDINE FAMILY SINGLE CRYSTAL FOR FREQUENCY MATCHING APPLICATIONS

Abstract

This article reviews the L-histidine family single crystal properties. Here, the techniques, through which better sized good quality single crystal has been grown in economic manner is also reported. This paper includes the structural review of the materials which gives the cell parameters of grown crystal. For determining the optoelectronics applications of crystal UV-Vis-NIR studies are analyzed. Mechanical strength of the material is determined through Vickers hardness test. Nonlinear behavior of the material has been analyzed by the SHG efficiency available in reported literature.

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

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I. INTRODUCTION

The rapid data processing, retrieval, and transfer typical of today's IT environment [1] has spurred a new generation of researchers to seek for innovative materials with increased capabilities for photonics applications. Nonlinear optics (NLO) was the subject that these phrases were referring to. When a powerful electromagnetic field interacts with a material, it creates new fields that are different in phase, frequency, and amplitude from the original field [2]. This phenomenon is focusing the nonlinear optics. Some materials change when exposed to light, with the details depending on factors like direction, temperature, light wavelength, etc. Applications such as data processing, photonics, THz generation, laser amplifiers, and many more [3,4] now rely heavily on these materials. Researchers are progressively focusing on finding novel NLO materials to meet the ever-increasing demand for such substances. There are three types of nonlinear optical materials based on their composition: organic, inorganic, and semi-organic [5]. Inorganic materials have great mechanical and thermal stability but low value of nonlinearity [6], whereas organic materials have efficient nonlinear properties but significant mechanical and thermal instability. Chemical engineering methods may be used to alter the characteristics of organic nonlinear materials to meet the evolving needs of a wide range of businesses [7]. In response to the need for enhanced performance, new materials named as semi organic NLO materials has emerged. These include significant nonlinearity in addition to superior mechanical and thermal stability. Anisotropic materials are crystalline solids that exhibit directional dependence on their characteristics. For NLO behavior, it is necessary that the nonlinear material crystallize in a space group that must be non-centrosymmetric.

Amino acids are capable to grow crystals with non-centrosymmetric space group. The two key features, zwitter ion production and chirality make amino acids ideal for forming NLO materials [8,9]. The key idea for selecting this material is its crystal hardness [10], which is in turn caused by the zwitter ionic behaviour of the molecule. Amino acids are distinguished in part by the presence of a carboxyl group (an electron donor) and an amino group (an electron acceptor) [11,12]. An amino acid, L-histidine forms a wide variety of single crystals with the NLO characteristic. A number of researchers have synthesized the crystals of L-histidine derivatives, analysed their properties, and determined that they are promising candidates for nonlinear applications.

L- Histidine is α-amino group amino acid. Because of its imidazole side chain, it has unique properties. In the reaction, this side chain functions as a nucleophilic reagent, proton donor, or proton acceptor [13]. Researchers have identified a multitude of L-histidine nonlinear optical materials. Some of these materials are as - L-histidine bromide (LHB), L-histidine -2- Fluoro -4- nitrophenolate -2- Fluoro -4- nitrophenol (LHFPFP), L- Histidine dihydrogenarsenate orthoarsenic acid (LHA), L-histidinium fumarate fumaric acid monohydrate (LHFFAM), L-histidine methyl ester dihydro chloride (LHMED), L-histidinium trichloro zinc (HZC), L- histidinium perchlorate (LHPCl), L-histidinium l-tartrate hemihydrate(LHT), L-Histidinium chloroacetate (LHCA), L-histidinium tetrafluorophthalate (LHFP), L-histidine L-aspartate monohydrate (LHLAM) [14-24] and many more. Researchers have grown the single crystals of these materials and evaluated them for potential uses by examining their properties for nonlinear optical applications. The X-ray diffraction technique was used to investigate the material's structural characteristics. The material has also been subjected to UV-Vis-NIR spectroscopy. Transmittance, absorbance, and reflectance were calculated using this spectroscopic method, yielding information on the compound's transparency, absorbance, energy band gap, and refractive index [24]. Tauc's curve, the plot between $(\alpha h\nu)^2$ and energy, is used to quantify the energy gap, which is confirmed by Plank's equation [25], i.e.

$$\mathbf{E_g = \frac{hc}{\lambda}}$$

The value of the nonlinear efficiency of second harmonic generation (SHG) is determined using the Kurtz- Perry powder approach. Nonlinear behaviour of a compound relative to another substance is represented by the SHG value. In most cases, KDP (potassium dihydrogen phosphate) is used as a standard for this purpose. Crystal samples are tested under a range of indenter stresses to establish the material's mechanical stability. The Meyer's index of the sample crystal was determined, which indicates whether the crystal material is soft or hard.

II. NONLINEAR OPTICS

The field of nonlinear optics investigates how intense light behaves in a nonlinear material. When intense light pass through this type of materials then new light of different wavelength has been collected from the other side [26]. The characteristics of this reflected light may vary from those of the incident light, including its frequency, phase, polarization, and even its path. Nonlinear phenomena in light-matter interaction have been empirically validated in the years thereafter, with many finding rapid commercial use in fields as disparate as telecommunications and medical imaging and characterization [27,28]. The polarization (**P**) of electromagnetic radiation effect, when it travels through nonlinear materials due to the influence of the electric vector of radiation. Here, power series of **E,** serve as an expression of polarization (**P**), as is given below [29].

$$\mathbf{P(t) = \varepsilon_0 [\, \chi^1 E(t) + \chi^2 E^2(t) + \chi^3 E^3(t) + \ldots\ldots]}$$

Here, **P** is the net polarization, ε_0 - 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 [30-32]. Fig. 1 represent the nonlinear phenomenon which shows the changing of single frequency to double frequency.

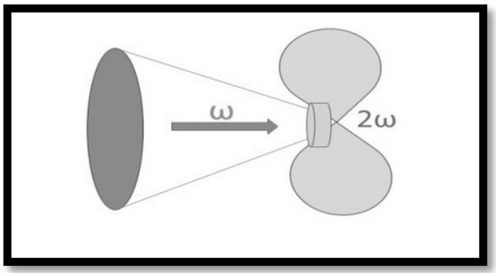

Figure 1: Nonlinear phenomenon

III. 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 [33].

2. **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 [34].

3. **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 [35-37].

Raw reagent ⟹ Salt Synthesis ⟹ Solution ⟹ Crystal

This approach, contains the simplest method of crystal growth i.e., Slow Evaporation Solution Growth technique (SEST). Using this method, a saturated solution of the raw reagents has been prepared by taking them in specific sociometric ratio. The undissolved substances are subsequently separated from the solution by filtering it via Whatman filter paper. Perforated poly sheet is used to cover the solution and allow for gradual evaporation at room temperature (RT). Grown single crystals have been collected from solution after few days.

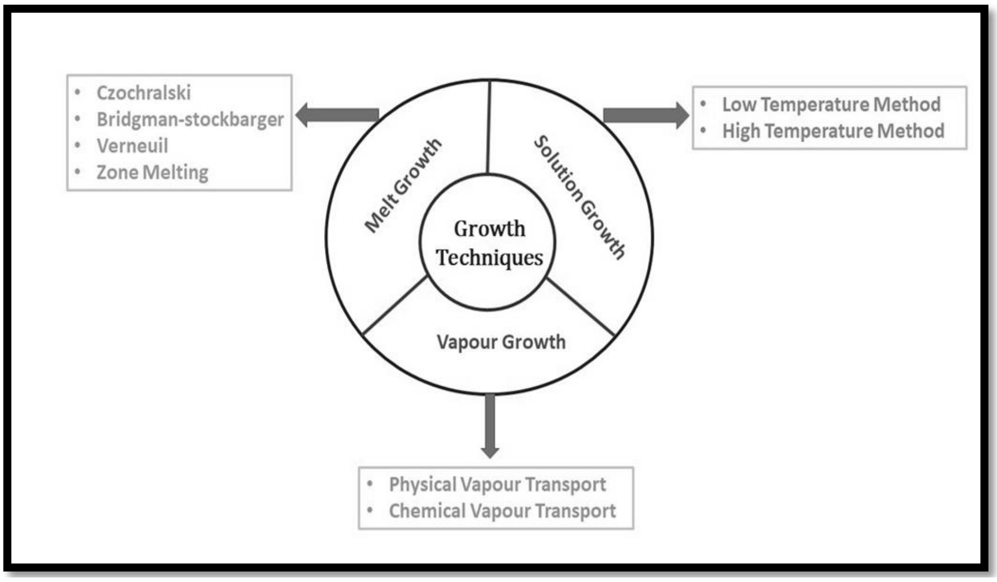

Figure 2: Crystal Growth Techniques

IV. NONLINEAR OPTICAL CRYSTAL OF L-HISTIDINE FAMILY

In the emerging area of photonics, nonlinear optics play an important role [38]. 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 [39-41]. 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, thermal and chemical 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. **LHB:** The **LHB**, NLO material was grown as single crystal through SEST method by Reena Ittyachan et al. [14] in 2003. The lattice parameters of the grown crystal were determined through SCXRD having the value of a, b, c as - 7.0530 Å, 9.0409 Å and 15.2758 Å respectively, α = β = γ = 90º, Whereas the cell volume was 974.0670 Å^3. This crystal belongs to orthorhombic system with space group $P2_12_12_1$. 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 SHG test which gives the efficiency value 0.97 times that of KDP standard [42].

2. **LHFPFP:** A single crystal of **LHFPFP** organic compound was grown using economic SEST method at RT by : R. Dhanjayan et al. [15]. The cell parameter of grown crystal i.e., a, b and c have value 8.9183 Å, 8.9258 Å, and 12.3953 Å respectively, with angles α = γ = 90º and β = 102.712 º, cell volume = 962.50 Å^3. The material belongs to non-centrosymmetric $P2_1$ space group under monoclinic crystal system. Optical analysis is used to calculate the band gap, which is 2.54 eV for LHFPFP. The value of SHG for LHFPFP is 1.57 times the KDP standard.

3. **LHA: LHA** has been grown as single crystal by H. Ratajczak et al. [16] in 2000 with monoclinic system and $P2_1$ space group. The SCXRD study provide the structural information of compound. it shows that the dimensions of LHA crystal are (9.264 × 8.929 × 8.874)Å, angles are α = γ = 90º and β = 108.61º and the cell volume = 695.7 Å^3. The efficiency of SHG for LHA compound is 0.46 times the KDP. The mechanical analysis reveals that it is soft material with Meyer's index 1.97 [43].

4. **LHFFAM:** The SEST approach has been used to successfully synthesis and develops the titled single crystal. Analysis of the crystal structure indicates that the material is grown using the monoclinic system and the C2 space group. Unit cell dimensions of grown crystal was 15.6985× 6.7466 ×16.7772 Å, α = γ = 90º and β = 98.748º and the cell volume V = 695.7 Å^3 . SHG efficiency was 0.92 times the standard KDP [17]. The optical properties were analysed through UV-Vis spectroscopy. This study signifies that LHFFAM crystal has band gap 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 [44].

5. **LHMED:** Single crystal of semi-organic LHMED material was grown with lattice parameters a, b and c as- 8.221 Å, 7.108 Å, and 9.505 Å respectively, with angles α = γ = 90º and β = 94.56º. The crystal synthesized under monoclinic system and $P2_1$ space group. Optical analysis reveals that band gap of the LHMED compound is 5.35 eV. The SHG value of the material is 1.6 times that of standard KDP [18].

6. **L- Histidinium Thiocyanurate Thiocyanuric Acid:** The L-histidinium thiocyanurate thiocyanuric acid was synthesized primarily by Mauro A. Pereira Gomcalves et al. in 2015 [45]. Through single crystal XRD structural data was collected and found that the crystal has monoclinic system with space group $P2_1$. The cell parameters of the grown crystal were as follows- a = 11.3096 Å, b = 6.94250 Å, c = 14.2779 Å, with β = 98.9193º, volume (V) = 1107.503Å^3. To determine the nonlinear behaviour of the compound SHG efficiency is determined which that of urea standard is 0.37 times.

7. **HZC:** Firstly, metal organic L-histidine based crystal of HZC was grown through SEST method by Radhakrishnan Anbarasan et al. in 2018 [19]. Structural analysis reveal that the compound was synthesized with orthorhombic system along with space group $P2_12_12_1$. The cell parameters are $13.656 \times 9.871 \times 8.6661$ Å with volume 1167.00 Å^3. For HZC compound, the band gap energy was calculated which got the value 3.45 eV. The aforementioned compound has SHG value 1.40 times that of KDP.

8. **LHPCl:** The crystal structure of semi-organic LHPCl single crystal was determined through SCXRD and shows that it is of monoclinic type crystal with space group $P2_1$. The cell dimensions of the compound were given as- 5.052 × 9.194 × 10.388 Å, α = γ = 90º and β = 92.34º with volume 482.1 Å^3 [20]. 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 efficiency of SHG for this compound comes out to be 3.19 times of KDP standard [46]. Mechanical behaviour of LHPCl compound was determined through microhardness test that signifies the value of Meyer’s index of compound is 2.31 which shows that the compound comes in the category of soft material [47].

9. **LHT:** For NLO applications, M.K. Marchewka et al. (2003) developed single crystal of LHT. The chemical forms crystals in the monoclinic (C2) space group, which is non-centrosymmetric. XRD analysis provides the structural information of the material. The crystal data of the material is as- 23.002 × 7.676 × 7.657 Å, and β = 96.91º with volume 1342.1 Å^3 [21]. Calculated band gap of LHT material is 5.39 eV. Mechanical studies reveal that LHT crystal belong to soft materials category confirmed by Meyer’s index, which have the value 3.5. The SHG value of the material is 0.5 time the KDP standard [48].

10. **LHCA:** Christuraj Paul chinnappan et al. synthesized a semi-organic crystal of LHCA with non-centrosymmetric P_1 space group under triclinic crystal system. The unit cell parameters of the crystal a ,b, and c are - 6.883 Å, 8.981 Å, and 15.386 Å respectively, angles are given as α = 90.014, β = 89.989º and γ = 89.99º with volume 951.10 Å^3. 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 value for LHCA is 1.8 times the KDP standard [22].

11. **LHFP:** For the synthesisation of LHFP crystal, SEST method has been employed. Cell dimensions of the crystal are determined through X-ray diffraction and is given as- $15.602\times 5.050 \times 16.152\,\AA$ having angles α = γ = 90º, β = 92.328º and volume V= 1297.006 $\AA^3$. The crystal transparency of LHFP is observed in the visible region. Work hardening coefficient of the compound is 4.3 which results that the titled compound is soft material. The nonlinear behaviour is determined through SHG value, which is 0.2 times the urea standard [23].

12. **LHLAM:** Semi-organic L-histidine L-aspartate monohydrate single crystal was synthesized and grown by CG Suresh et al. [24,49] at room temperature through slow evaporation method with $P2_1$ space group and cell dimensions $5.131 \times 6.881 \times 18.277\,\AA$ and cell volume V = 632.820 $\AA^3$. With angles α = γ = 90º, β = 94.19º. LHLAM crystal shows maximum transparency in UV and visible region. The efficiency of SHG for LHLAM is measured through powdered sample and found that it has value 3.8 times that of KDP standard [24].

V. 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 saturated solution, was used. SEST method 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 L-histidine 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	$7.0530\times 9.0409 \times 15.275$	$P2_12_12_1$	0.97	-	-	14,42
LHFPFP	$8.9183 \times 8.9258 \times 12.3953$	$P2_1$	1.57	2.54	-	15
LHA	$9.264 \times 8.929 \times 8.874$	$P2_1$	0.46	-	1.97	16
LHFFAM	$15.6985 \times 6.747 \times 16.777$	C2	0.92	3.9	2.75	17
LHMED	$8.221 \times 7.108 \times 9.505$	$P2_1$	1.6	5.35	-	18
HZC	$13.656 \times 9.871 \times 8.6661$	$P2_12_12_1$	1.4	3.45	-	19
LHPCl	$5.052 \times 9.194 \times 10.388$	$P2_1$	3.19	3.9	2.31	20,46

LHT	$23.002 \times 7.676 \times 7.657$	C2	0.5	5.39	3.5	21,48
LHCA	$6.883 \times 8.981 \times 15.386$	P_1	1.8	3.52	4.28	22
LHFP	$15.602 \times 5.050 \times 16.152$	-	0.2(urea)	-	4.3	23
LHLAM	$5.131 \times 6.881 \times 18.277$	$P2_1$	3.8	-	-	24

VI. 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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