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Synthesis growth and characterization of a crystal l-histidine sodium nitrate

R Ashwini[1], Dr. D Mohan Radheep[2]
[1] M.Phil Research Scholar, Department of Physics, PRIST University, Puducherry, Tamil Nadu, India
[2] Associate Professor and Head, Department of Physics, PRIST University, Puducherry, Tamil Nadu, India

Abstract
Now-a-days technology is fully occupied by the semiconductor materials, piezoelectric, ferroelectrics, sensitive crystals and crystalline films. The significance of crystal growth both in theoretical and experimental aspects can be realized by developing such above cited single crystal materials. The preparation of single crystal is mainly based on the availability and nature of the starting materials and their physical-chemical properties. The present photonic technology mainly depends on the organic non-linear optical (NLO) materials.
The idea of combining the in-organic molecules with the organic molecules results in production of new non-linear crystals. These crystals are ideally had large non-linearity of conjugated organics and favorable crystal growth characteristics and mechanical properties of ionic salts. In the present investigation, large size crystal of L-Histidine sodium nitrate has been successfully grown by slow evaporation technique. The grown crystals were colourless and transparent.
The grown crystals were characterized by single crystal and powder X-ray diffraction to find the structure of crystals. The FT-IR analysis was made to identify the functional groups. The UV/Visible absorption studies were carried out to determine the optical transparency of the crystals. The dielectric and SHG studies have been carried out. The mechanical behavior of the grown crystal was analysed by micro hardness test.

Keywords: L-histidine sodium nitrate, X-ray, non-linear optical, semiconductor, piezoelectric, ferroelectrics, crystalline films

1. Introduction

We live in a nonlinear world. While linearization beautifies physics, nonlinearity provides excitement in physics. In recent years, significant advances have been made in the field of nonlinear optics and optical computing. These areas have grown rapidly in the last decade, so that today these have emerged as important disciplines in their own right. Optics is already preferred for many applications owing to its wide bandwidth and autonomy from electromagnetic interference. Since fiber optic cable has low absorption, they can transmit signals for several kilometers, but the signal must be amplified. It is more efficient if the light beam is amplified directly by a laser beam in a suitable medium. Such a process has come into the realm of nonlinear optics. The science that deals with the interactions of a laser beam with an optical medium is called nonlinear optics and the medium itself is called a nonlinear optical (NLO) material.

Since the invention of the first laser 40 years ago, the frequency conversion of laser radiation in nonlinear optical crystals has become an important technique widely used in quantum electronics and laser physics for solving various scientific and engineering problems. The fundamental physics of the wave light interactions in nonlinear optical crystals is now largely understood.

This has enabled the production of the various harmonic generators, sum and difference frequency generators, and parametric oscillators based on nonlinear crystals that are now commercially available. At the same time, scientists continue an active search for novel high-frequency optical materials. The propagation of electromagnetic waves through nonlinear media gives rise to vibrations at harmonics of the fundamental frequency, at sum and difference frequencies, and so on. When one or more sufficiently powerful beams of laser radiation pass through the dielectrics, the frequency of the radiation may be transformed to the second, third and higher harmonics and to combination (sum and difference) frequencies. In this way, the range of wavelengths generated by a certain laser source can be considerably increased.

Now, after 40 years of research with NLO materials, it is possible to cover almost continuously the range from 170 nm to 18 μm. Furthermore, the transparency region of NLO crystals now reaches 165 nm to 125 μm. As a result, further extension of applications to the ultraviolet (UV) and far-infrared regions will be possible. However, materials limitations are significantly slowing the development of required optical devices.

The 'ideal' nonlinear crystal does not exist. Only a very few crystals in practice are found to satisfy the major requirements and out of thousands of nonlinear crystals studied, only a handful of crystals have been found to be suitable in some way or other. The applicability of a particular crystal depends on the nonlinear process used, the desired device characteristics and the pump laser. Special material properties that are important in one application may not be significant in another. For a material that has favourable features such as large nonlinearity, high damage threshold, favourable crystal growth habits etc., an application can invariably be found that uses the crystal efficiently.

During the last four decades, optics has made rapid inroads into application areas previously dominated by electronics. In applications where bandwidth and electrical interference are presenting severe limitations on electronics, fiber based communication systems are considered as viable alternatives. The study of linear and nonlinear optical properties of materials has led to the development of a large number of optical devices. Nonlinear optical materials will be the key elements for future photonic technologies because of the fact that photons are capable of carrying information at the speed of light. It may be stated without doubt that while electronics revolution dominated the twentieth century technologies, it is strongly believed that photonics may dictate the technological trends in the twenty first century. A nonlinear optical material has many applications. Materials which can generate highly efficient second harmonic light by laser are of great interest in various applications including telecommunications, optical computing, optical information processing, optical disk, laser remote sensing, laser driven fusion, color displays and medical diagnostics. Materials with large nonlinearities, short transparency cut-off wavelengths and with stable physicochemical properties are needed for these applications.

Single crystals play a vital role in most of the optics based applications. So the growth of single crystals and their characterization towards device fabrication are important in both basic and applied research. Crystal growth is an interdisciplinary subject covering physics, chemistry, electrical engineering, metallurgy, crystallography, mineralogy etc. In the past few decades, there has been a growing interest in crystal growth process, particularly in view of the increasing demand for materials for technological applications. The strong influence of single crystals in the present day technology is evident from the 5 recent advancements in the fields of semiconductors, polarizers, transducers, infrared detectors, ultrasonic amplifiers, ferrites, magnetic garnets, solid state lasers, nonlinear optic, piezoelectric, acousto-optic, photosensitive materials and crystalline thin films and the computer industry. Hence, to achieve higher performance in the fabricated devices, good quality single crystals are needed.

The overwhelming success of molecular engineering on controlling nonlinear optical (NLO) properties has attracted the attention of the researchers to search for a variety of new types of nonlinear optical materials and to improve the NLO efficiency of the known materials. In the context of NLO, organic materials have advantages such as large NLO coefficients and structural diversity or flexibility, compared to the inorganic counterparts. They also have some inherent drawbacks, for example, poor physico-chemical stability and low mechanical strength.

As a result, the quest for new frequency conversion materials is presently concentrated on semi-organic crystals due to their large nonlinearity, high resistance to laser induced damage, low angular sensitivity and good mechanical hardness. semi-organics include organic–inorganic salts and metal–organic coordination compounds. Among these classes of materials, amino acids are interesting and useful materials for NLO applications. The salts of basic amino acid L-histidine gain much interest as promising nonlinear optical materials after the early works of Marcy *et al*., that the nonlinearity of L-histidine tetra flouroborate is much greater than that of potassium dihydrogen phosphate.

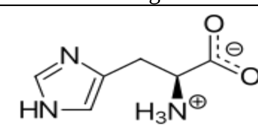
Due to its basic nature, L-histidine forms a number of salts with different organic and inorganic acids which have shown NLO properties. As a result, very good semi organic nonlinear optical materials such as L-valine hydrochloride [8], L-arginine phosphate monohydrate (LAP), L-histidine hydrochloride, L-alanine cadmium chloride and L-valine cadmium chloride are some of the good examples which proved very suitable materials for NLO applications

In this chapter, the author discusses the l-histidine sodiumnitrate development of the nonlinear optical crystals along with the theory of nonlinear optics and the various methods of growing these crystals and in particular the solution growth method

2. Terials and Methods

2.1 L-Histidine Properties

Table 1

Chemical Name	L-Histidine
Molecular Formula	C6H9N3O2
Molecular Weight	155.16 g/mol
Structure	
Physical Description	white powder
Melting Point	25^{o}c
Solubility	Soluble in water

L-Histidine is an essential amino acid that is physiologically active in the L-form.

Histidine is an α-amino acid that is used in the biosynthesis of proteins. It contains an α-amino group ($–NH_3^+$), a carboxylic acid group ($–COO^−$), and an imidazole side chain, classifying it as a positively charged amino acid at physiological pH. Initially thought essential only for infants.

Histidine was first isolated by German physician Albrecht Kossel and Sven Hedin in 1896. It is also a precursor to histamine, a vital inflammatory agent in immune responses. The acyl radical is histidyl

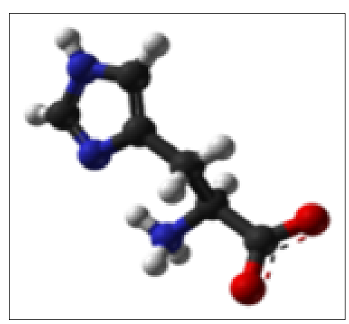


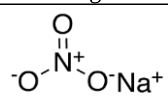
Fig 1

L-Histidine Uses and Benefits

- L-Histidine is used in the treatment, control, prevention, & improvement of the following diseases, conditions and symptoms:
- Allergic diseases
- Ulcers
- Anemia caused by kidney failure or kidney dialysis

2.2 Sodium Nitrate

Table 2

Chemical Name	Sodium nitrate
Molecular Formula	NaNO3
Molecular Weight	84.99 g/mol
Structure	
Exact Mass	84.99 g/mol
Density	2.257 g/cm^3, solid
Melting point	308 °C (586 °F; 581 K)
Solubility	Solubility in water

Sodium nitrate with chemical formula Na $(NO)_3$ This alkali metal nitrate salt is also known as Peru saltpeter to distinguish it from ordinary saltpeter, potassium nitrate. The mineral form is also known as nitratine, nitratite or soda niter.
Sodium nitrate is a white solid very soluble in water. It is a readily available source of the nitrate anion (NO_3^-),

Sodium nitrate

Industrial scales for the production of

- Fertilizers,
- Pyrotechnics and smoke bombs,
- Glass and potteryenamels, food preservatives (esp. meats), and
- Solid rocket propellant

3. Methods of Crystal Growth

The consistency in the characteristics of devices fabricated from the crystals depends on the homogeneity and defect contents of the crystals. Hence, the process of producing single crystals, which offers a homogeneous media in the atomic level with directional properties, attracts more attention than any other process. The methods of growing crystals are very wide and mainly dictated by the characteristics of the material and its size [1]. The methods of growing single crystals may be classified according to their phase transformation as given below.

Growth from Solid Solid → solid phase transformation
Growth from liquid Liquid → solid phase transformation
Growth from vapour Vapour → solid phase transformation

The conversion of a polycrystalline piece of material into a single crystal by causing the grain boundaries to be swept through and pushed out of the crystal takes place in the solid-growth of crystals.
An efficient process is one, which produces crystals adequate for their use at a minimum cost. The growth method is essential because it suggests the possible impurity and other defect concentrations. Choosing the best method to grow a given material depends on material characteristics.

3.1 Growth from Solution

The crystal growth from liquid can be classified into four categories namely

a. Melt growth
b. High temperature solution growth (Flux growth)
c. Hydrothermal growth and
d. Low temperature solution growth

There are a number of growth methods in each category. Among the various methods of growing single crystals, solution growth at low temperature occupies a prominent place owing to its versatility and simplicity. Growth from solution occurs close to equilibrium conditions and hence crystals of liquid perfection can be grown. Study of anisotropy of the properties of crystals requires specimens cut in different orientations from the same single crystal. This can be easily done from crystals of large size.

3.2 Growth from Melt

This method is very popular because the growth rate of crystals grown by this method is quite high. The preferential role of the electrochemical process responsible for the change in composition of the crystals when they grow in melt in an applied field has been studied [2]. In this method, the following popular techniques are used.

a. Bridgman technique
b. Czochralski technique
c. Zone melting technique and
d. Verneuil technique

3.3 High Temperature Growth (Flux Growth)

Flux and hydrothermal growths form the category of high temperature solution growth. In this method, a solid (molten salt/flux) is used as the solvent and the growth takes place well below the melting point [3] of the solute. This technique can be applied to incongruent melting materials. Mixed crystals of solid solution can also be grown by the choice of optimum growth parameters.
This technique can be used for the crystallization of oxide compounds which generally have high melting points as well as for materials which have phase transitions below the melting point [4]. The crystals grown from melt will have lower concentration of equilibrium defects and lower dislocation density

3.4 Hydrothermal Growth

A number of metals, metal oxides and other compounds, practically insoluble in water up to its boiling point, show an appreciable solubility when the temperature and pressure are increased well above 100 ^{o}C and 1 atmosphere respectively. The requirements of high pressure cause practical difficulties and there are only a few crystals of good quality and large size grown by this technique. Quartz is the crystal grown industrially by this technique.

3.5 Low Temperature Solution Growth
Low temperature solution growth is the most widely used method for the growth of single crystals, when the starting materials are unstable at high temperatures [5]. This method demands that the materials must crystallize from solution with prismatic morphology. In general, this method involves seeded growth from a saturated solution. The driving force i.e., the supersaturation is achieved either by temperature lowering or by solvent evaporation. This method is widely used to grow bulk crystals, which have high solubility and have variation in solubility with temperature [6]. After many modifications and refinements, the process of solution growth now yields good quality crystals for a variety of applications. Growth of crystals from solution at room temperature has many advantages over other growth methods though the rate of crystallization is slow. Since growth is carried out at room temperature, the structural imperfections in solution grown crystals are relatively low.
Low temperature solution growth can be subdivided into the following methods:

a. Slow cooling technique
b. Slow evaporation technique and
c. Temperature gradient technique

3.6 Slow Cooling Method
This is the most suitable method among various methods of solution growth. However, the main disadvantage of slow cooling method is the need to use a range of temperatures. The possible range of temperature is usually narrow and hence much of the solute remains in the solution at the end of the growth run. To compensate this effect, large volume of solution is required. The use of wide range of temperature may not be desirable because the properties of the grown crystals may vary with temperature. Temperature stability may be increased by keeping the solution in large water bath or by using a vacuum jacket. Achieving the desired rate of cooling is a major technological difficulty. This technique needs only a vessel for the solution in which the crystals grow. The height, radius and volume of the vessel are so chosen as to achieve the required thermal stability. Even though this method has technical difficulty of requiring a programmable temperature controller, it is widely used with great success.

3.7 Slow Evaporation Technique
As far as apparatus is concerned, slow cooling and slow evaporation methods are similar in nature. In this method, the saturated solution is kept at a particular temperature and provision is made for evaporation. If the solvent is non-toxic like water, it is desirable to allow evaporation into the open atmosphere. Typical growth conditions involve a temperature stabilization of about 0.05oC and rate of evaporation of a few mm3/h. The evaporation technique has an advantage viz. the crystals grow at a fixed temperature. But inadequacies of the temperature control system still have a major effect on the growth rate. This method can effectively be used for materials having very low temperature coefficient of solubility. But the crystals tend to be less pure than the crystals produced by slow cooling technique, as the size of the crystal increases more impurities find place in the crystal faces. Evaporation of solvent from the surface of the solution produces high local supersaturation and unwanted nuclei are formed. Small crystals are also formed on the walls of the vessel near the surface of the liquid from the material left after evaporation. They tend to fall into the solution and hinder the growth of the crystal. Another disadvantage lies in controlling the rate of evaporation. A variable rate of evaporation may affect the quality of the crystal. In spite of all these disadvantages, this is simple and convenient technique of growing single crystals of large size.

3.8 Temperature Gradient Method
This method involves the transport of the materials from hot region containing the source material to be grown to a cooler region where the solution is supersaturated and the crystal grows.

3.9 Gel Growth
This is a very convenient laboratory process. Henisch (1988) [7] gave an excellent survey of the process. Only small crystals can be grown by this technique. Gels are two-phase systems comprising a porous solid with liquid filled pores. The pore dimensions depend on the concentration of the gel material. The most frequently used gels are based on silica. But gels based on gelatine, various soft soaps and pectin are also used. Seed crystals should be used to reduce flaws in the center of the crystals. It is very important to provide constant ambient temperature. It has been shown that the application of electric fields within the gel can influence the crystal growth processes.

4. Conclusion
Single crystals of L-Histidine sodium nitrate (LHSN) were grown by slow evaporation technique within a period of 70-85 days. The single crystal XRD data proves that LHSN crystal is monoclinic C in structure with space group of P_{121}:$P_{112.}$ And the powder XRD proves their phase difference. The FT-IR studies confirm the existence of L-Histdine in its salt form with nitrate. From the optical absorption study confirm that the crystal has lower cut off wavelength 243 nm and the band gap energy is E= 5.452303 J which reveals that the crystal is highly suitable for NLO application. The NLO studies confirm the SHG efficiency of the semi-organic compound LHSN is about 0.42 times as larger as that of the standard KDP crystal. Dielectric studies infer the possible usage of LHSN for photonics devices. The mechanical behavior of the LHSN crystal shows that the LHSN belongs to the hard category.

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