

Growth and Characterization of Semi Organic Single Crystal – Urea Doped Tartaric Acid Potassium Bromide

Abstract

Semi organic nonlinear optical crystal Urea doped Tartaric acid Potassium Bromide was grown by solution growth method. By subjecting the sample to single crystal X-ray diffraction the unit cell parameters of the grown single crystal was calculated. The optical characters were determined by UV-Vis spectral studies. Thermal stability of the crystal was found by TG/DTA analysis. The various functional group of the grown crystal was measured using Fourier Transform Infrared Spectroscopy (FTIR). Second harmonic generation efficiency of Tartaric Acid Potassium bromide single crystal was tested using Nd: YAG laser and it was found to be 0.7 times that of potassium di hydrogen phosphate (KDP crystal).

Keywords: Semi Organic NLO Crystal; Transmittance Absorption Spectrum; TG/DTA Analysis; SEM Studies.

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Introduction

In the past few decades many researchers showing interest on nonlinear optical (NLO) materials because of their mesmerizing second and third-order non linear properties. Many investigations are being done to synthesize new semi organic materials with large second-order non linear optical property in order to satisfy day-to-day technological requirements. Nonlinear optical (NLO) frequency conversion materials have a major impact on laser technology, optical communication and optical storage technology [1]. Since NLO materials have very high potential applications in harmonic generation, amplitude and phase modulation, switching and other optical signal processing devices [2 - 4].

Currently semi organics are being explored which is having both the properties of organic and inorganic material like optical transparency, their large double refraction to use as frequency converters and second harmonic generation [5-6]. Further, since the organic ligand is ionically bonded with inorganic host, they have higher mechanical strength and improved chemical stability. Among the amino acids Tartaric acid is the simplest molecule having SHG efficiency, one-third of that of the well-known Potassium di hydrogen phosphate crystal [7-9]. In general, amino-acid single crystals have special features like wide transparency in UV as well as in visible region and have considerable second harmonic nonlinear optical (NLO) efficiency.

In this paper, we report the synthesis, growth and characterization of Urea doped Tartaric acid Potassium bromide single crystal. The grown crystals have been subjected to different instrumentation methods such as XRD, UV-Vis, TG/DTA, FTIR, SEM and Second harmonic generation SHG analysis. To the best of our knowledge there is no one report or literature survey for the grown semi organic single crystal Urea doped Tartaric acid Potassium bromide, and hence we report these for the first time.

Material and Methods

Crystal Growth

The synthesized single crystal Urea doped Tartaric acid Potassium bromide was obtained by mixing Tartaric Acid (amino acid) with inorganic salt potassium bromide in solution within the magnitude ratio of 1:1 stirred unendingly for 6 hours to get an identical solution [10]. Once the sample attained the saturated condition the solution was filtered and allowed to evaporate at normal temperature without any disturbance. Beautiful transparent crystals were obtained in a period of 15 days and the grown crystal is shown in Figure 1.

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Figure 1 Grown crystal of Urea Doped Tartaric Acid Potassium Bromide



Characterization Techniques

In order to calculate the lattice parameters of the grown single crystal, single crystal XRD studies were performed using ENRAF NONIUS CAD / MACH 3 single crystal diffractometer instrument. The UV-Vis-NIR analysis was performed between 400 and 4000 nm covering the whole ultraviolet, visible and near infrared regions by means of VARIAN CARY 5E spectrophotometer. The SHG efficiency was found by Kurtz and Perry powder technique through a Q switched Nd: YAG laser emitting 1064 nm. The functional group of the grown crystal was studied using BRUKER RFS 27: Stand alone FT-Raman Spectrometer. The thermal behavior of Tartaric acid Potassium bromide was studied using TG/DTA method between the temperature ranges of 20°C to 800°C using model Q600 SDT in nitrogen atmosphere.

Results and Discussion

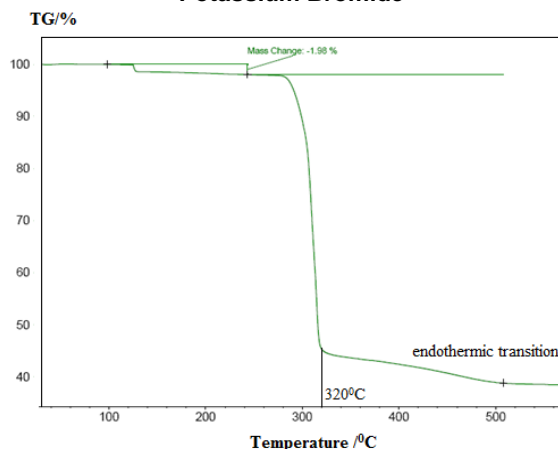
Single Crystal X-Ray Diffraction

The single crystal XRD analysis was implemented to know the cell parameters of the grown single crystal Tartaric acid potassium bromide using ENRAF NONIUS CAD / MACH 3 single crystal diffractometer instrument. The compound shows Orthorhombic structure, with cell constants: $a = 8.68 \text{ \AA}$, $b = 8.89 \text{ \AA}$, $c = 10.70 \text{ \AA}$, $\alpha = \gamma = \beta = 90^\circ$, $V = 643 \text{ \AA}^3$.

Thermo gravimetric Analysis

The thermal stability of the grown crystal was identified by the TGA (Thermo gravimetric analysis). To heat the sample a crucible was used and an analysis was done in nitrogen atmosphere at a heating rate of 20K/min between the temperature ranges of 20°C to 800°C. From the TGA graph shown in Figure 2, we can say that the material can withstand the temperature of up to 320°C after that the material begins to achieve an endothermic transition and begins to decompose [11].

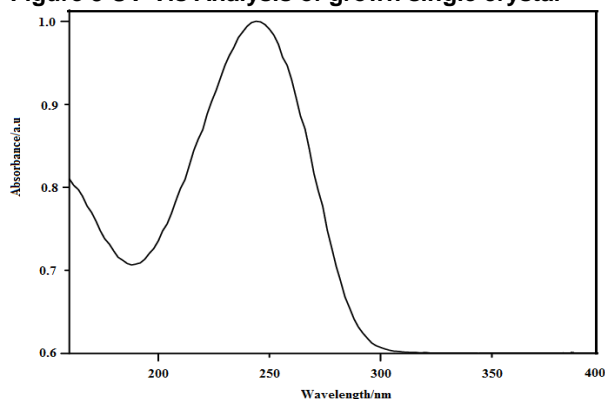
Figure 2 TG Analysis of Urea doped Tartaric acid Potassium Bromide



UV - Vis - NIR studies

The optical transmittance study for a material is very important to identify the optical transmission range and cut-off wavelength for the grown crystal. It is also very useful to know about its electronic transition states, when interaction of light with a molecule. The UV-Vis-NIR spectrum for the grown crystal was accomplished between 400 and 1100 nm using VARIAN CARY 5E Spectrophotometer and is shown in Figure 3. It is determined from the spectrum that there is terribly low absorbance within the entire visible region and shows most absorption at UV region. The UV cut-off wave length is found to be at 230 nm. This terribly low absorption property of the grown crystal within the entire visible region suggests its suitability for second harmonic generation [12].

Figure 3 UV-Vis Analysis of grown single crystal

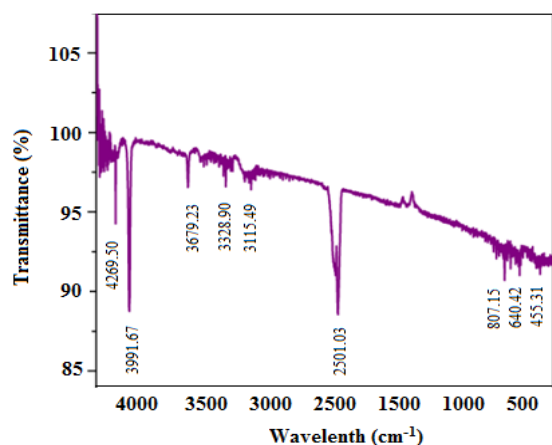


FTIR Analysis

The FTIR spectrum was recorded using BRUKER RFS 27: Stand alone FT-Raman Spectrometer by KBr pellet technique in the region 4000 – 400 cm^{-1} . From the figure 4, the FTIR spectrum of TAKBr shows absorbance peaks at wave number 3679.21 cm^{-1} (which is an O-H functional group), at wave numbers 3328.90–3115.49 cm^{-1} (which is a C-H functional group), FTIR spectra around 2501.03 cm^{-1} represents the (C-H functional group) and at wave number 807.15 cm^{-1} (which is a C-O functional group). The bands between 900 and 600 cm^{-1} correspond to (aromatic bending modes and) are mainly due to aromatic HCC (hydrogen-

carbon-carbon) rocking vibrations in aromatic and condensed aromatic ring systems. All these vibrations in figure 4 and the functional groups existing in the grown sample and are in good congruent with reported literature values.

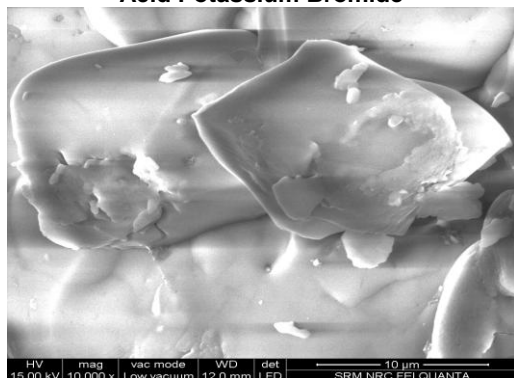
Figure 4 FTIR Analysis of Tartaric Acid Potassium Bromide



SEM Analysis

The Scanning electron microscopy (SEM) was performed using HITACHI S4800 at high vacuum with accelerated voltage of 30 KV. Fig.5. represents the layer formations over the surface of grown crystal at 10 μ m scale range. It is clear-cut that the grown crystals having stepped growth with constant evaporation of solutions at room temperature.

Figure 5 SEM Analysis of Urea doped Tartaric Acid Potassium Bromide



SHG studies

The second harmonic generation efficiency of grown crystal was confirmed by Kurtz-Perry powder method. The crystals were grounded in to a homogenous powder of particles in the range of 100-115 μ size and is tightly packed within two transparent glass slides illuminated by using Q-switched Nd-YAG laser emitting a fundamental wavelength of 1064nm with the pulse width of 8ns. The green light radiation (532nm) emitted as output beam which is found to be 0.7 times greater than that of KDP crystal.

Conclusion

Semi organic nonlinear optical crystal Tartaric acid Potassium Bromide was grown successfully by slow evaporation solution growth method. The lattice parameters were determined using single XRD method. The optical characters

were measured by UV-Vis spectral studies. Thermal stability of the crystal was found by TG/DTA analysis. FTIR Analysis confirm the formation of various functional group. Second harmonic generation efficiency of Tartaric Acid Potassium bromide single crystal was tested using Nd: YAG laser and it was found to be 0.7 times that of potassium di hydrogen phosphate (KDP crystal).

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