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Single Crystal XRD Studies and Physical Properties of Potassium Ammonium L-Tartrate Crystals

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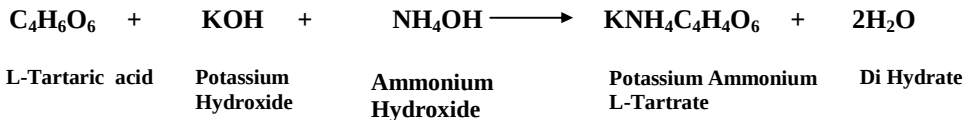
Abstract: A semi-organic nonlinear optical crystal of Potassium Ammonium L-Tartrate (PAT) has been grown by slow evaporation solution growth technique. The crystal system and lattice parameters are determined from the single crystal X-ray diffraction analysis. Fourier transform infrared (FTIR) studies confirm the various functional groups present in the grown crystal. The optical transmittance is studied through UV Visible spectroscopy. The mechanical property of grown crystals is analyzed by Vicker's Microhardness method. The thermal behavior of the grown crystals is investigated by DTA and TGA analysis. The second harmonic generation is comfired by using Nd: YAG Laser and compared with KDP.
Keywords: Semi-organic; nonlinear; slow evaporation; single crystal X-ray diffraction.

1. Introduction and Experimental

Recent research is focused on semi organic materials due to their large nonlinearity, high damage threshold, good mechanical and thermal stability [1-4]. The present investigation is focused on the growth and characterization of crystals of Potassium Ammonium L-Tartrate (PAT), a new semi organic material. The PAT crystals are grown by slow evaporation technique at room temperature. The PAT crystals obtained are subjected to various characterization studies such as single crystal XRD, FTIR, UV-Visible, SHG efficiency, Thermal studies and MicroHardness test.

1.1 Synthesis and Growth of Pat Raw Material

L-Tartaric acid, Potassium Hydroxide and Ammonium Hydroxide were taken in equimolar ratio (1:1:1).



The calculated amount of L-Tartaric acid was first dissolved in 100ml of deionized water. Potassium Hydroxide and Ammonium Hydroxide were added to the solution slowly by continuous stirring to get a homogeneous mixture. Then the solution was left undisturbed. After few days, Potassium Ammonium L-Tartrate (PAT) raw material was collected from the bottom of the beaker.

The saturated solution of PAT was prepared at room temperature and the solution was then filtered using Wattmann filter paper to remove the suspended impurities. The filtered solution was allowed to evaporate slowly at room temperature. Good, optically transparent crystals were harvested in a growth period of three weeks. PAT crystals are shown in Fig.1.

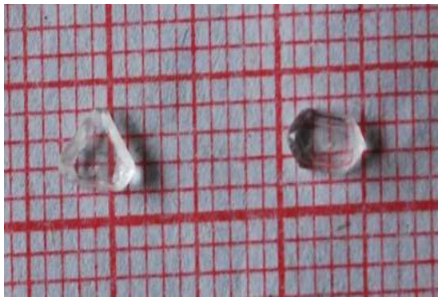


Fig 1. As grown PAT crystals

2. Results and Discussion

2.1 Single Crystal X-Ray Diffraction Analysis

The single crystal X-ray diffraction data were collected using BRUKER X8 Kappa APEXII diffraction meter. The analysis of the collected data reveals that the PAT crystal is monoclinic in structure with space group P1 and Lattice parameter are a=9.2909(Å), b=15.5424(Å), c= 6.2512(Å), α= γ= 90°and β = 101. 507°.The unit cell volume is 884.56(Å)^3.

2.2. FTIR and UV-Visible Spectral Analysis

The IR- spectrum (Fig.2) was recorded in the range 400 cm^{-1} to 4000 cm^{-1} with the help of PERKIN ELMER FTIR spectrometer using KBr pellet.The broad band at 3483 cm^{-1} is due to O-H stretching vibration. The bands at 2943 cm^{-1} and 2357cm^{-1} is due to C-H vibrations. C=O stretching vibrations are found at 1642cm^{-1}. The strong band at 1573 cm^{-1} is due to NH_2^+ symmetric bending. The peak at 1320 cm^{-1} is due to OH in plane blend [5-8]. The assignments confirm the presence of various functional groups in PAT.

UV-Vis transmission spectrum of PAT crystal was recorded using Perkin Elmer make Lambda 35 UV-Visible Spectrometer in the range 190nm to 1100nm. The UV-Vis spectrum of PAT crystal is shown in Fig.3. The PAT crystal is transparent in the entire UV-Visible region. It has a transparency of about 98% with a lower cut-off wavelength at 197 nm.

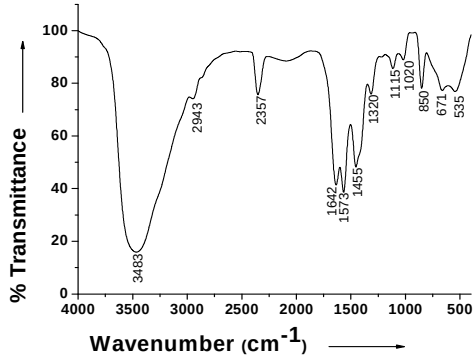


Fig. 2 FTIR spectrum of PAT

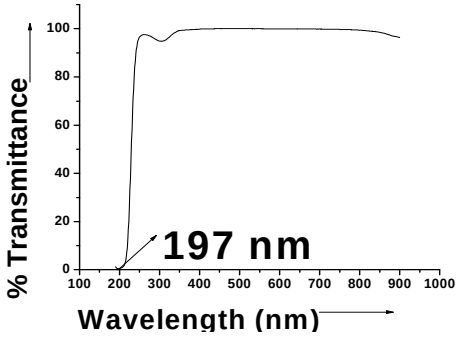


Fig 3 UV-Visible Spectrum of PAT

2.3. NLO Studies and Thermal Analysis

The SHG efficiency has been measured by Kurtz and Perry powder method using the 1064 nm fundamental beam of Q-switched Nd: YAG laser. The SHG efficiency of PAT is found to be nearly equal (0.5943 times) to that of KDP (8.8 mJ). But the SHG efficiency of PAT is very much less than that of other tartrate crystals reported earlier [9-11].

TGA and DTA for PAT have been carried out using a CNST thermal analyzer. The results are presented in Fig. 4. The results are presented in Fig. 4. In TGA, it is found that PAT crystal is stable upto 258°C. The sharp endothermic peak at 288°C indicates that the melting point of the substance is 288°C.

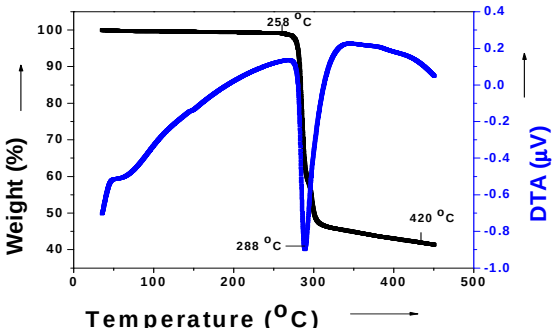


Fig. 4 TGA and DTA curves of PAT

2.4. Microhardness Study

Mechanical properties of the PAT crystal were studied by Vicker's Microhardness test using MMT-X MATSUZAWA hardness tester fitted with a diamond pyramidal indenter and the indentation time was fixed as 5 sec. Mechanical properties of the PAT crystals were examined by varying load. The microhardness of the crystal increasing with applied load[12]. Beyond the load of 100g, a significant crack developed around the indentation mark, which may be due to the release of internal stresses generated at the corners of the indentation.

2.5. Conclusions

Semi-Organic crystals of PAT were grown from slow evaporation technique. Single crystal X-ray diffraction study shows the crystal belongs to monoclinic crystal system. The FT-IR spectrum confirms the functional groups present in the grown crystals. The thermal studies show that PAT is thermally stable up to 258^{o}C and the melting point of the crystal is 288^{o}C. UV-Visible spectral analysis reveals that PAT crystals are optically transparent in the entire UV-Visible range and has a transparency of about 98% with a lower cut-off wavelength at 197 nm .The SHG efficiency of PAT is found to be nearly equal 0.5943 times of KDP. As PAT crystals have high thermal stability, good optical transparency, moderate mechanical strength and reasonably good NLO properties, these materials could be potential candidates for NLO applications.

3. References

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