ISSN: 0974-2115
www.jchps.com Journal of Chemical and Pharmaceutical Sciences

Growth of Sodium acid phthalate (NaAP) single crystals for harmonic generation applications

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ABSTRACT

Good quality optical single crystals of Sodium acid Phthalate (NaAP) single crystal were grown by different techniques such as solvent evaporation, slow cooling and seed rotation techniques. Transparent and optically good quality single crystals were obtained from, seed rotation technique. X-ray diffraction analysis confirms that the crystal belongs to orthorhombic system with a space group of B2ab. Spectroscopic analysis were carried out confirm the presence of functional groups present in the compound. Linear and non-linear optical properties of the grown crystals were confirmed through spectroscopic and through harmonic generation measurements. These preliminary investigations confirm that the present sodium acid phthalate crystals can be a potential candidate for nonlinear optical applications. The results are discussed in detail.

KEY WORDS: Crystals, growth, generation.

1. INTRODUCTION

The nonlinear optical (NLO) properties of large organic molecules and polymers have been the subject of extensive optical and experimental investigations during the past two decades. Alkali Acid phthalate (AAP) ($C_8H_5O_4$) [Li^+, Na^+, K^+ and Rb^+], crystals were then under use for second (SHG), third (THG) and higher order harmonic generators for Nd:YAG laser. These crystals are also widely used for the electro-optic Q-switches for Nd:YAG, Nd:YLF, Ti:Sapphire and Alexandrite lasers as well as for acoustio-optical applications. Currently, alkali Phthalic acid compounds have gained attention due to their excellent nonlinear and electro-optical properties. In this series some of the Alkali Phthalic acid complexes have been crystallized and reported by many researchers. Sodium acid phthalate (NaAP) has been chosen for the present study NaAP an excellent candidate for SHG applications in the phthalic acid family. It was already grown by slow evaporation solution growth technique and indeed the magnitude of laser-induced damage threshold and SHG efficiency were calculated to be higher than that of KAP crystal. In this paper, we describe the bulk growth of sodium acid phthalate single crystals for nonlinear optical applications.

2. MATERIALS AND METHODS

Sodium Acid Phthalate (NaAP) single crystal was synthesized by the reaction between Phthalic acid and sodium hydroxide in an equimolar ratio 1:1 dissolved in a de-ionized water. The solution was magnetically stirred for 24 h continuously and the homogeneous mixture was left standing for several days. Large platelet crystals on slow evaporation, elongated in (100) direction have been obtained within 4-5 days. The reaction mechanism is

$$C_8H_6O_4 + NaOH \rightarrow C_8H_5O_4Na\ 0.5\ H_2O + H_2O$$

Phthalic acid + Sodium hydroxide → Sodium acid Phthalate hemi hydrate + Water

Bulk crystal with good optical transparency was harvested by slow cooling technique and seed rotation technique was used in the present investigation. Large and transparent crystal of NaAP having dimension (37x37x10 mm^3) was obtained after a growth period of 45 days and the crystal grown are shown in Fig.1.

Figure.1.NaAP crystals grown from (a) slow evaporation (b) seed rotation and (c) platform rotation technique

3. RESULTS AND DISCUSSION

Three-dimensional intensity data of a transparent good quality crystal were collected on an Enraf-Nonius CAD-4 diffractometer equipped with MoKα radiation λ-0.71073Å.ω/2θ scan mode was employed for data collection. The dimension of the crystal used for the measurements was 0.1x0.2x0.1 mm^3. The crystal belongs to orthorhombic with a space group of B2ab having unit-cell dimensions a = 9.2597(7) Å; b = 26.261(2) Å; c = 6.7355(6) Å; α = 90°; β = 90°; and γ =90°; Z=8, at 298 (2) K.

Fourier transform infrared (FTIR) spectra of NaAP was recorded with a Bruker IFS66 FTIR spectrometer at room temperature in the frequency range of 400 -4000 cm^{-1} by KBr pellet method and is shown in Fig.2. The peak at 3502 cm^{-1} is due to OH stretching vibrations of the water molecule. A peat at 1599 and 1627 cm^{-1} are due to C=O stretching of the COO^- group. The aromatic ring structure of the compound is indicated by a peak at 1572 cm^{-1}.

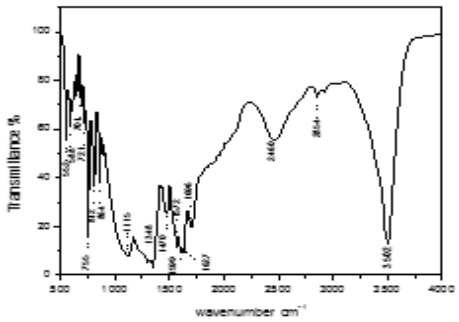

Figure.2. FTIR spectra of NaAP

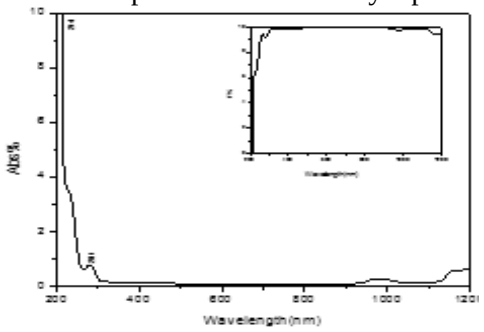

Figure.3. UV-Vis-NIR spectra of NaAP

The transmittance spectrum of the crystal was recorded using a Varian Cary 5E UV-Vis-NIR spectrophotometer in the wavelength range of 200 – 1200 nm and it is shown in the Fig.3. In order to determine the transmission range and the suitability of NaAP single crystals for optical applications, UV-Vis-NIR spectrum was recorded. The UV cut-off wavelength occurs between 210-220 nm. It is worth noting here that the transparency range for NaAP is much larger than other well characterized organic NLO crystals. The Second-harmonic generation (SHG) test on the Sodium Acid phthalate crystal was performed by Kurtz-Perry Powder SHG method. Samples were well packed in a compacted capillary quartz tube. The Crystalline powders were calibrated using powder X-ray diffractometer and the average grain sizes of the samples used for the study were in the range of micrometer. Then, the packed quartz tube is illuminated using prolab 170 Nd:YAG laser of fundamental output of 1064 nm with a gaussion pulse width 10ns and having a repetition rate of 10Hz. The input energy of the beam is 2.6 mj/pulse. The second harmonic efficiency of the polycrystalline sample was found to be equal to 0.45 d_{eff} (KDP), whereas the SHG efficiency of KAP crystals was about 0.15 d_{eff} (KDP).

4. CONCLUSION

The Nonlinear metallo-organic material sodium acid phthalate single crystals were grown by slow evaporation, slow cooling and seed rotation and platform rotation techniques. The unit cell parameters of the grown crystals were determined by single crystal X-ray diffraction. The vibrational structure of the synthesized compound has been elucidated by FTIR spectrum. The electron transition spectrum showed the good transparency window of the crystal in both the visible and IR region of electromagnetic spectrum. The SHG efficiency NaAP crystal was found to be higher than the of KAP crystal due to its higher dipole momentum of O^--Na^+ bonds than between potassium and oxygen.

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