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Influence of potassium chloride doping on the properties of urea succinic acid single crystals

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Abstract —*An organic single crystal of potassium chloride doped Urea Succinic Acid (USA) was successfully grown by slow evaporation technique. The grown crystal was characterized by X-ray diffraction (XRD) which confirms the lattice parameters. FTIR spectral studies were performed to identify he vibrations of functional groups. TGA/DTA studies revealed the thermal stability and the mechanical strength of grown crystal. The UV- vis spectral study reveals the enhancement optical property of grown crystal.*

Keywords-*Crystal Growth, XRD, FTIR, UV, Thermal studies.*

I. INTRODUCTION

Many organic crystals have been found potential applications in nonlinear optics and have more application in telecommunication, frequency mixing, optical parametric oscillation, optical bi stability, optical image processing and under water communication etc.[1-3]. The organic NLO materials have large dipole moment, spiral structure, high nonlinear figure of merit for frequency conversion, high laser damage threshold and fast optical response time as compared with inorganic materials [4]. Urea $[(NH_2)_2CO]$ is one such efficient organic non linear crystal. It is used for high power laser frequency shifting in a wide spectral range because of its large transparency window, large birefringence, relatively high damage threshold and low temperature dependence of the refractive indices. Succinic acid is used as a biodegradable polymer and biological production by fermentation has been focused as the alternative to petro chemical process [5-6]. The growth of succinic acid and urea succinic acid (USA) [7-9] crystals and their properties have been investigated earlier.

In the present study, potassium chloride doped with urea succinic acid crystal has been grown by the conventional slow evaporation technique. Attempts have been made to study the characterization of the grown crystal through powder XRD, FTIR, UV, and TGA.

II. CRYSTAL GROWTH

The title compound was synthesized by taking urea and succinic acid in a molar ratio of 1:1 and is dissolved in deionized water. Then 2 M percentage dopant potassium chloride solution is added slowly. The solution was agitated with a magnetic stirrer for 6 hours continuously and filtered. The solution was allowed to evaporate at room temperature. After three weeks of grown period, a colorless transparent crystal has been harvested. The photograph of the grown KCl doped USA crystal is shown in Fig. 1.

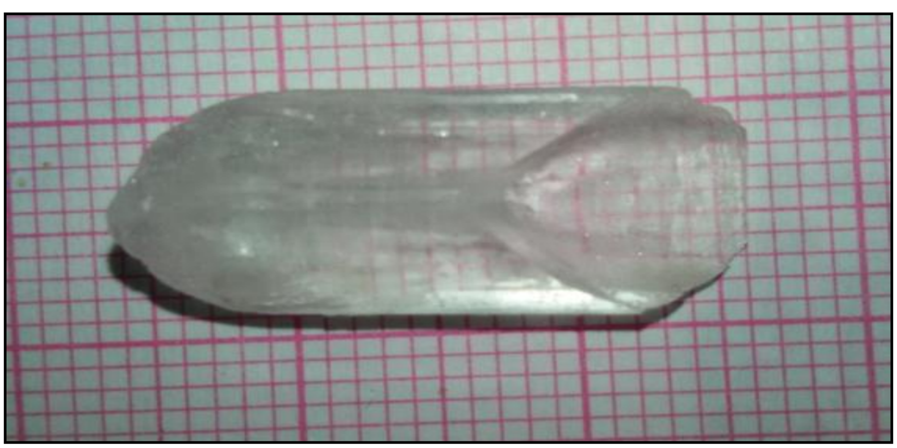

Fig.1: Photograph of KCl doped USA crystal

III. CHARACTERIZATION

The USA crystal doped with potassium chloride has been subjected to powder X-ray diffraction studies by using X'pert pro powder X-ray diffractometer with Cuk$_\alpha$ radiation (λ=1.5418Å). FTIR spectrum was rewarded in the range of 400-4000 cm^{-1} employing Brukel IFS-66V spectrometer using KBr Pellet technique. The optical absorption spectra of the grown crystals were recorded in the range of 100-1100nm using Varian Carey 5E Spectrophotometer. Thermal stability and decomposition stages were analyzed by recording TGA/DTA spectrum (TGA Q 500 V 20.10 Build 36 thermal analyzer) in the temperature range of 30-800°c in nitrogen atmosphere at a heating rate of 20°c/min.

3.1. XRD Analysis

The well defined Braggs peaks at specific 2θ angles have shown high crystalline nature of USA crystal doped with KCl. The intense peaks of the observed powder XRD pattern has been compared with the standard XRD pattern of the reported literature and indexed hence the dielectric constant decreases gradually. The observed powder XRD pattern of the grown crystal is shown in Fig. 2. It is noted that the doped USA crystals are nearly identical with pure USA with some slightly shifting of some intensities which proved that the dopant entered into the crystal matrix of USA [9].

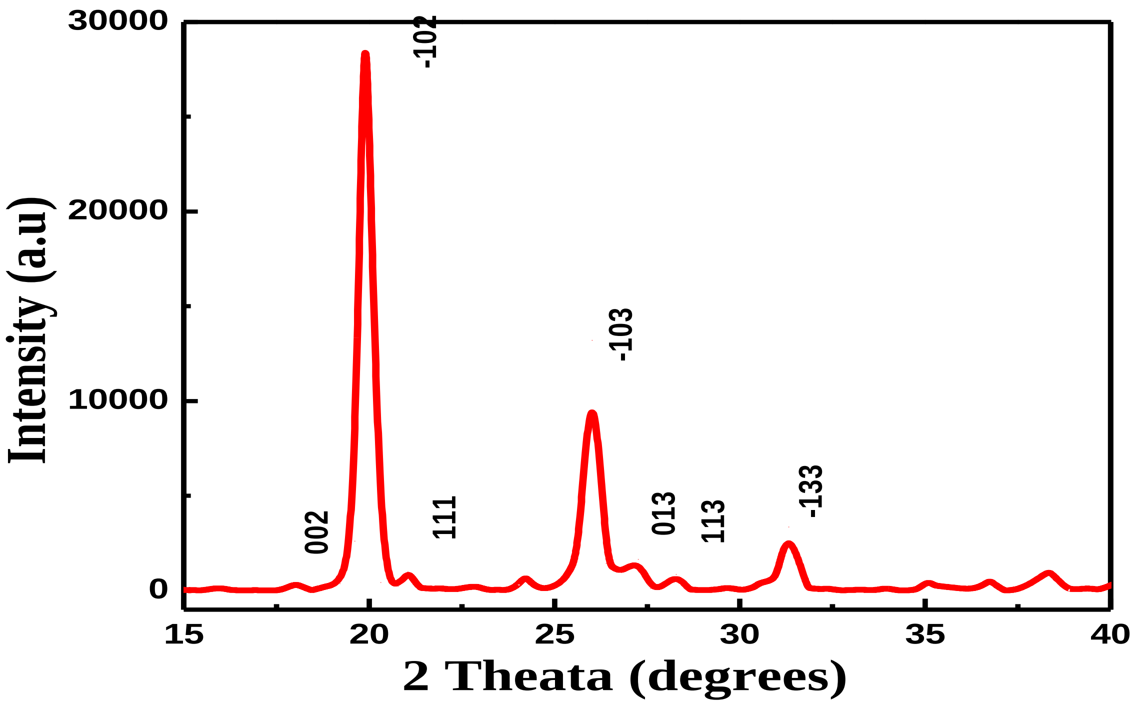

Fig. 2: PXRD pattern of KCl doped USA crystal

3.2. FTIR Analysis

FTIR spectrum is used to find out the functional groups present in the compound. The observed FTIR spectrum of the grown USA crystal doped with KCl is shown in Fig. 3. The O-H symmetric stretching bond of water occurs at 3480 cm^{-1} confirms the pressure of water in the crystal. The medium intensity peaks obtained at 3340 and 3230 cm^{-1} is due to asymmetric and symmetric stretching of N-H. The very strong bands at 2540 and 1700 cm^{-1} were associated with CH_2 and C=O stretching respectively. The appearance of medium intensity bands at 1310 and 1020 cm^{-1} have been assigned to symmetric stretching vibration of C-C and C-N. The presence of carbonyl group has been confirmed by the appearance of the band at 1420 cm^{-1} in USA crystal doped with KCl. In the present investigation, sharp peak at 901 cm^{-1} assigned most probably due to O-H out of plane vibration. The rocking mode of NH_2 and COO^- are wagging mode of COO^- are expected at 800, 442 cm^{-1} and 571 cm^{-1} respectively. The FTIR spectrum also shows the high transmission which is used in NLO application [10].

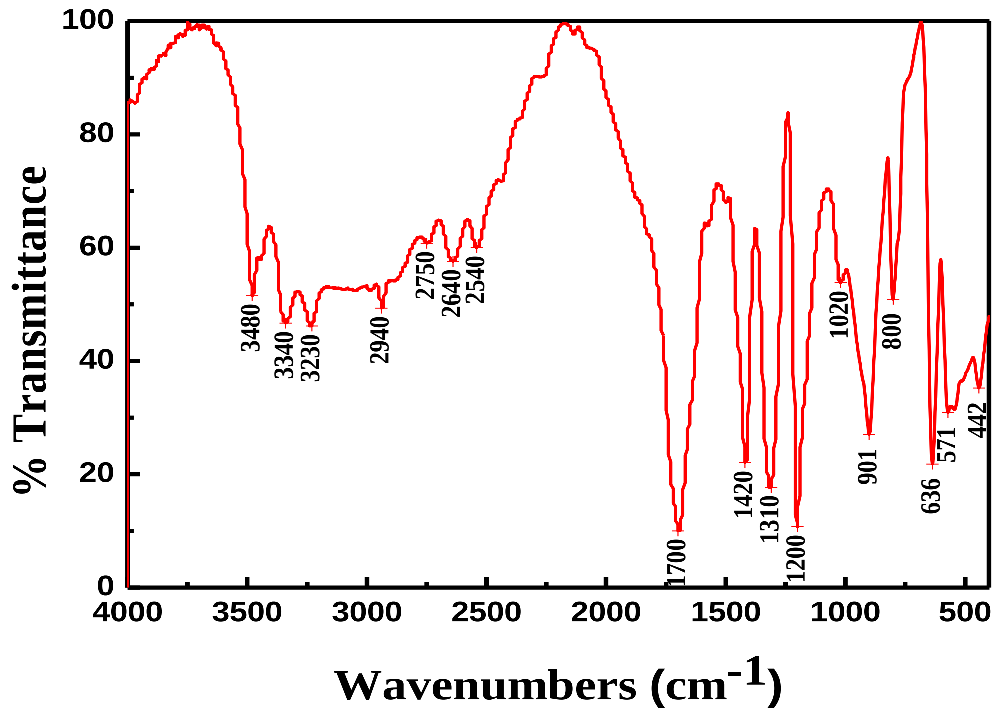

Fig. 3: FTIR spectrum of KCl doped USA crystal

3.3. Thermal analysis

The thermo gram and differential thermo gravimetric (TGA/DTA) traces of KCl doped USA crystal is shown in Fig. 4. In TGA, there is no weight loss up to 140°C. This indicates that there is no inclusion of water in the crystal lattice. It is found that the first stage of decomposition starts at 140°C and ends at 166°C with a weight of 32% due to the removal of succinic acid [11]. The second stage (168°C-217°C) is associated with a weight loss of 62% due to the release of gaseous product NH_2, CO etc. The strong endothermic peak at 217.29°C in DTA curve corresponds to the melting point of the grown crystal. The prolonged heating beyond 217°C up to 343°C with a minor weight loss of 3% is due to removal of residual substances. In the DTA curve at 166.08°C a phase change is occur, maybe due to the change from ferro electric phase to para electric phase which may be used for Electro optic devices [11].

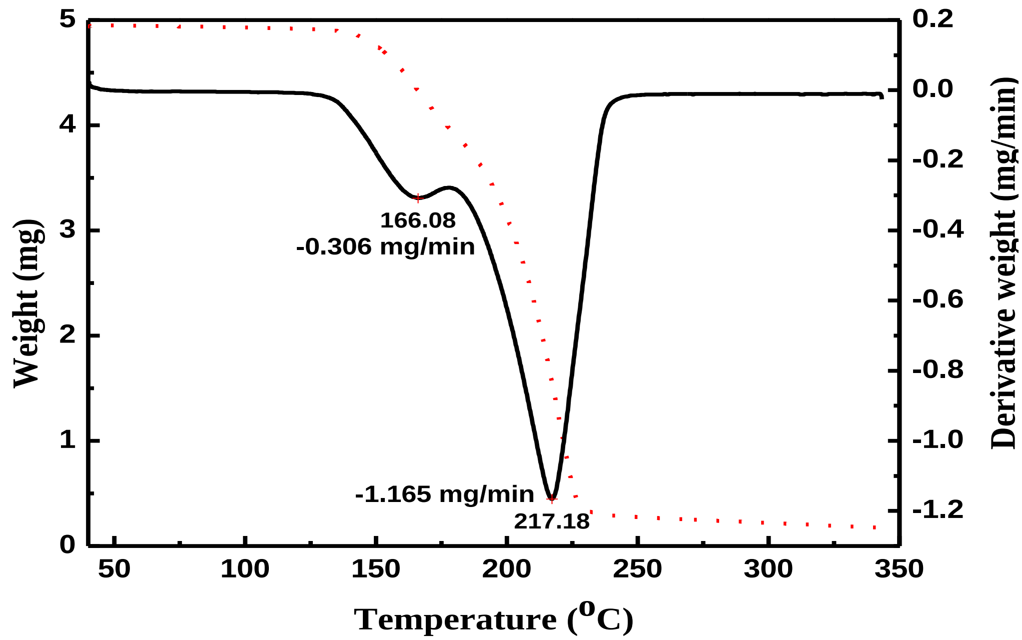

Fig. 4: TGA/DTA Thermograms of KCl doped USA crystal

3.5. Optical studies

The optical studies absorption spectrum nm of the USA crystal doped with KCl is shown in Fig.5. It is seen from the absorption spectrum that the crystal is transparent in the entire near UV and visible region (220 nm to 1100 nm). It has been found that the UV cut off wavelength is 219 nm. The low percentage of absorbance in the entire visible region is a desirable property for NLO application [12].

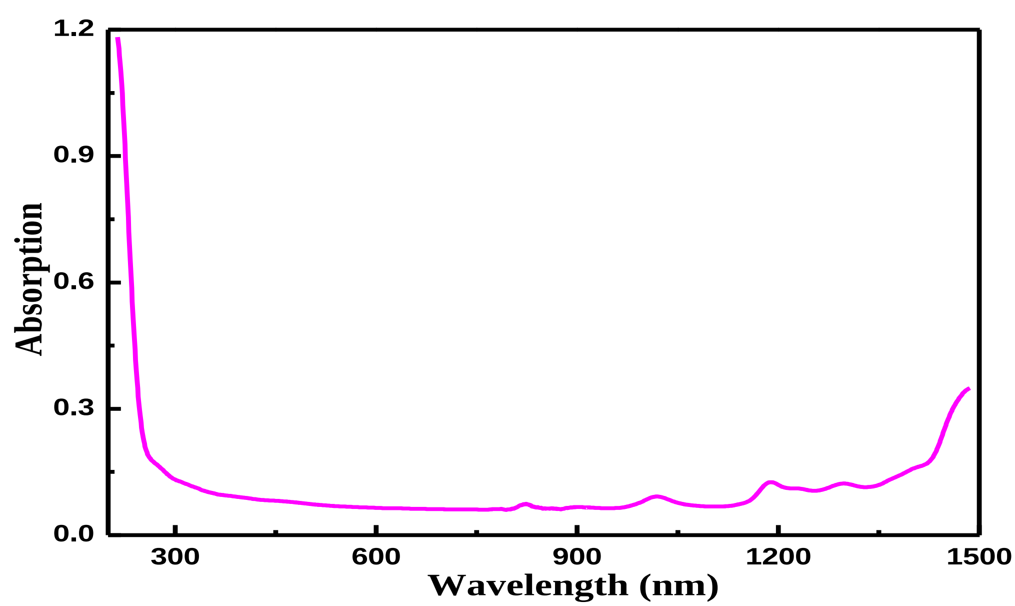

Fig. 5: Absorption spectra of USA crystal doped wih KCl

IV. CONCLUSION

In the present study the USA crystal, doped with potassium chloride has been grown by the conventional slow evaporation technique. The identity of the crystal has been confirmed by powder XRD techniques. The functional groups presented in the synthesized compound have been verified by FTIR studies. The UV cut off wavelength is found out as 219 nm. The melting point of the grown crystal is to be found out as 217.18°C. All characterization properties revealed the NLO application of grown crystal.

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