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SYNTHESIS, GROWTH AND CHARACTERIZATION ON TARTARIC ACID AND UREA (TAU) MIXED SINGLE CRYSTALS BY SLOW EVAPORATION METHOD

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ABSTRACT

A new semiorganic material of Tarataric acid and Urea (TAU) mixed crystals has been synthesized and successfully grown by the slow evaporation solution growth method. The grown crystals have been subjected to various characterization techniques such as single crystal XRD, SEM, FT-IR and UV-VISIBLE studies. XRD studies are used to carry out the crystallographic analysis. Fourier transform infrared spectum (FT-IR) is used to confirm the presence of various functional groups in the grown crystal. SEM studies are used to study the morphological identification. UV-VISIBLE studies are used to study the optical absorbance analysis.

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INTRODUCTION

Nonlinear Optical (NLO) materials can be classified into organic, inorganic and semiorganic NLO materials. Inorganic NLO materials possess high melting point, high mechanical strength, and high degree of chemical inertness but poor optical nonlinearity (Rajasekaran *et al*., 2001; Choudhury *et al*., 2003). Organic NLO materials have low melting point, low mechanical strength, high degree of delocalization due to their weak Van der Waal's and hydrogen bondings and also they have the flexibility in the methods of synthesis, scope for altering the properties by functional substitution, inherently high nonlinearity, high laser damage threshold values (Arun *et al*., 2009; madurambal *et al*., 2010). Semi Organic non linear optical materials wide range of application in the field of telecommunication, optical information storage device. These materials have nonlinearity, high resistance, too large induced damage, low angular sensitivity, good mechanical hardness.

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In the present investigation Tartaric Acid and Urea (TAU) mixed single crystals were grown by slow evaporation technique. The grown crystals have been subjected to various characterization techniques such as single crystal XRD, SEM, FT-IR and UV-VISIBLE studies. The results obtained from various studies of Tartaric Acid and Urea (TAU) mixed single crystals are reported and dicussed.

Growth of Crystals

Synthesis of tartaric acid and urea (tau) mixed single crystals

The synthesis of mixed crystals are done under low temperature slow evaporation method. 5g of each Tartaric acid and Urea is weighed accurately and dissolved in 50ml of double distilled water and stirred it vigorously at 28^0c for 15minutes. Then, the saturated solution was decanted into the 100ml beaker. The remaining solute was dissolved in 5ml of distilled water; the solution is stirred continuously for 15 minutes. The above process is continued until the solute is fully saturated. After the saturation, the solution was taken in a

100ml beaker which was tightly closed with the filter paper, so that the rate of evaporation could be minimized. Good transparent mixed crystals are obtained after 4 weeks. The grown crystals are shown in Fig.1. Then, the synthesized crystals are characterized using XRD, SEM, FT-IR, UV-VISIBLE studies.

Fig. 1. Tartaric acid and Urea (TAU) mixed single Crystals

Characterization Methods

XRD Analysis: X-ray powder diffractograms were recorded for Butylamine, Ascarpic acid and Leucine mixed crystals at room temperature. The sample for the powder X-ray diffraction was prepared by crushing a small piece of a crystal into fine powder. The fine calcined particles were characterized for crystal phase identification by powder x-ray diffraction (XRD) using a XRD-6000 (Shimadzu, Japan). The powder was then spread over a glass plate with uniform thickness. Methanol was used as a binder to spread the powder on the glass plate with uniform thickness. The radiation used was Ni filtered Cu Kα (λ=1.5418Å). The scan speed was 10°/minute and the scan range was 4.0000°-90.0000°.

FT-IR Spectral Analysis: Fourier Transmission Infrared (FT-IR) spectra of the Butylamine, Ascarpic acid and Leucine mixed crystals (as pellets in K_{Br}) were recorded using IR Affinity-1 Fourier Transmission Infrared spectrometer (Shimadzu, Japan) in the range of 4000-400 cm^{-1} with a resolution of 1cm^{-1}.

Sem Analysis: The particle size and external morphology of the fine calcined powders were characterized by scanning electron microscopy Jsm-6390 (Shimadzu, Japan).

UV-Visible Spectrum: The optical Absorption spectra was studied using UV–1700 Pharma spectrophotometer (Shimadzu, Japan) in the range of 200–800 nm.

RESULTS AND DISCUSSION

XRD Spectrum: The powder XRD patterns of Tartaric acid ($C_4H_6O_6$) and Urea (CH_4N_2O) mixed crystals are shown in figures a=7.69,b=2.33, c=1.24 and a=b=5.5890, c=4.6947 and the data's are the lattice parameters and the crystal system are presented. The data indicates that TAL crystal crystallizes in orthorhombic structure (Madhavan *et al*., 2007). To confirm the XRD data, powder XRD studies were also carried out for the sample. The grown crystals of TAU were crushed into fine powder and powder X-ray diffraction analysis has been carried out using a powder X-ray diffractometer. The recorded pattern is shown in Figure.2. The sharp peaks of XRD pattern indicate high degree of crystalline structure of grown crystal. The observed diffraction pattern has been indexed and Miller indices were estimated by Indexing software package.

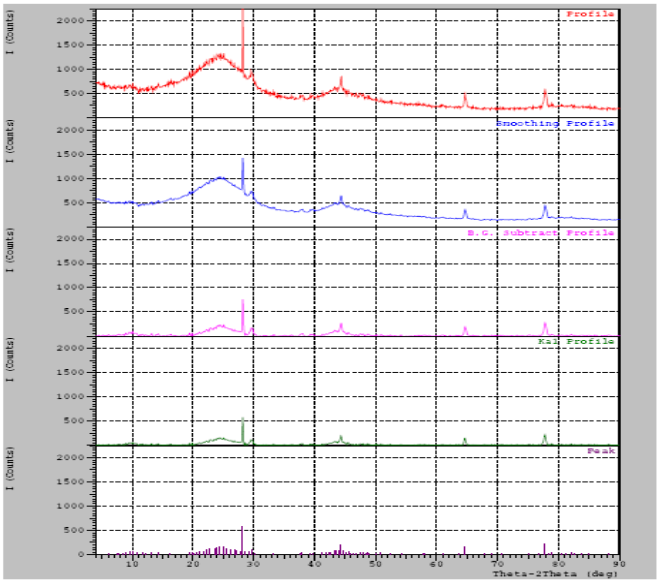

Fig. 2. XRD diffraction Tartaric acid and Urea (TAU) mixed single crystals

FT-IR Spectral Analysis: The presence of N-O stretching asymmetric is indicated by a sharp absorption peak at 2700.11cm^{-1}. The sharp absorption peak's observed at 1708.04cm^{-1} respond to CO_2 symmetric stretching. The sharp absorption peak observed at 1550.01 cm^{-1}. The absorption peak observed at 1150.07 cm^{-1} respond to C=O asymmetric stretching of Acid group. Stretching of N-O linkage produces very strong & broad peak at 700.09 cm^{-1} is attributed to Nitrate group. The sharp absorption observed at longer wavelength near 680.03 cm^{-1} likely result from NO_2 bending vibration.

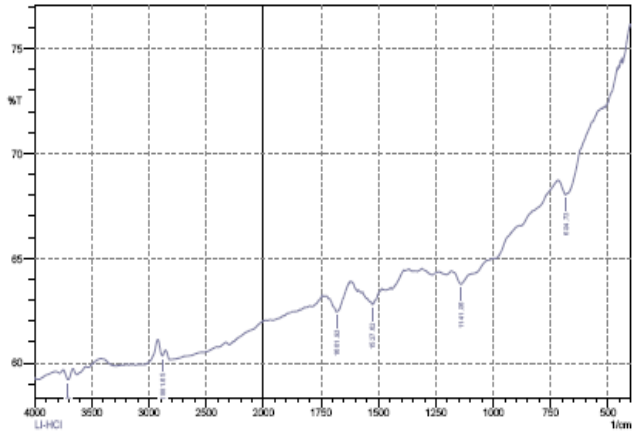

Fig. 3. FT-IR spectrum of Tartaric acid and Urea (TAU) mixed single crystals

Table 1. Vibrational Assignments for TAU Crystal

Frequency cm^{-1}	Assignment
3600.01	OH -streching
2700.11	N-O stretching asymmetric
1708.04	CO_2 symmetric stretching
1550.12	sharp absorption
1150.07	C=O asymmetric stretching of Sulphate group
700.06	N-O linkage of Nitrate group
680.03	NO_2 bending vibration

SEM Analysis: Typical SEM micrographs for powder synthesised at different magnifications are shown in fig.4. it is evident from the photograph that the background of the surface was crystalline and morphology crystalline Tartaric mixed crystals the size of the particle determined from the SEM image are 5-100 µm.

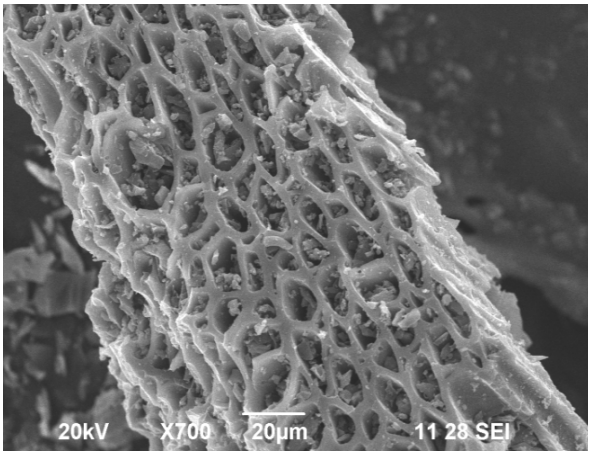

Fig. 4. Scanning electron micro graph of Tartaric acid and Urea (TAU) mixed single crystals

UV-Visible Absorbance: The lower cut-off the weak concentration of Tartaric acid and Urea mixed crystals is around strong absorbance 1.253 in the mid range uv-302 nm and high concentration of Tartaric acid and Urea mixed crystals is around strong absorbance 3.010 in the mid range uv-301 nm.

A) Weak Concentration

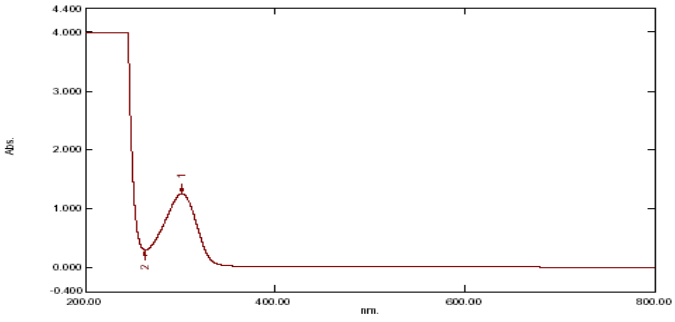

Fig. 5a. UV-visible absorption spectrum of Tartaric acid and Urea (TAU) mixed single crystals

No.	P/V	Wavelength	Abs.
1		302.00	1.253
2		263.00	0.297

B) High Concentration

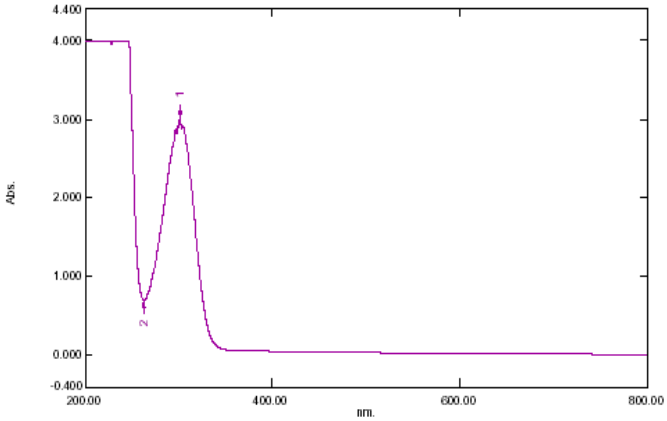

Fig. 5b. UV-visible absorption spectrum of Tartaric acid and Urea (TAU) mixed single crystals

No.	P/V	Wavelength	Abs.
1		301.00	3.010
2		262.50	0.696

Conclusion

A Tartaric acid is don't have NLO application because that compound having centro symmetry nature and NLO application of Tartaric acid and Urea mixed crystals having non centro symmetry nature crystals were successfully grown by low temperature slow evaporation solution growth method.The grown crystals have been subjected to various characterization studies such as XRD, FT-IR, SEM, analysis, UV-VIS-Spectrum. The mixed crystals XRD analysis confirms that the Tartaric acid and Urea mixed single crystals is Orthorhombic in structure with space group Cmc_{1}. The molecular structure of the synthesis compound was confirmed by FT-IR analysis. The background of scanning electron microscope shows the formation of microparticles. The weak concentration of Tartaric acid and Urea (TAU) mixed crystals is around strong absorbance 1.253 in the mid range uv-302 nm and high concentration of Tartaric acid and Urea mixed crystals is around strong absorbance 3.010 in the mid range uv-301 nm. The Tartaric acid and Urea mixed (TAU) single crystals are used in the field of medical phototherapy and dermatological, solar experimentation.

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