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Research Article

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Effect of EDTA Doping on Uracil Crystals

V Beaula Premavathi[1*], G Madhurambal[2] and B Kavitha[1]

[1]*TBML College, Porayar, Tamil Nadu, India*
[2]*ADM College for Women, Nagapattinam, Tamil Nadu, India*

ABSTRACT

Ethylenediamine teraacetic acid (EDTA) doped Uracil were grown by slow evaporation method at room temperature with double distilled water as solvent. The grown crystals have been subjected to spectroscopic and structural properties. FTIR analysis has been carried out to characterize the grown crystals. The range and percentage of optical transmission as well as absorption are determined by recording UV-Vis spectrum. From the UV data, Band gap determination is also carried out. Fluorescence spectra has also been carried out to characterize the grown crystals to study the absorption and emission spectra. The powder X-ray diffraction methods are used for identifying the structural parameters of grown crystals. The effect of the influence of dopant on the surface morphology of Uracil-EDTA crystal faces are analyzed by SEM Elemental composition were also confirmed by EDAX method.

Keywords: Band gap; EDAX; FTIR; SEM

INTRODUCTION

Crystal growth is a major stage of crystallization process, and consists in the addition of new atoms, ions (or) polymer strings into the characteristic arrangement of a crystalline Bravais lattice. Uracil is a common and naturally occurring pyrimidine derivative compounds, which are worth to study for many reasons, chief among them being their prevalence among biologically active molecules [1-4]. It is a planar, unsaturated compound that has the ability to absorb light. Uracil's use in the body is to help carry out the synthesis of many enzymes necessary for cell function through bonding with riboses and phosphates [5]. Uracil serves as allosteric regulator and coenzymes for reactions in the human body and in plants [6]. Uracil also has the capability to react with elemental halogens because of the presence of more than one strongly electron-donating group [7]. The dopant Ethylenediamine tetraacetic acid is a typical chelate ligand, which has attracted much attention over the past five decades because of its usefulness for industrial applications as well as basic study applications. Nowadays, organic crystals are highly recognized as the materials of the future because their molecular nature can be used to maximize the non-linear properties. In this paper, we report the results of our work on the growth of new Uracil-EDTA crystals for the first time along with the characterization of powder XRD, Fourier transform infrared transmission (FITR) spectrum, UV-Vis analysis, Band Gap determination, Fluorescence spectra, SEM and EDAX analysis studies.

EXPERIMENTAL SECTION

Crystal Growth

The Selection of the solvent is an important step in the growth of crystals from solution by slow evaporation method. In this method, water is used as a solvent. Commercially available Uracil and EDTA were used for the Crystal growth. Initially, the material was purified by repeated recrystallization then, the saturated solutions of Uracil and EDTA at room temperature were prepared. Then the solution is filtered. The filtered solution was taken in a beaker which was sealed with small size hole to control the evaporation of the solvent. The well-defined crystals are obtained within a period of 15 days.

Crystal Characterization

FTIR spectral analysis:

The fourier transform infrared spectral analysis is a technique in which almost all functional groups in a molecule absorb characteristic frequencies. The FTIR spectrum was recorded in the range 400-4000 cm^{-1} by perkin-Elmer Spectrometer (KBR Pellect Technique). The FTIR spectrum of Uracil and doped Uracil are shown in Figures 1a and 1b and the fundamental vibrations and the assigned frequencies are shown in the Table 1. It is seen from the spectrum that the Uracil and doped Uracil is having different peaks with different frequencies. In all the heterocyclic compounds, the N-H stretching vibration occur in the region 3500-3000 cm^{-1}. Hence the FT-IR band observed at 3099 cm^{-1}, 3059.60 cm^{-1}, 3022.81 cm^{-1} in pure Uracil correspond to N-H stretching vibrations which is completely absent in doped Uracil. This shows that EDTA may form vanderwaal’s bond with Uracil in this area. The FT-IR bands observed at 1234.87 cm^{-1} in pure Uracil corresponds to C-N vibrations is absent in doped Uracil. Similarly, the band occurred at 1615.67 cm^{-1} assigned to C-C stretching vibrations modes in pure Uracil is absent in doped Uracil. But the stretching assignments proposed in this study, for OH out-of-plane bending group are identified at 767.29 cm^{-1} in the doped Uracil which is completely absent in pure Uracil. The two C=O groups of Uracil are chemically in different positions; they seem to be interacting with each other within the Uracil molecule. The interaction of the carbonyl group with a hydrogen-donor group does not produce drastic and characteristic changes in the frequency of the C=O stretch as does by interaction of N-H stretch. It great deal of structural information can be derived from the exact position of the carbonyl stretching absorption peak. The carbonyl frequency obtained at 1678.28 cm^{-1} in pure Uracil may be shifted to 1684.34 cm^{-1} in doped Uracil which confirms the presence of C=O bond with adjacent C-C bond increases the energy required for C=O stretching and thus increases the carbonyl stretching frequency in doped compounds. The C=C stretching vibrations of 1648.69 cm^{-1} and C-C stretching vibrations of 1615.67 cm^{-1} are completely absent in doped Uracil which confirms the EDTA may be entered into the crystal lattice of Uracil. Further the O-H in plane bending of 1391.48 cm^{-1} are found in doped crystal which are completely absent in pure Uracil. The N-H bending vibrations of 858.59 cm^{-1}, 818.95 cm^{-1} in pre Uracil are completely absent in doped crystal. This vibrations may be the further evidence for incorporation of EDTA into the host molecule [8,9].

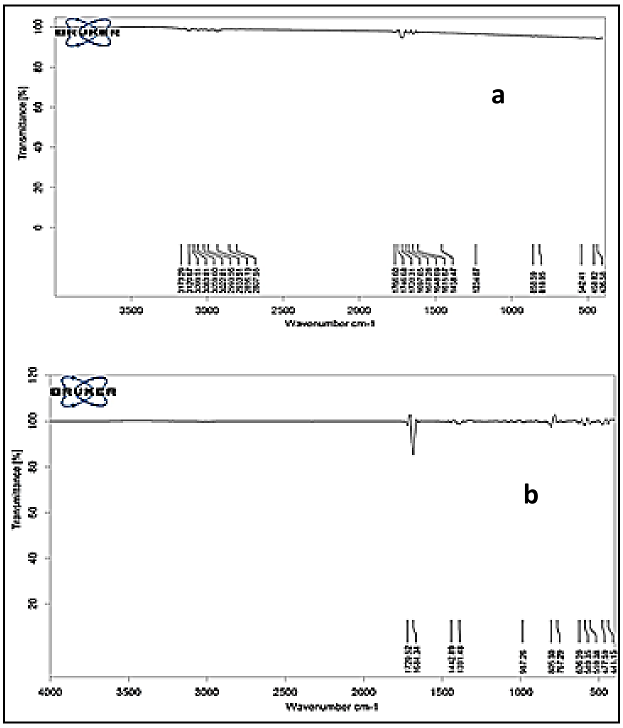

Figure 1: a) FTIR spectrum of pure uracil; b) FTIR spectrum of doped uracil

UV spectral analysis:

The UV-Vis absorption spectrum of the grown crystal were recorded in the wavelength range of 200 nm and 1100 nm using lambda 35 UV-Vis spectrophotometer in order to determine the transmission range and hence the suitability of the crystals for optical applications. The UV-Vis absorption spectrum of pure Uracil and doped Uracil are shown in Figures 2a and 2b.

Table 1: Vibrational frequencies of pure and doped uracil

Wave Number(cm^{-1})		Assignment
Uracil	**Doped EDTA**	
30993.51	-	O-H Stretching
3120.67		N-H Stretching
3083.81		
3059.6		
3022.81		
2993.56		C-H Stretching
2933.51		
1678.28	1684.34	C=O Stretching
1648.69		C=C Stretching
1615.67		C-C Stretching
-	1391.48	In plane O-H bending
1234.87		C-N
858.59		N-H bending
818.95		
-	767.29	OH-out-of-plane bending

It is seen that there is a slight change in the UV transparency cut off wavelength in pure Uracil and also in doped Uracil and there is a residual absorption in the entire UV-Visible region in both Uracil and doped Uracil. Regarding the electronic absorption, the peak in the range is 258.8 nm is attributed to n-π^* transition of the carbonyl group. Usually, the C=O group show a band in the higher region, but when the carbonyl group is substituted by an aurochrome, the n-π^* transition in this compound is shifted to lower wavelength region. When the EDTA entered into the Uracil crystal, the n-π^* transition of imide group, made a bathochromic shift from 258.8 nm to 260.1 nm considering the transition of imide. Bathochromic shift is also observed in the range of 200.8 nm to 203.3 nm. All these facts suggested that the doped crystals has a wide range of application in opto-electronic files [8,9].

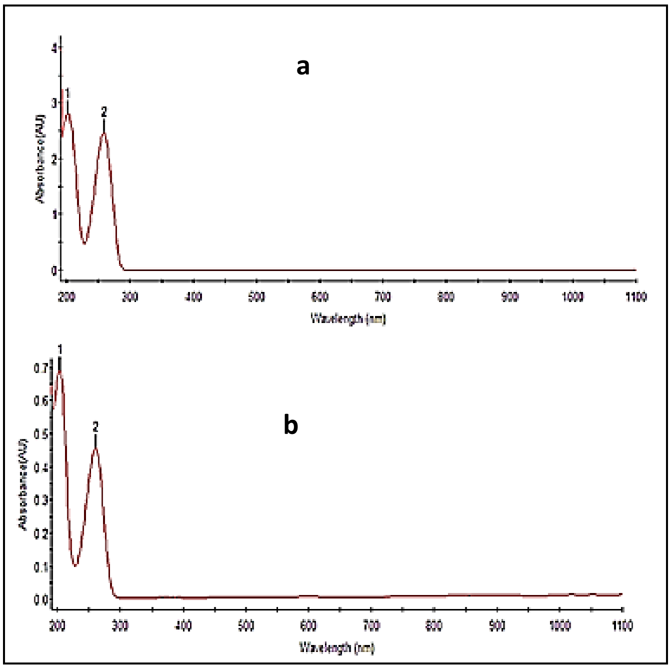

Figure 2: a) UV-Vis spectrum of pure uracil; b) UV-Vis spectrum of doped uracil

Band gap energy determination:

The band gap measurement was also carried out for Uracil and doped Uracil crystals. The plot of $(\alpha h\gamma)^2$ against $h\gamma$is shown in Figures 3a and 3b, a trend line was added to extrapolate and it cut the X-axis of 5.64 eV in Uracil and 5.73 eV in doped crystal which is taken as the direct band gap of the crystal. Due to the addition of EDTA with Uracil, change in band gap is observed. As a result of wide band gap the doped crystals possess wide transmittance window in the UV-visible region.

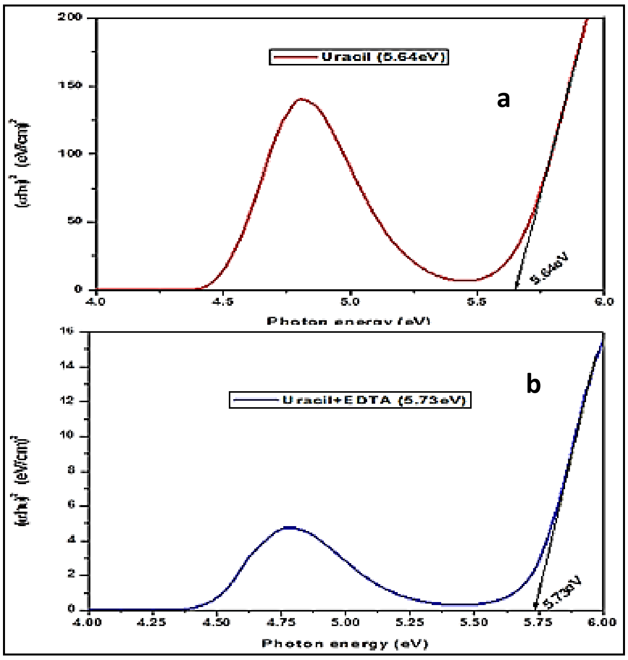

Figure 3: a) Band gap energy of pure uracil; b) band gap energy of doped uracil

Fluorescence spectra:

The Fluorescence spectrum of Uracil and doped crystal are measured in the range of 320.00 nm to 750 nm are shown in Figures 4a and 4b. It has been observed that fluorescence spectra observed at excitation absorption of slight broad band at 335.49 cm^{-1} and emission of sharp band at 606.68 cm^{-1} in pure Uracil. When the EDTA entered into the Uracil Crystal, the fluorescence slightly changes in the absorption of broad band at 337.49 cm^{-1} and the excitation process confirms the presence of broad band at 611.75 cm^{-1}. All these facts suggested that both Uracil and doped Uracil has fluorescence activity.

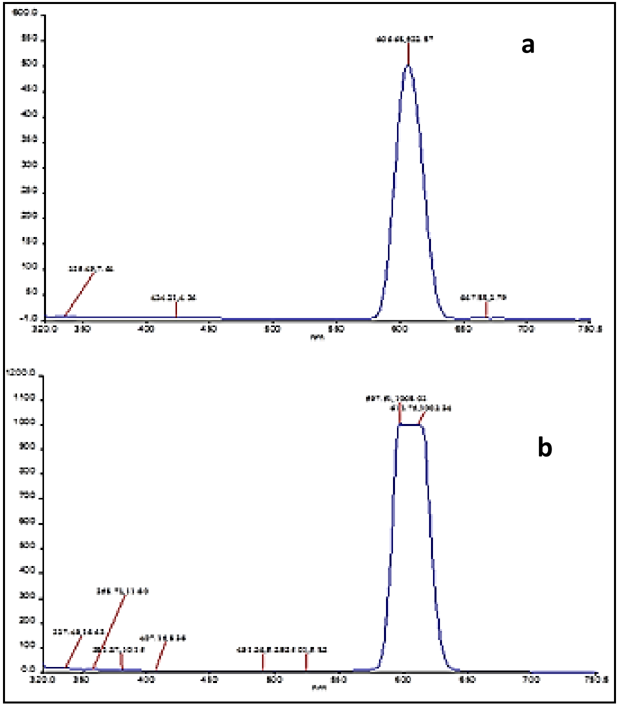

Figure 4: a) Fluorescence spectra of pure uracil; b) fluorescence spectra of doped uracil

Powder X-ray diffraction analysis:

X-ray diffraction technique is used to investigate the inner arrangement of atoms (or) molecules in a crystalline material. The grown Uracil and EDTA doped Uracil crystal were subjected to powder X-ray diffraction studies and the observed patterns are shown in Figures 5a and 5b respectively. Well defined peaks at specific 2θ values shown high crystalline nature of the grown crystals. The powder XRD patterns of doped Uracil are compared with that of pure materials. The XRD profiles show that all the samples were of a single phase without detectable impurities. The slight shift of the reflection peaks in the powder XRD patterns of the doped samples indicates that the dopant has entered into the lattice of the host crystal XRD patterns of doped crystals are not similar to that of pure Uracil. Additional peaks are present in the XRD patterns of doped crystals. This shows that doped crystals have different crystalline structure as that of pure Uracil [10-12].

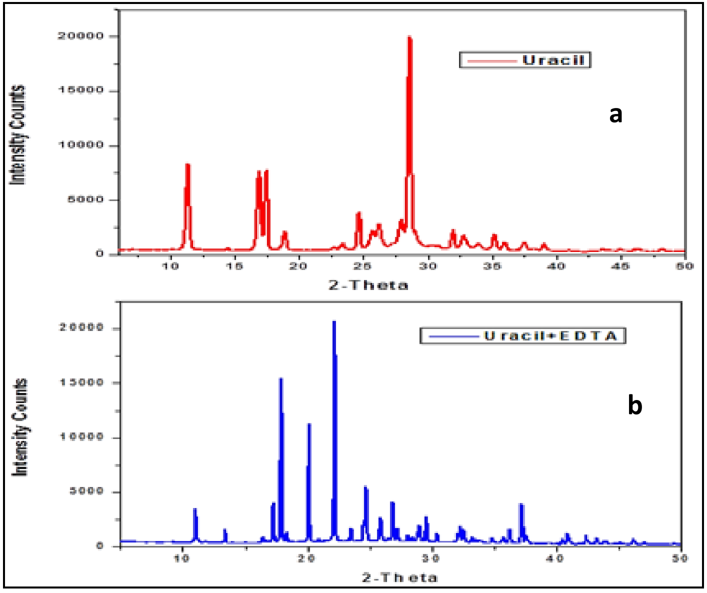

Figure 5: a) Powder XRD spectra of pure uracil; b) powder XRD spectra of doped uracil

Scanning electron microscopic analysis:

SEM analysis provided information about the nature and also it is used to check the presence of imperfections. The SEM image of pure Uracil and doped crystal are shown in the Figures 6a and 6b. It is found that Uracil crystal has well developed morphology exhibiting several faces and then all the units are arranged in different layers. But the doped crystal has entirely different morphology. Due to this fact, it may be revealed that the EDTA acts as a growth modifier [13].

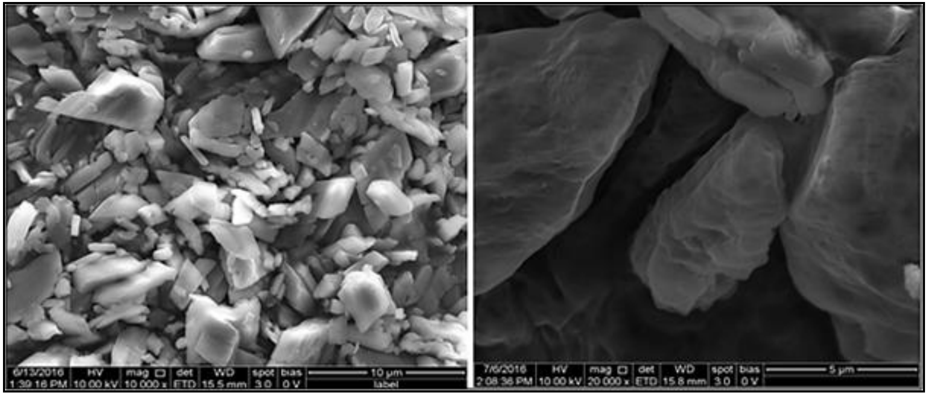

Figure 6: a) SEM image of pure uracil; b) SEM image of doped uracil

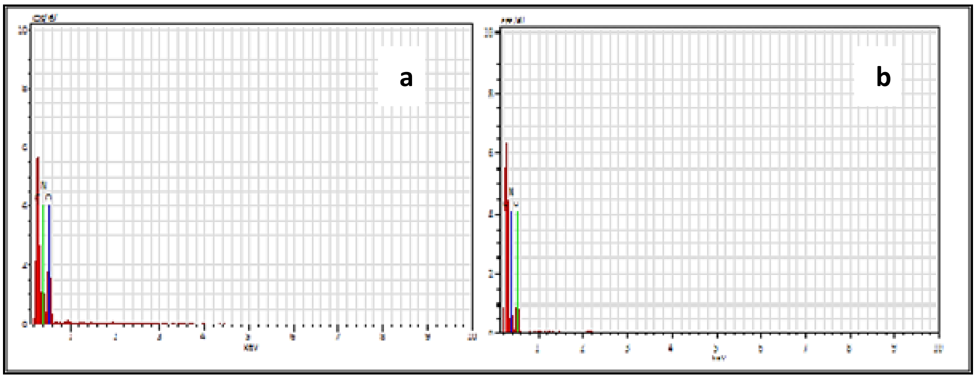

Figure 7: a) EDAX Spectrum of pure uracil; b) EDAX spectrum of doped uracil

Energy dispersive X-ray analysis:

In order to confirm the presence of the elements in the grown Uracil and doped crystal, the sample of grown crystals were subjected to Energy dispersive X-ray analysis and shown in Figures 7a and 7b. The obtained spectrum confirms the presence of C,O and N and the corresponding atomic percentages are 44.32, 26.84, 28.85 respectively. When the EDTA entered into the host crystal, the atomic percentage changed into 57.37, 21-44 and 21.18 respectively. These facts revealed the absence of imperfections in the crystallisation.

CONCLUSION

EDTA doped Uracil were synthesized at room temperature by slow evaporation method. Good optical quality single crystals were grown. The functional groups present in the grown crystal have been confirmed by FTIR spectral analysis. UV-Vis spectra show that the crystal has a wide range of application in opto-electronic field. Band gap measurement indicated that the doped crystals possess wide transmittance window in the UV-Visible region. Fluorescence spectra confirm the fluorescence activity of grown crystals. Powder X-ray diffraction confirms the crystallinity nature of the sample. SEM analysis confirms the morphology of the grown crystals and EDAX analysis shows the elemental analysis of the crystal and confirms the absence of any impurities and imperfection in the crystal.

REFERENCES

[1] J Cahn; Hillig; WG Sears. *Acta Metall Mater*. **1964**, 12(12), 1421.
[2] WK Burton; N Cabrera; FC Frank. *Philos T Roy Soc A*. **1951**, 243, 299.
[3] KA Jackson. Growth and perfection of crystals, RH Doremus; BW Roberts; D Turnbull (eds), Wiley, New York, USA, **1958**.
[4] J Chan. *Acta Metall Mater*. **1960**, 8(8), 554.
[5] RH Garrett, CM Grisham. Principles of biochemistry: with a human focus. Thomson Brooks/Cole, **2001**.
[6] EG Brown. Ring nitrogen and key biomolecules: The biochemistry of N-heterocycles. Springer Science & Business Media, **2012**.
[7] DJ Brown. Heterocyclic compounds, The pyrimidines. Inter Science, New York. **1994**, 52.
[8] RM Silverstein; GC Bassler; TC Morrill. Spectrometric identification of organic compounds. John wiley & sons, **2014.**
[9] YR Sharma. Elementary organic spectroscopy. S Chand Publishing, India, **2000**.
[10] H Senhil Pandian; P Ramasamy. *J Cryst Growth.* **2008,** 310, 2563-2568.
[11] T Balakrishnan; K Ramamurthi, *Cryst Res Technol.* **2006**, 41, 1184.
[12] R Muralidharan; R Mohankumar; PM Ushasree; R Jayavel; P Ramasamy. *J Cryst Growth.* **2002**, 234, 545.
[13] N Balamurugan; M Lenin; G Bhagavan Narayana; P Ramasamy. *Cryst Res Technol.* **2007,** 42, 151.