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Growth and characterization of some chlorides doped n-benzoyl glycine crystals

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ABSTRACT

Pure and Chlorides doped n-benzoyl glycine (Hippuric acid) single crystals have been grown by slow evaporation technique at room temperature. The crystal system has been identified and lattice dimensions have been measured using X-ray diffraction analysis. The functional groups of the grown crystals were qualitatively examined using FTIR analysis. Its optical character has been assessed by UV- spectral transmission analysis. The thermal behavior of the crystals has been investigated by TGA/DTA analysis.

Keywords: Crystal growth, Organic compounds, n-benzoyl glycine.

INTRODUCTION

It is thought that development of a new optical device is needed to satisfy the demand in the society. The large non-linear optical properties of large organic molecules and polymers have been the subject of extensive theoretical and experimental investigations during past few decades. Considerable efforts have been made to investigate organic non-linear optical materials. Organic non-linear optical materials are often formed by weak Vander walls and hydrogen bonds and hence possess high degree of delocalization. n-benzoyl glycine (Hippuric acid) is a colorless NLO crystal with chemical formula $C_6H_5CONHCH_2$-COOH [1-7]. It is believed to be a natural regulator of urinary saturation with regard to calcium oxalate crystallization which is the most chemical constituent encountered among all the urinary stones. It crystallizes in orthorhombic structure. It has been demonstrated that organic crystals can have very large non-linear susceptibilities compared with inorganic crystals [5]. In this paper we have reported the growth and characterization of pure and some chlorides doped n-benzoyl glycine crystals through XRD, TGA/DTA, FTIR and UV transmission studies.

MATERIALS AND METHODS

Experimental procedure

n-benzoyl glycine is a white crystalline powder, has a molecular formula $C_9H_9No_3$ with molecular weight of 179.18. The solubility of n-benzoyl glycine is moderately high in acetone when compared to hot water. So we have chosen acetone as the solvent. The chemicals used in this study were AR grade obtained from E-Merck (Germany) and used without purification. The solvent was taken in a beaker and the purified material was added gradually and continuously stirred for about 2 hours to avoid the co-precipitation of the salt and to get the super saturated solution. The above supersaturated solution was carefully filtered and covered for the controlled evaporation. The crystals of n-benzoyl glycine were grown by slow evaporation technique. The seed crystals grew to optically transparent and mechanically stable bulk crystals. To improve the purity of the synthesized salt, recrystallization process was repeated for two times. In order to obtain the doped crystals, 0.5M of Barium Chloride ($BaCl_2$), Lithium Chloride ($LiCl_2$) and Copper Chloride ($CuCl_2$), solutions were added to n-benzoyl glycine dissolved in acetone separately. The grown crystals are optically transparent and obtained after 15 days and the photographs of the grown crystals were shown in Figure 1(a), 1(b), 1(c) and 1(d) respectively.

Figure 1(a)

Figure 1(b)

Figure 1(c)

Figure 1(d)

RESULTS AND DISCUSSION

X-ray diffraction analysis:

The grown crystal of pure and doped has been subjected to powder X-ray diffraction. The powder form of the above said crystal was taken for the analysis using P - Analytical X'Pert Pro Philips X-diffractometer with CuKα1 (λ =1.5406Å) radiation. The resulted powder X-ray diffraction pattern of pure and Barium chloride, Lithium chloride and Copper chloride doped n-benzoyl glycine are shown in Figure 2(a), 2(b), 2(c) and 2(d) respectively. Experimental d values of pure samples are in well agreement with standard JCPDS values [6]. The XRD studies confirm the crystallinity of the salt and it belongs to orthorhombic structure. The variations in intensity of peaks of doped crystals may be attributed to the incorporation of dopants in crystal lattice.

FTIR Spectroscopic analysis:

The FTIR analysis of the samples was carried out between 400 to 4000cm^{-1} using spectrum RXI instrument. The FTIR spectra of pure and Barium chloride, Lithium chloride and Copper chloride doped n-benzoyl glycine crystals are shown in Figure 3(a), 3(b), 3(c) and 3(d) respectively.

The intense sharp peak at 3332 cm^{-1} the higher energy region is assigned to NH-stretching mode. The broad band [7] with maximum at 3076 cm^{-1} is due to hydrogen bonded OH-stretching. There are less intense resolved bands between 1900 and 2700cm^{-1} and these are attributed to hydrogen bonding interaction in the crystal lattice. The intense sharp peak at 1744cm^{-1} is due to the C=O stretching of COOH. The peak at 1558.22 cm^{-1} is assigned to aromatic ring skeletal vibrations .The O-H stretching of COOH group appears at 3077 cm^{-1} and 3090 cm^{-1}. The CH_2 bends are observed at 1398 cm^{-1}. The C-O-H bends is observed at 1393 cm^{-1}. The spectra of doped crystals illustrates that there is an appreciable shift of peak positions to lower or higher values, broadening of bands and reduction in peak intensities suggesting incorporation of dopants in the crystal lattice. The FT-IR analysis confirms the substitution of dopants.

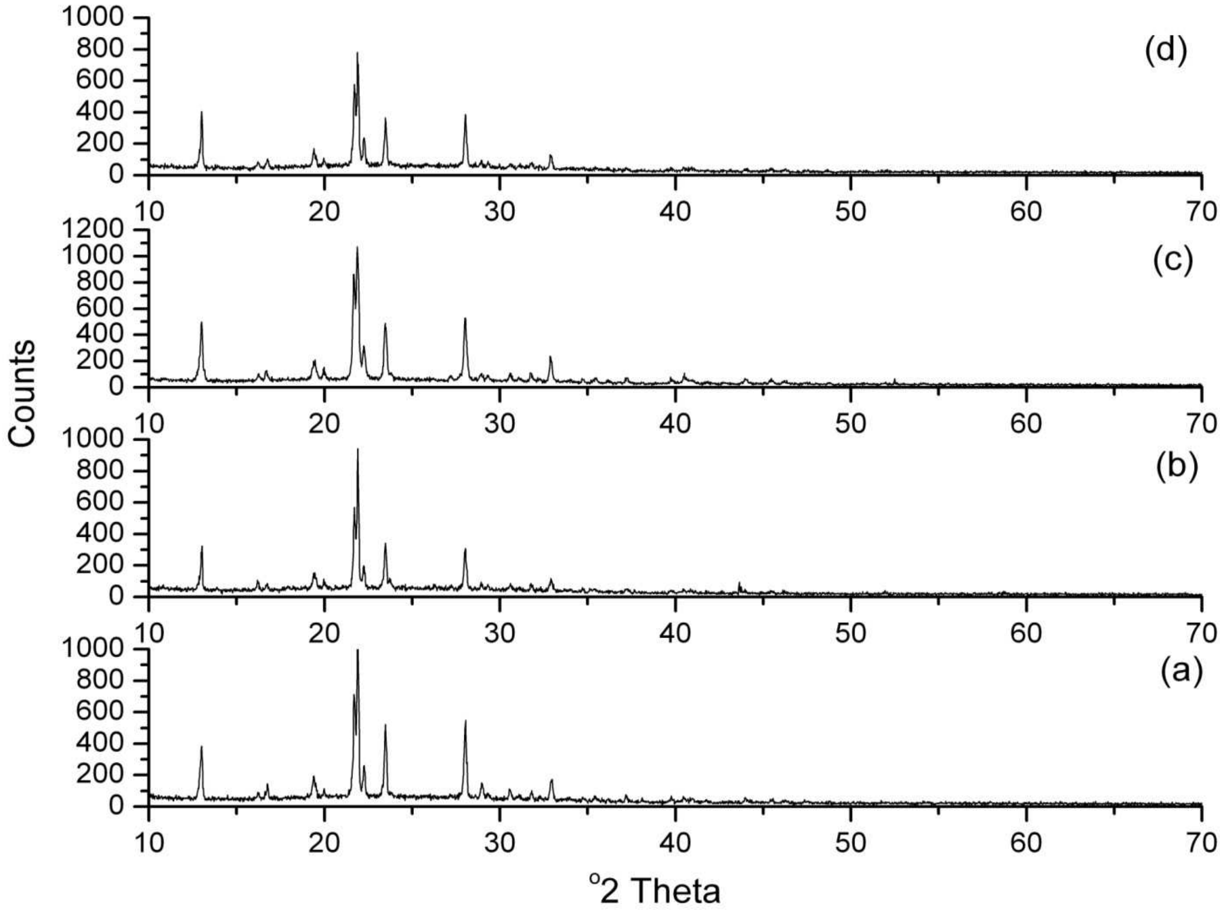

Figure (2) XRD Pattern of pure and doped n-benzoyl glycine crystals

Thermal analysis:

The thermal properties (TGA/DTA) of the pure and doped crystals were carried out by using the instrument SDT Q600 V20.9 Build 20. The TGA was carried out in air atmosphere between 20°C to 400°C at a heating rate of 10°C/m. The trace of TGA and DTA are recorded in the same spectrum. The material exhibits sharp weight loss starting at 210°C. But below this temperature no weight loss is observed. Hence the crystal is completely devoid of any inclusion of acetone, which was used as the solvent for crystallization. Hence the compound is found to be stable up to 210°C. The resulting thermogram (TGA) and its differential derivative (DTA) of pure and Barium chloride, Lithium chloride and Copper chloride doped n-benzoyl glycine are shown in Figure 4(a), 4(b), 4(c) and 4(d) respectively and appear nearly similar. Thermogram of pure crystal shows that the decomposition starts at 210°C and afterwards a sharp decrease in weight is observed up to 278°C. Thermal decomposition of doped crystals begins at 213°C and ends at 312°C. A slight increment is observed in first stage of decomposition of temperature of doped crystal suggesting that $BaCl_2$ impurity has modified the thermal stability of n-benzoyl crystal.

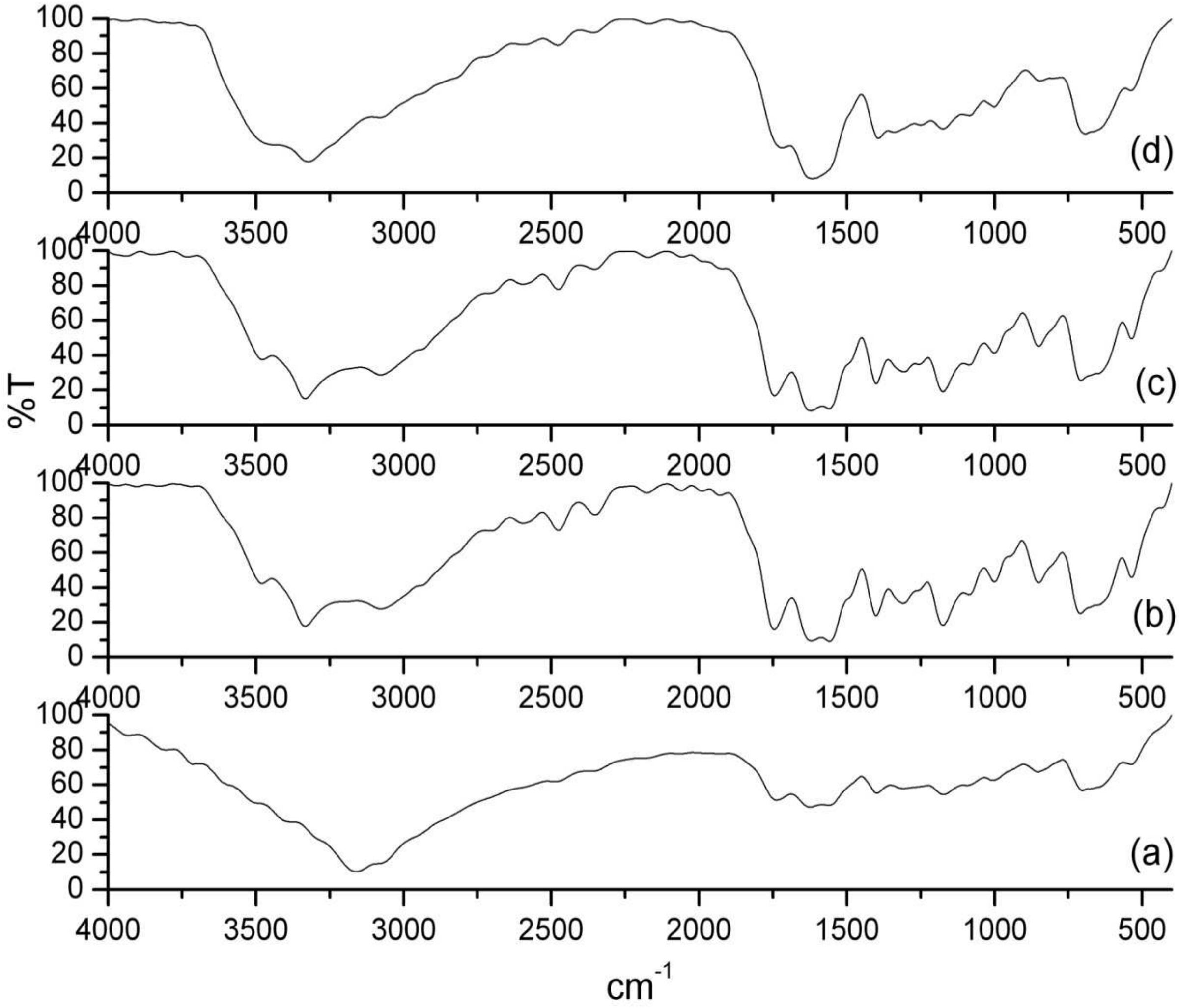

Figure (3) FTIR Pattern of pure and doped n-benzoyl glycine crystals

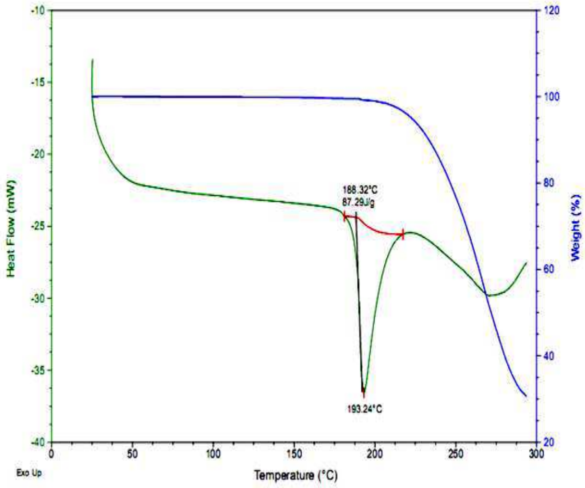

Figure 4(a)

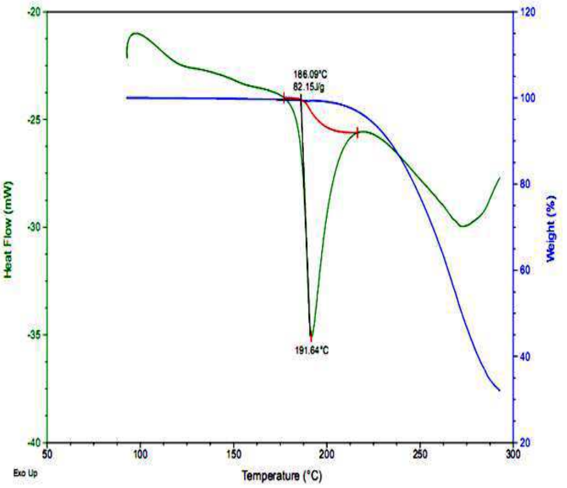

Figure 4(b)

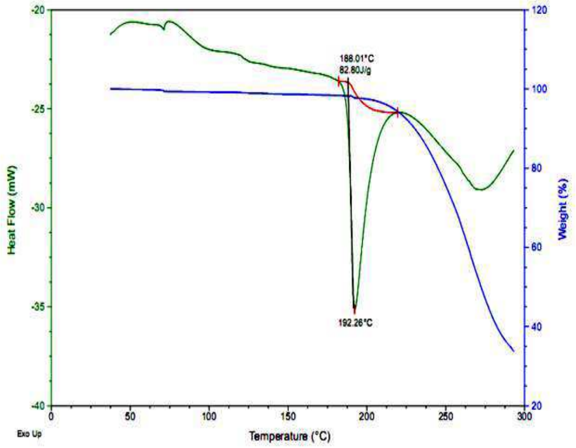

Figure 4(c)

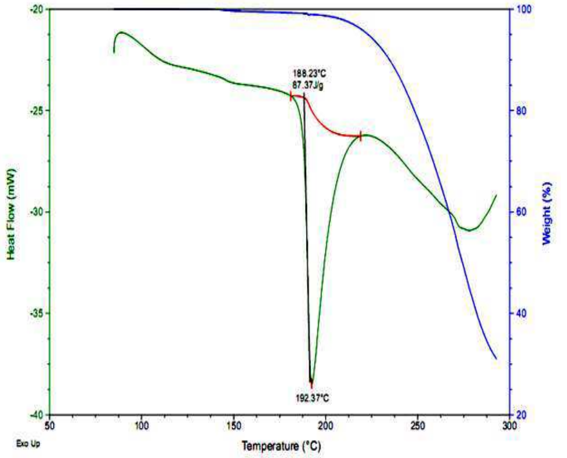

Figure 4(d)

The DTA curves of pure and Barium chloride, Lithium chloride and Copper chloride doped n-benzoyl glycine are shown in Figures 4(a), 4(b), 4(c) and 4(d) respectively. A sharp endotherm at about 188.32°C for pure is due to the melting point of n-benzoyl glycine crystal, which is very close to the reported values of melting point [8]. In case of doped crystal, it is shifted to 188.01°C, 186.09°C and 188.23°C which agree with measured experimental values of melting point.

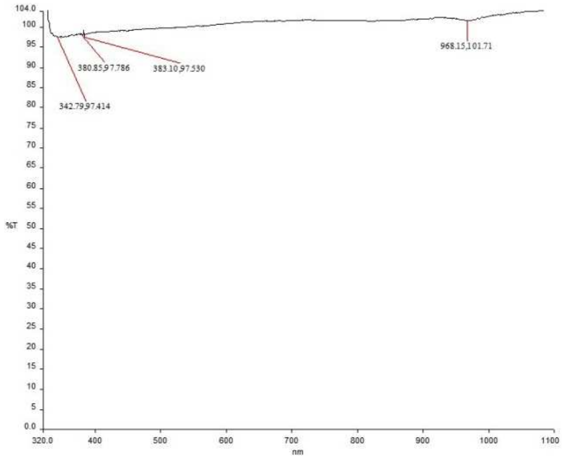

Figure 5(a)

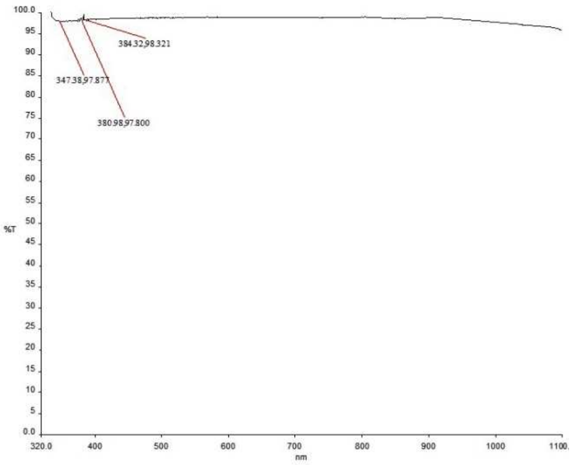

Figure 5(b)

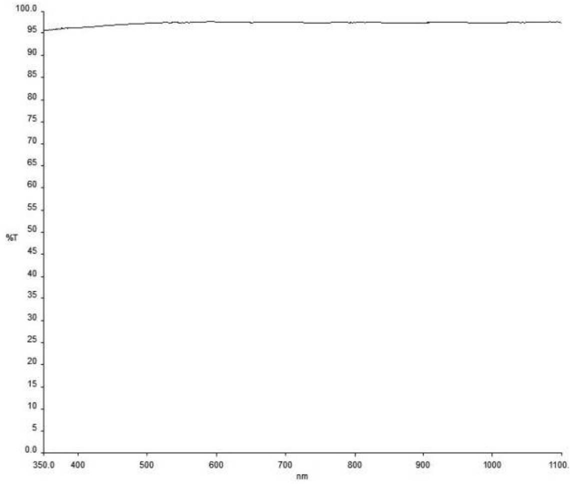

Figure 5(c)

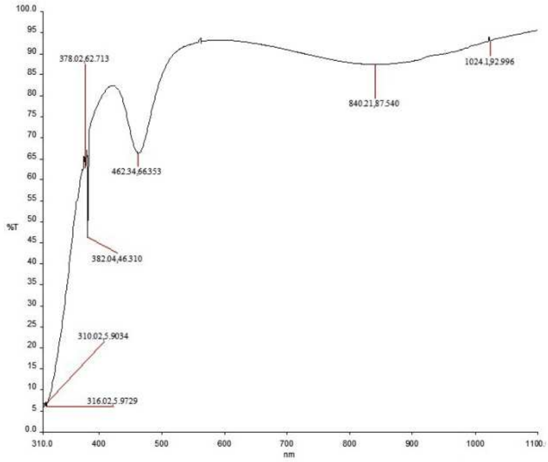

Figure 5(d)

UV-spectral analysis:
The UV transmission spectrum of pure and doped crystals was recorded using Lambda35 UV-spectrometer in the range of 300-1100 nm covering the entire ultraviolet, visible and the higher energy part of near infrared region to find the transmission range to know the suitability for optical applications [9].

The grown crystals of pure n-benzoyl glycine are transparent and colourless and there is no absorption in the visible region. The optical transmissions are not altered much in the case of doped crystals. But around 300nm there is a sharp decrease in transmittance due to absorbance leading to electronic excitation in this region. Absence of absorption in the region between 400 and 1100nm is an advantage as it is the key requirement for materials having NLO properties. In the case of coloured dopant copper chloride there is an absorption peak in the visible region shows that the transparency is slightly reduced. The absorbance may be assigned to electronic transition in the aromatic ring or C=O groups. The recorded transmission spectrum of pure and Barium chloride, Lithium chloride and Copper chloride doped n-benzoyl glycine are shown in Figure 5(a), 5(b), 5(c) and 5(d) respectively.

CONCLUSION

The optically transparent pure and doped n-benzoyl glycine single crystals were successfully grown by slow evaporation technique at room temperature. X-ray diffraction studies confirmed the structure of the grown crystals. TGA/DTA measurement reveals that the presence of impurities slightly increases the thermal stability of n-benzoyl glycine crystals. UV spectral studies confirmed that doped crystals may find useful optical window applications in the wavelength region 400-1200 nm. The functional groups were identified using FTIR technique.

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