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Comparison of physical properties of pure and doped new metal organic crystal Bisthiourea sodium nitrate (BTSN)

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ABSTRACT

Single crystals of pure and Na^+ doped Bisthiourea Sodium Nitrate (BTSN), semi organic crystal were grown by slow evaporation technique. The grown crystals were subjected to various studies such as single crystal X-ray diffraction (XRD), UV–vis studies, Fourier transform infrared (FTIR), TG–DTA, and Vickers's microhardness were also measured. Single crystal X-ray diffraction studies reveal the crystals belong to orthorhombic crystallographic system. The mechanical property of the grown crystal was determined by microhardness studies.

1. Introduction

Thiourea a centrosymmetric molecule which on coordination with metal ion gives a non centrosymmetric material. Metal complexes of thiourea commonly called semiorganics include the advantage of both organic and inorganic part of the complex. Thiourea molecules can coordinate with metal-ions to form a stable coordinate complex which can be crystallized by solution growth technique. Single crystals of thiourea are being used extensively and have vast demand in the electronic industry as polarization filter, electronic-acoustic devices. The thiourea crystals also exhibit pyroelectric effect, which utilized in infrared (IR), ultraviolet (UV), scanning electron microscopy (SEM) detection and infrared imaging. This new class of materials has come to be known as semi-organic [1,2,3] with metal organic coordination compounds [4]. Thiourea possesses large dipole moment and it forms number of NLO active metal coordination compounds [3]). In the present investigation an attempt has been made to grow pure and Na^+ doped bisthiourea sodium nitrate.

2. Experimental

2.1. Crystal growth

Raw materials for the growth of the title compound was synthesized by mixing aqueous solutions of sodium nitrate and thiourea in the ratio 1:2 and the corresponding chemical reaction is given below as,

$$Na(No_3)_2 + CS(NH_2) \rightarrow Na[CS(NH_2)](NO_3)_2$$

The super saturated solution was prepared using the synthesized salt. The product was purified by repeated recrystallization before it is used for the crystal growth. Single crystals of bisthiourea sodium nitrate were obtained from solution using slow evaporation technique after a period of two weeks. The seed crystals of BTSN were used to grow bulk size Crystals. For the growth of the doped crystals, 2% of Na^+ were added to the pure solution. Colorless optically good quality single crystals are grown and shown Fig. 1.

2.2. Characterization techniques

The single crystal XRD data of the pure and Na^+, doped single crystals are obtained to find the lattice dimensions using ENRAF NIUS CAD-4 X-ray diffractometer. The FTIR was recorded in the range of 4000–400 cm^{-1} BERKIN ELMER RXI spectrometer. The optical transmission spectrum was recorded in the range of 190–1100 nm using BERKINELMER LAMBDA 35 spectrometer.

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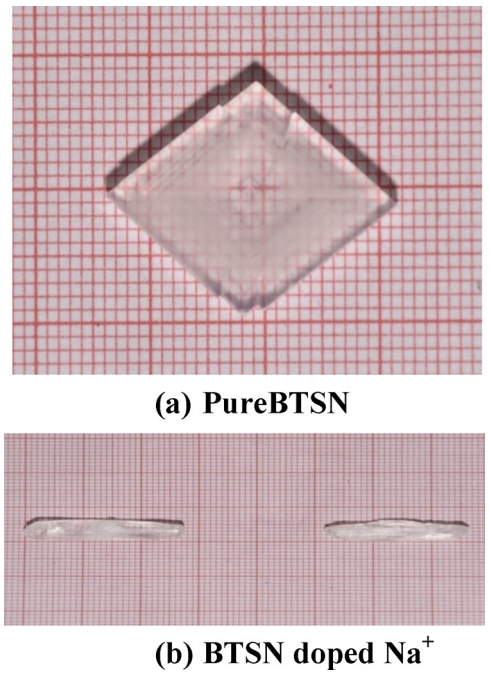

Fig. 1. Photographs of grown crystals.

Powder XRD studies were carried out using XPERT-PRO Diffractometer system. Thermal stability of the sample has been analyzed using DTA/TGA.

3. Results and discussions

3.1. Single crystal XRD

The single crystals of BTSN with dopants have been subjected to X-ray diffraction studies NONIUS CAD4 X-RAY diffractometer. The calculated values are tabulated in Table 1. It is observed that the grown crystals belong to orthorhombic system and the space group is PMMN.

3.2. Powder XRD studies

X-ray powder diffraction pattern of BTSN, BTSN-Na^+, was recorded using XPERT-PRO diffractometer system. The powder sample was scanned in step size [°2Th.] 0.0170. For a interval of 2 s over 2θ range of 10–70°. The powder XRD values were indexed for BTSN, BTSN-Na^+ is shown in Fig. 2.

3.3. FTIR spectral studies

The FTIR spectrum of the grown crystal is shown in Fig. 3. A number of reports are available on the IR absorption studies of thiourea [5,6]. When BTSN spectrum was compared with spectra of dopants, a shift in the peaks observed confirmed the metal coordination with thiourea [7,8]. In the complex, the coordination of sodium with thiourea may occur either through nitrogen or sulphur [9]. Most of the metals form a complex through sulphur [11]. The peaks observed in BTSN-3423 cm^{-1}, BTSN-Na^+ 3795 cm^{-1},are

Table 1
Single crystal XRD data.

Sample name	*a* (Å)	*b* (Å)	*c* (Å)	Volume (Å)^3
BTSN	5.483	7.646	8.564	359.0
BTSN-Na^+	5.641	7.364	8.356	347.1

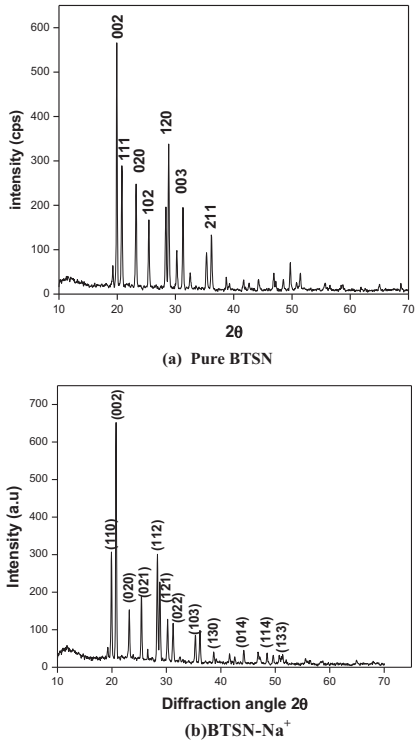

Fig. 2. Powder XRD pattern. This shows the powder XRD pattern of pure BTSN and doped single crystals

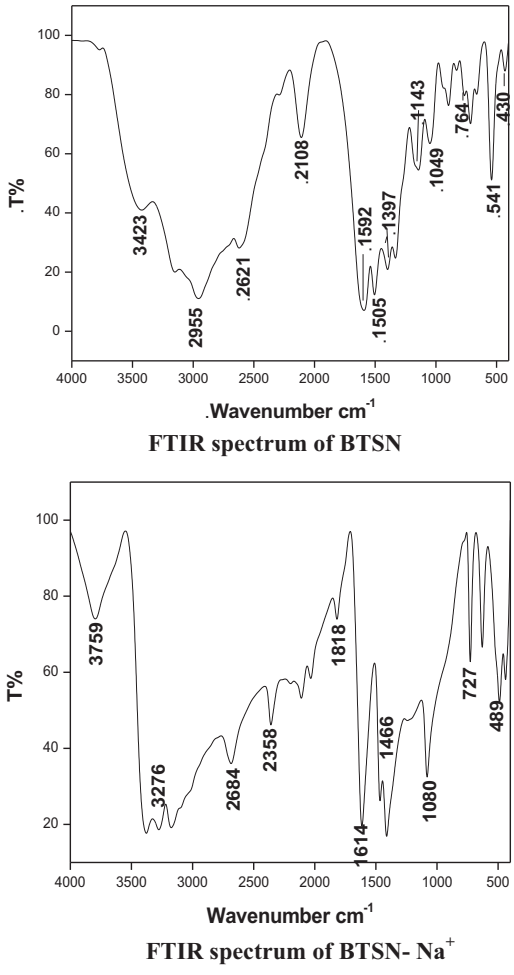

Fig. 3. FTIR spectrum. This shows the FTIR of pure BTSN and doped single crystals.