Growth and characterization of 2-aminothiazole – 3,5-dinitrosalicylic acid complex

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A novel organic material 2-AT and 3,5-DNS has been synthesized and good quality crystals were grown by slow evaporation method. UV-Vis transmittance spectrum has been taken for the crystal. FT-IR spectrum was recorded to identify the various functional groups present in the crystal. The powder X-ray diffraction study was used to analysis the crystalline nature of the complex. The mechanical study was carried out by Vickers diamond pyramid indenter. Photoluminescence studies were also carried out for the grown crystals.

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Keywords: Organic compounds, Crystal growth, X- ray diffraction, Microhardness, Band gap

1. Introduction

Optics has emerged as one of the most attractive fields of current research. In order to satisfy today technological requirement, new materials are very essential [1]. The organic materials has great importance for applications in areas such as optical modulation, optical switching, optical logic, frequency shifting, high optical damage threshold and optical data storage for developing technologies in telecommunications and signal processing [2-6]. The title compound has molecular formula of $C_{10}H_8N_4O_7S$ with molecular weight 328.20 and the crystallographic properties, IR, NMR, and UV were reported by Hassan A. Mohamed et al. [7]. However, to best of our knowledge no through report is available in this material so in this paper we reported powder XRD, photoluminescence and microhardness studies of the crystal.

2. Experimental

Commercially available 2-AT 3,5-DNS were taken in equimolar ratio. The calculated amounts of reactants were dissolved in THF/Methanol mixed solvents and stirred well about two hour using the magnetic stirrer. The saturated solution was filtered twice by Whatmann 41 filter paper to remove the impurities. The solution of 2-AT 3,5-DNS was taken in a 250 ml beaker with a perforated lid in order to control the evaporation rate, and kept at room temperature in undisturbed condition for crystallization. After ten days good quality and well developed crystals were taken from the mother solution. A quality of single crystals of 2-AT 3,5-DNS shown in Fig.1.

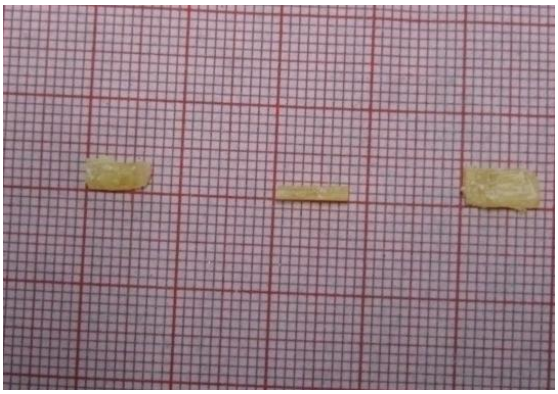

Fig.1 . 2AT- 3,5-DNS single crystals.

3. Results and discussion

3.1 X-Ray diffraction analysis

The grown single crystals of 2-AT 3,5-DNS have been subjected to powder X-ray diffraction. Powder form of the above mentioned crystal was taken for the analysis using a REICH SEIFERT X-ray diffractometer. The sample was scanned over the range 10-80^0 at the rate of 1^0 per min. The powder X-ray diffraction pattern of the grown crystal is shown in Fig. 2. A well defined and sharp peaks signify the good crystalline nature of the compound.

3.2 FT-IR analysis

The FT-IR spectrum of the grown crystal was carried out using BRUKKER IFS 66 V spectrophotometer using KBr pellet technique and the resultant spectrum is depicted in Fig. 3. The spectrum carries sharp intense

peak at 3364 cm^{-1} for OH stretching vibration. The peak at 3089 cm^{-1} confirms the NH stretching vibration. C=O stretching vibration occurs at 1704 cm^{-1}. The CH bending vibrations is observed in the range of 1159 $cm^{-1.}$ The absorption band at 1345 cm^{-1} assigned to C=N stretching vibration. Also the peaks at 1345 cm^{-1}, 1441 cm^{-1} and 1413 cm^{-1} confirm the asymmetric and symmetric NO_2 stretching vibrations. CH out-of-plane bending vibration is observed in the range of 717cm^{-1}.

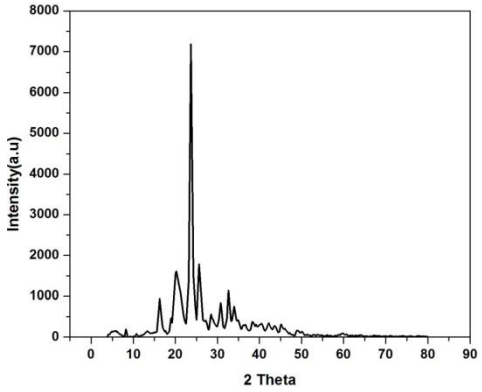

Fig. 2. Powder XRD spectrum of 2 AT - 3 5 DNS crystal.

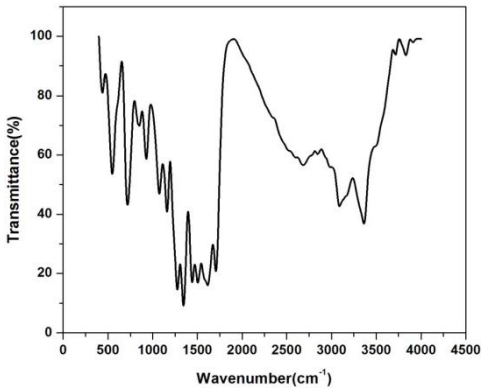

Fig. 3. FT-IR spectrum of 2 AT - 3, 5 DNS crystal.

3.3 UV – Visible transmittance studies

The UV-Visible spectrum was recorded using a Perkin-Elmer lambda 35 spectrophotometer in range 200-1100 nm covering the entire ultraviolet, visible and higher energy part of near infrared region to find the transmittance range to know the suitability for optical application. The transparency range is shown in Fig. 4. The figure shows that the crystal has lower cutoff wavelength at 299 nm and it has 95% transparency of the grown crystal. This makes the crystal potential candidate for optoelectronic applications [8].

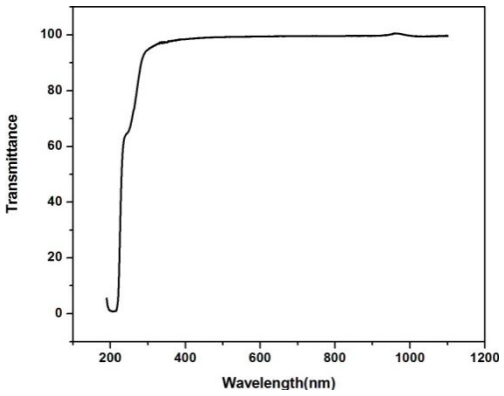

Fig. 4. Optical transmittance spectrum of 2 AT - 3, 5 DNS crystal.

3.4 Microhardness study

Microhardness measurements were carried out on a flat polished face of the crystals at room temperature using a diamond pyramid Vickers indenter. The length of the two diagonals of the indentations were measured and the Hardness number was calculated using the formula

$$H_v = 1.854p/d^2 \ (kg/mm^2).$$

where p is the applied load in kilogram and d is the mean diagonal length in millimeters. The indentation time was kept as 5 s for all the loads. Since crack initiation and material chipping became significant beyond 100 g of the applied load, hardness test could not be carried out above this load. Fig. 5 shows that hardness value increases with the applied load [9-10].

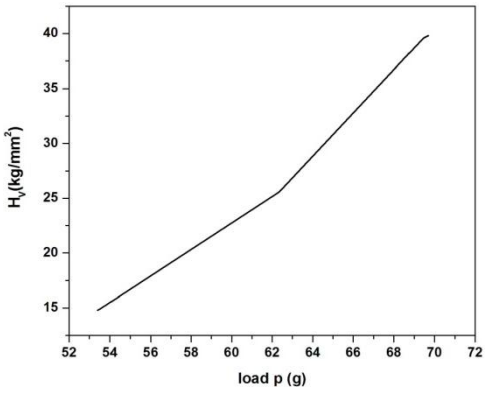

Fig. 5. Vickers hardness Plot.

3.5 Photoluminescence

The Photoluminescence studies were carried out for the crystals in the room temperature. The excitation spectrum was recorded in the range 200–1100 nm. The sample was excited at 230 nm (Fig. 6(a)). The emission spectrum was measured in the range 400–650 nm. The maximum emission wavelength is observed at 430 nm

(Fig. 6(b)). The results indicate that 2-AT 3,5-DNS crystals have a blue fluorescence emission. Also the crystal shows low UV absorption throughout entire visible region is one of the desirable properties for the optoelectronic applications. The direct band gap of the material was calculated using the relations h, c, λ (Eg=2.885 eV).

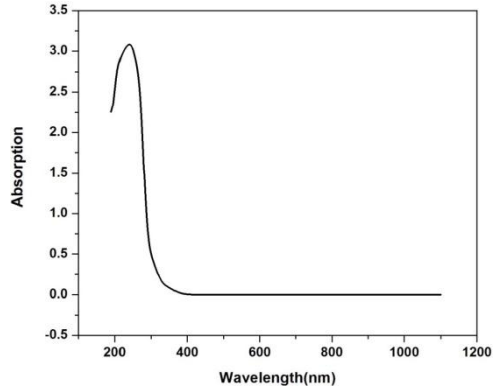

Fig. 6(a). Excitation spectrum of 2 AT - 3, 5 DNS crystal.

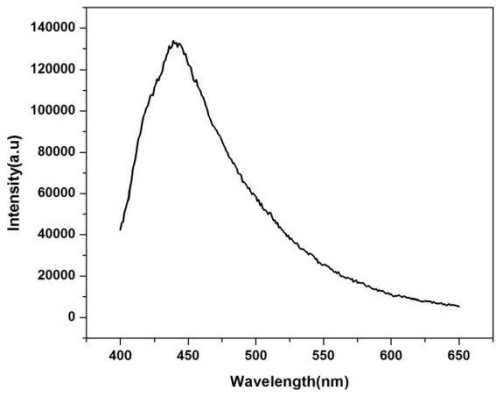

Fig. 6(b). Emission spectrum of 2 AT - 3, 5 DNS crystal.

4. Conclusions

The 2-aminothiazole and 3,5-dinitrosalicylic acid single crystals has been grown by slow evaporation technique at room temperature. The UV–Visible spectrum shows that the transmittance of the crystal. The presence of various functional groups was confirmed by FTIR spectrum. Powder X-ray diffraction study was used to confirm the crystallinity nature and show that 2-AT 3,5-DNS crystal has monoclinic structure. The microhardness value increased with the applied load. The direct band gap was calculated using photoluminescence studies.

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