

# Growth, Optical and Microhardness Studies of 2-Aminothiazole- 3, 5-Dinitrobenzoic Acid Complex

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**ABSTRACT:** Single crystals of 2-Aminothiazole 3,5-Dinitrobenzoic acid has been synthesized and good quality optical crystals were grown by slow evaporation technique at room temperature. The crystallinity nature of the grown crystal was confirmed from X-ray diffraction technique. An optical transmittance study was also carried out by UV – Vis spectra. FTIR spectra confirm the presence of functional groups in the grown crystal. The dielectric measurements were carried out in the range of 50Hz to 2MHz. The dielectric constant was seen to increase exponentially at lower frequencies. The microhardness studies were carried out using Vickers hardness indenter. Photoluminescence study shows that maximum emission occurs at 435nm.

**Keywords:** 2AT 3,5DNB, Microhardness, XRD, band gap, photoluminescence.

## 1. Introduction

With rapid advancement of the microelectronic and the optoelectronic industry in the country, the demand for crystals has increased dramatically during the past two decades. The requirement for better and well characterized single crystals has been a driving force behind extensive research and development in crystal growth [1, 2]. Organic materials have very high optical non-linearity and great possibilities for twisting the molecular structure using molecular engineering and chemical synthesis. Also they have attracted much attention because of their application in frequency shifting, optical modulation optical switching, optical logic and optical memory for the emerging technologies in areas such as optical interconnections, telecommunications and signal processing [3,4].

Hassan A. Mohamed et al [5] reported the structural and optical studies of this crystal but to best of our knowledge there is no detailed studies are available on this material. So, in the present paper we report the powder XRD, Dielectric, Microhardness and Photoluminescence studies of 2AT 3, 5DNB crystals.

## 2. Materials and Methods

In the present investigation the title compound was synthesized by dissolving 2-Aminothiazole and 3,5-Dinitrobenzoic acid in equimolar ratio using THF/Methanol Mixed solvents. The product was stirred well and filtered twice using whatmann filter paper to remove the impurities and covered with thick paper with perforated lid in order to

control evaporation rate. Slow evaporation technique was employed to grow the single crystals. After 15 days good quality of crystals were harvested from the mother solution. The grown crystal is shown in Fig.1.

## 3. Results And Discussion

### 3.1 Powder X-Ray Diffraction Analysis

Powder X-ray diffraction study was carried out for the grown crystal by employing SEIFERT JSO DEBYEFLEX diffractometer with Ni filtered CuK $\alpha$  (Wavelength  $\lambda$ =1.5405 Å) radiation.

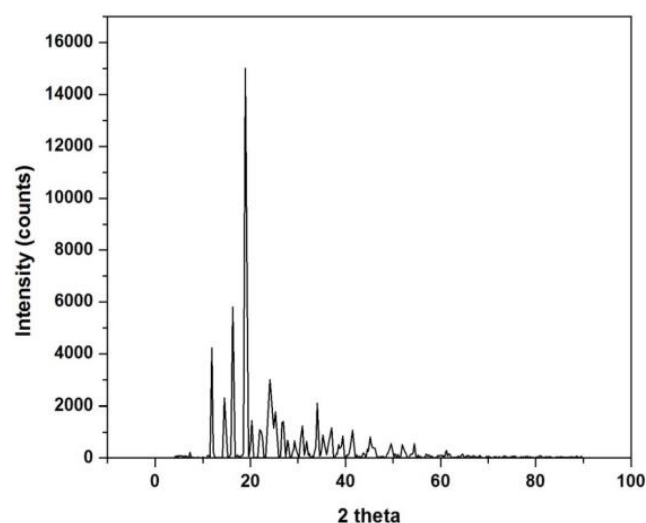


Fig 2. Power XRD Spectrum of 2 AT - 3, 5 DNB Crystal

The powdered sample was scanned over the range  $20-70^\circ$  at a rate of  $1^\circ/\text{min}$ . The powder X-ray diffraction spectrum is displayed in Fig. 2. The sharp intense peaks on the pattern reveal that the crystallites are pure and dislocation free.

### 3.2 UV – Visible Transmittance Studies

The transmittance spectra of the grown crystals were recorded in the wavelength region from 200-1100 nm using PerkinElmer Lambda 35 UV-Vis spectrometer. The scanned spectrum is displayed in Fig. 3 and the spectrum shows the crystal is transparent in the entire visible region. This makes the crystal a potential candidate for optical applications.

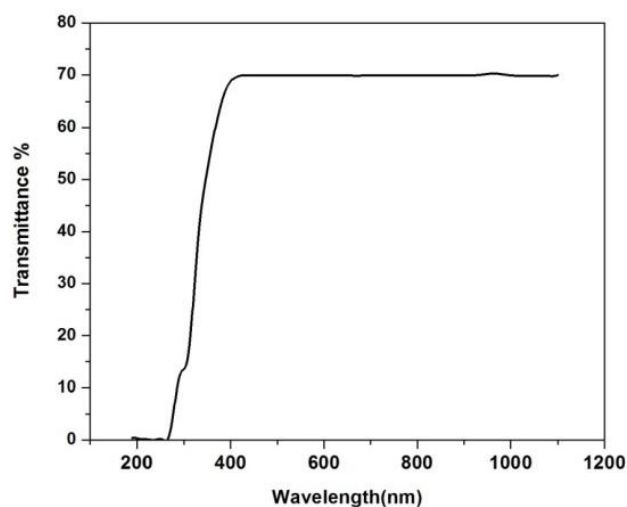


Fig.3. UV- Vis transmission Spectrum

### 3.3 FT-IR Analysis

The FT-IR spectra of 2-AT 3,5-DNB crystal were recorded in the wave number range of 4000 to  $450\text{ cm}^{-1}$  using the KBR pellet technique.

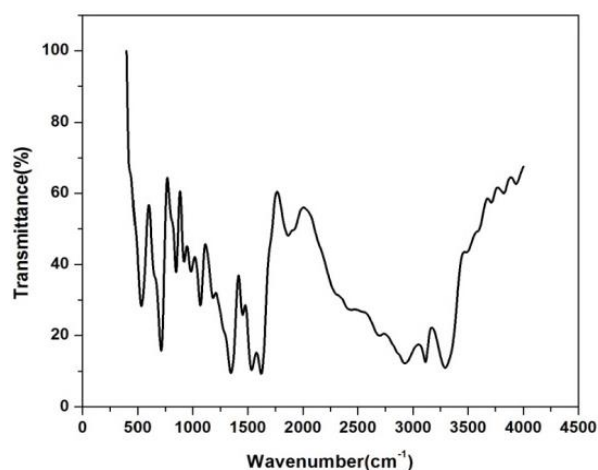


Fig 4. FTIR Spectrum of 2 AT - 3, 5 DNB Crystal

The observed peak at  $3292.19\text{ cm}^{-1}$  is assigned to OH stretching vibration. The peak broadening in this region is assigned to the intermolecular hydrogen bonding. The NH stretching vibration is observed at  $3112\text{ cm}^{-1}$ . C=O stretching vibrations are observed in the range of  $1866.01$  and  $1620\text{ cm}^{-1}$  respectively. NO stretching vibration occurs

at  $1420$  and  $1345\text{ cm}^{-1}$  also it is assigned to CN stretching modes. CH out-of-plane bending vibration takes place in the region of  $919.08\text{ cm}^{-1}$ . The FTIR absorption spectrum of 3,5-DNB is shown in Fig. 4.

### 3.4 Dielectric Studies

The grown crystal is Polished and silver plated on the opposite faces were two probe connected to a 3532-50 Hioki LCR Meter (Japan). Dielectric Constant and dielectric loss were recorded at room temperature Fig. 5(a) and 5(b) shows the variation of dielectric constant and loss with log frequency. The dielectric constant is very high at lower frequency range and found to decrease with the increase in frequency due to the presence of space charge polarization [6]. The dielectric loss is very low at high frequency this shows that the crystal contains low level defects. The low value of dielectric loss at high frequency suggests the sample possesses an enhanced optical quality which is of vital importance for optoelectronic applications.

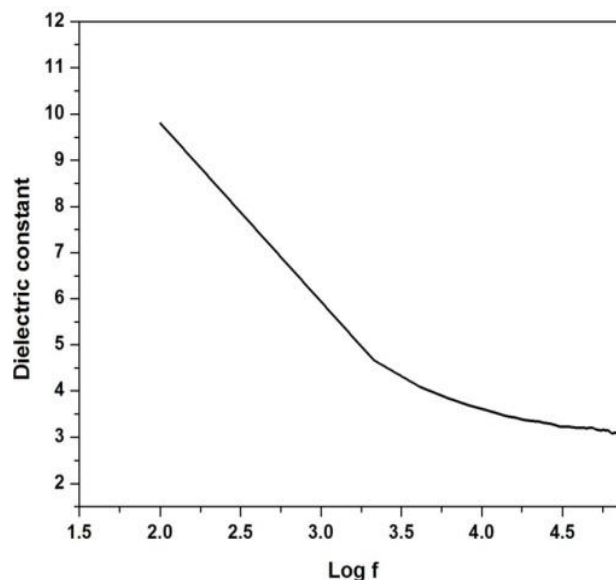


Fig 5. Dielectric constant Vs log f

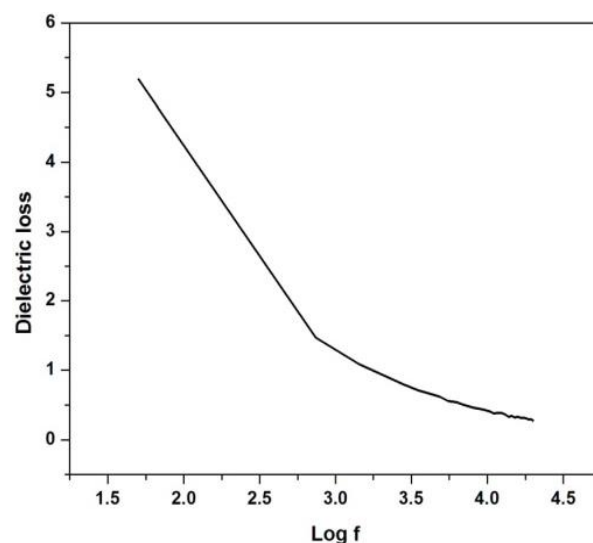


Fig 6. Dielectric loss Vs log f

### 3.5 Microhardness Studies

Vickers microhardness test was carried out for the grown crystals. The measurements were made at room temperature at constant indentation time of 5s. Indentation impressions were measured using leitz Wetzlar miniload hardness tester, fitted with a diamond pyramidal Vickers indenter. The microhardness was calculated using the formula  $H_v = 1.854p/d^2$  ( $\text{kg/mm}^2$ ). Where  $p$  is the applied load in kilogram and  $d$  is the diagonal length of the indent in mm. for applied load above 100g micro cracks were observed around the impression [7-8]. It is observed from the Fig. 6 that when load increases hardness also increases this may be due to the release of internal stresses.

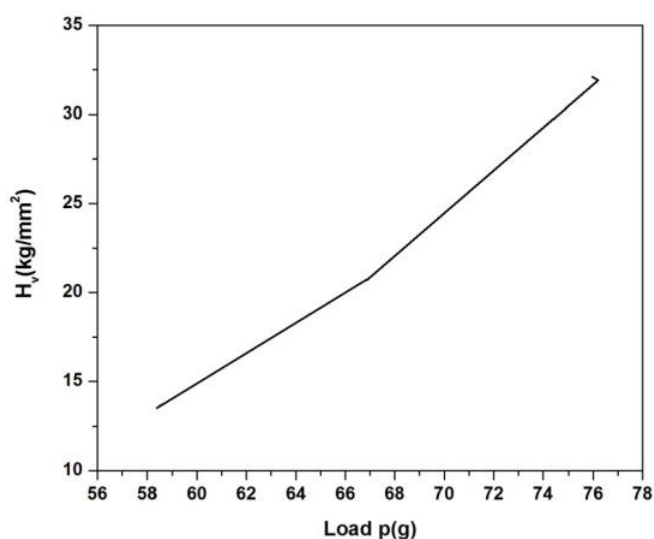


Fig 7. Vickers hardness Plot

### 3.6 Photoluminescence

Fluorescence may be expected generally in molecules that are aromatic which contains multiple conjugated double bonds with a high degree of resonance stability.

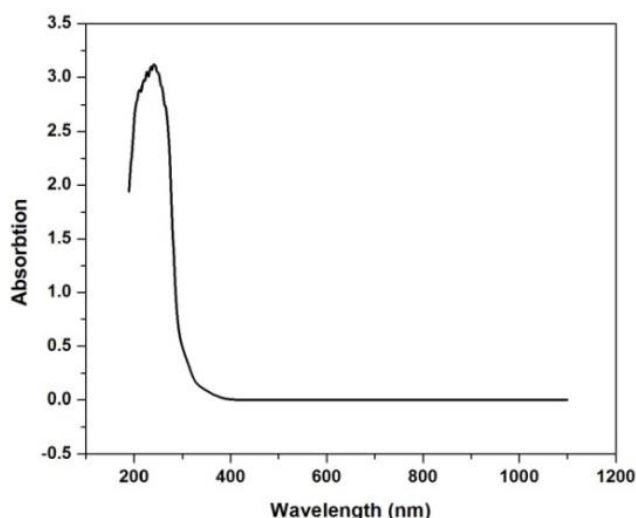


Fig 8. Excitation spectrum of 2AT, 3,5-DNB Crystals

The emission spectrum was recorded in the range of 200–1200 nm at room temperature. Spectra are displayed in

Fig. 7(a) and 7(b). The sample was excited at 233 nm. The emission spectrum was measured in the range 250–500 nm. The maximum emission wavelength is observed to be at 435 nm. The results indicate that 2 AT 3,5 DNB crystals have a blue fluorescence emission. Also 2 AT 3,5 DNB crystals shows low UV absorption throughout the entire visible region which poses the crystal a suitable candidate for optical applications. The direct band gap of the material was calculated using the relations  $h, c, \lambda$  ( $E_g = 2.852$  eV).

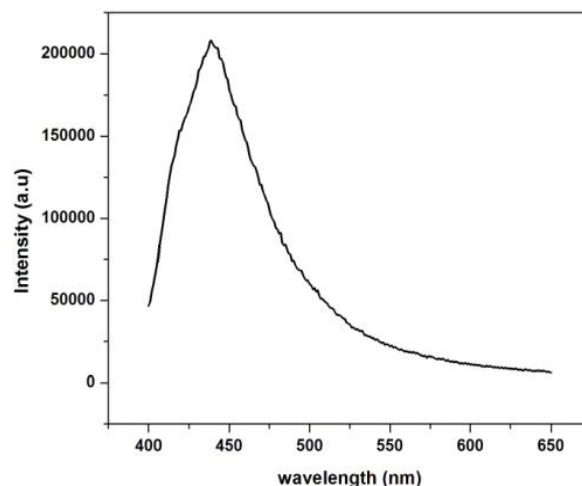


Fig 9. Emission spectrum of 2AT, 3,5-DNB Crystals

### Conclusions

The 2-Aminothiazole and 3,5-Dinitrobenzoic acid complex has been prepared and grown by slow evaporation technique. The UV-Visible spectrum reveals that the grown crystals have a cut-off wavelength of 240 nm, which can be employed in the optical applications in the entire visible region and the near IR region. The presence of various functional groups was confirmed by FTIR spectrum. Powder X-ray diffraction studies confirm the crystallinity and show that 2-AT 3,5-DNB crystal has monoclinic structure. Dielectric constant increases in the lower frequency and the low value of dielectric loss indicates the purity of the crystal. The Microhardness value increases with the applied load. From photoluminescence study the direct band gap of the material has been calculated and crystals have a blue fluorescence emission.

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